Piezoresistive Effect in 4H Silicon Carbide towards Mechanical Sensing in Harsh Environments

by

Tuan-Khoa Nguyen
BE, ME

Principal Supervisors
Assoc. Prof. Dzung Viet Dao
Dr. Yong Zhu

School of Engineering and Built Environment
Queensland Micro- and Nanotechnology Centre
Griffith University

Submitted in fulfilment of the requirements of the degree of
Doctor of Philosophy

November 2018
Declaration of Authorship

I, Tuan-Khoa Nguyen, declare that this thesis entitled, “Piezoresistive effect in 4H silicon carbide towards mechanical sensing in harsh environments” and the work presented in it are my own. I confirm that:

“This work has not previously been submitted for a degree or diploma in any university. To the best of my knowledge and belief, the thesis contains no material previously published or written by another person except where due reference is made in the thesis itself.”

(Signed) ______________________________
Tuan-Khoa Nguyen

(Date) 30/07/2018
Abstract

The fast-growing demand for mechanical sensors in harsh environments (e.g. mining/deep oil explorations, power/chemical plants and space explorations) urges the development of advanced materials which can replace silicon to work in these conditions. The superior mechanical properties of 4H silicon carbide (4H-SiC) combined with the physical stability at high temperatures offer new capabilities to develop MEMS sensors for those challenging situations. The piezoresistive effect is positioned as one of the most significant sensing mechanisms used in MEMS/NEMS sensors to detect or monitor mechanical signals, such as pressure, inertia, acceleration and deflection. Additionally, the use of micromachined sensors enables the miniaturization and integration capabilities while requiring low power consumption and simple readout circuitries.

The main goals of this thesis are to investigate the piezoresistive effect in p-type 4H-SiC and to develop 4H-SiC based sensors which can be utilised for mechanical sensing in harsh environments. First, a literature review of developments and current research interests in the piezoresistive effect and silicon carbide materials for mechanical sensing are presented. Next, theoretical analyses on the strain induced effect in the silicon carbide energy band structure are thoroughly conducted. Moreover, the calculation of the coordinate transformation is performed to determine the fundamental piezoresistive coefficients in the (0001) plane of 4H-SiC. To verify the theoretical results, the fabrication of 4H-SiC sensing devices and experimental measurements are carried out. As such, the piezoresistive effect in p-type 4H-SiC at room and high temperatures is discovered by measuring the effect in longitudinal and transverse piezoresistor configurations. Additionally, the piezoresistive coefficients in the (0001) plane of 4H-SiC are investigated, providing insight into the orientation dependence of the piezoresistive effect in p-type 4H-SiC for the optimization of the sensing design. Subsequently, 4H-SiC based sensing devices are fabricated and characterised including a 4H-SiC based van der Pauw strain sensor and a 4H-SiC based pressure sensor. The excellent linearity, good repeatability, and stability of these sensors are favourable for mechanical sensing applications. Additionally, the 4H-SiC based pressure sensor exhibits high sensitivity and good reliability in cryogenic and high temperatures, indicating the potential for hostile environmental sensing.
Acknowledgements

First and foremost, I would like to express my sincere gratitude to my principal supervisors Assoc. Prof. Dzung Viet Dao and Dr. Yong Zhu for their enthusiastic guidance, continuous support and encouragement during my candidature. Assoc. Prof. Dzung Viet Dao is a renowned expert in micro- & nano- scaled sensors/devices and the piezoresistive effect in advanced materials. His immense knowledge and passion have inspired me to pursue my research career. I am also grateful to work with Dr. Yong Zhu who I had insightful discussions with.

It was my pleasure to receive invaluable advices and have fruitful discussions with Prof. Nam-Trung Nguyen, the director of the Queensland micro- and nanotechnology centre (QMNC), who has been supportive and provided inspiring talks which are really helpful for young researchers like me. My sincere thanks to my lab members, Dr Hoang-Phuong Phan, Dr Toan Dinh, Mr Abu Riduan Md Foisal, Mr Jarred Fastier-Wooler, Mr Quan Nguyen, Mr Thanh Nguyen, for their cordial support in the sample fabrication and experiments.

I am indebted to QMNC staff members, Mr Alan Iacopi, Prof. Sima Dimitrijev, Dr. Jisheng Han, Mr Glenn Walker, Mrs Leonie Hold, and Mr Kien Chaik, who have assisted and trained me in using the state-of-the-art micro- and nano- fabrication facility at QMNC.

I am also thankful to Prof. Toshiyuki Toriyama at Ritsumeikan University and Prof. Koichi Nakamura at Kyoto University, who taught me the insight of the physics in the silicon carbide band structure.

I am also grateful to Assoc. Prof. Debbie G. Senesky, Ms Karen M. Dowling and other members of Xlab at Stanford University, who supported me in the fabrication and characterisation of the 4H-SiC based pressure sensor.

I thank with love to my wife who has always been with me even throughout the toughest time of my life. Without her love, care and understanding, I would not be what I am today. To my family, thank you for your love, support, and unwavering belief in me.

A special gratitude goes out to my friends Thao, Khoi, and Van Anh for their friendship and help.

Last but not least, I would like to thank Griffith University for providing me the prestigious scholarships, GUPRS and GUIPRS, which supported me in doing my PhD. It is my great pleasure to study and do research at Griffith University, a wonderful and dynamic environment for students all over the world.
List of publications during candidature

Papers with “†” mark are the components of this thesis and papers with “‡” mark are relevant to this thesis. (IF= Impact factor)

Published journal papers as the first author

† (J.26) **Tuan-Khoa Nguyen**, Hoang-Thuong Phan, Toan Dinh, Abu Riduan Md Foisal, Nam-Trung Nguyen and Dzung Viet Dao, “High-temperature tolerance of piezoresistive effect in p-4H-SiC for harsh environment sensing,” accepted for publication in *Journal of Materials Chemistry C* on **July 24th, 2018**. (IF=5.976)  
DOI: 10.1039/C8TC03094D

DOI: 10.1016/j.matdes.2018.07.014

DOI: 10.1063/1.5037545

† (J.23) **Tuan-Khoa Nguyen**, Hoang-Thuong Phan, Ji-Sheng Han, Toan Dinh, Abu Riduan Md Foisal, Sima Dimitrijev, Yong Zhu, Nam-Trung Nguyen, and Dzung Viet Dao, “Highly sensitive p-type 4H-SiC van der Pauw strain sensor,” *RSC Advances* vol. 8(6), pp. 3009–3013 (2018). (IF=2.936)  
DOI: 10.1039/C7RA11922D

DOI: 10.1109/LED.2017.2700402

**Publications**

DOI: 10.1021/acsami.7b15381

DOI: 10.1109/LED.2017.2726016

**Published journal papers as co-author**

(J.19) Toan Dinh, Hoang-Phuong Phan, Navid Kashaninejad, **Tuan-Khoa Nguyen**, Nam-Trung Nguyen, Dzung Viet Dao, “An on-chip SiC MEMS device with integrated heating, sensing and microfluidic cooling systems,” accepted for publication in *Advanced Materials Interfaces* on July 12th, 2018. (IF=4.834)
DOI: to be updated

DOI: 10.1016/j.matdes.2018.06.031

DOI: 10.1109/LED.2018.2850757

DOI: 10.1109/LED.2018.2808329

DOI: 10.1109/LED.2018.2806181


DOI: 10.1063/1.4979701

DOI: 10.1063/1.4979834

DOI: 10.1016/j.matlet.2017.03.118

DOI: 10.1063/1.4963258

DOI: 10.3390/s16081244

DOI: 10.1109/LED.2016.2579020

(J.2) Toan Dinh, Hoang-Phuong Phan, Tuan-Khoa Nguyen, Afzaal Qamar, Abu Riduan Md. Foisal, Thanh Nguyen Viet, Canh-Dung Tran, Yong Zhu, Nam-Trung Nguyen, and Dzung Viet Dao, “Environment-friendly carbon nanotube based flexible
Publications


(J.1) Abu Riduan Md Foisal, Hoang-Phuong Phan, Takahiro Kozeki, Toan Dinh, **Tuan-Khoa Nguyen**, Afzaal Qamar, Mirko Lobino, Takahiro Namazu, and Dzung Dao, “3C-SiC on glass: an ideal platform for temperature sensors under visible light illumination,” *RSC Advances*, vol. 6(90), pp. 87124–87127 (2016). (IF=2.936) DOI: 10.1039/C6RA19418D

**Published conference papers**


Contents

Declaration of Authorship i

Abstract ii

Acknowledgements iii

List of Publications iv

Contents ix

List of Figures xiii

List of Tables xvi

Abbreviations xvii

Physical Constants xix

Symbols xx

1 Introduction and research scopes 1

1.1 Introduction and research objectives 1

1.2 Research scope 4

1.3 Thesis structure 4

Bibliography 7
2 Research background and literature review

2.1 SiC material aspects

2.1.1 Crystallography of SiC

2.1.2 Physical properties of SiC

2.1.3 Growth process of SiC

2.1.3.1 Vapour phase growth

2.1.3.2 Solution phase growth

2.2 Piezoresistive effect

2.2.1 Piezoresistive effect in semiconductors

2.2.2 Theoretical analysis of piezoresistive effect

2.2.2.1 Tensor representation of piezoresistive effect

2.2.2.2 Physics of piezoresistive effect

2.2.3 Piezoresistive effect in SiC

2.2.3.1 Piezoresistive effect in SiC micro structures

2.2.3.2 Piezoresistive effect in SiC nano structures

2.2.3.3 Carrier concentration, orientation and temperature dependences of piezoresistive effect in SiC

2.3 SiC based sensors for harsh environments

2.4 Micromachining of SiC materials

2.4.1 SiC etching

2.4.2 SiC thin film transferring

2.4.3 Bulk micromachining

2.5 Summary of literature review and research goals

Bibliography
3 Methodology

3.1 Investigation on piezoresistive effect in 4H-SiC

3.1.1 Theoretical methods

3.1.1.1 Analysis of strain induced effect to the electrical conductance of p-type 4H-SiC

3.1.1.2 Graphical representation of piezoresistive coefficients

3.1.2 Experimental methods

3.1.2.1 Strain inducing method

3.1.2.2 Measurement of piezoresistive effect at high temperatures

3.2 Device development

3.2.1 Fabrication process

3.2.1.1 Micromachining

3.2.1.2 Laser scribing

3.2.2 Metallisation and contact characterisation

3.2.2.1 Metallisation

3.2.2.2 Ohmic contact and current leakage

Bibliography

4 Piezoresistive effect and piezoresistive coefficients in (0001) plane of p-type 4H-SiC

4.1 Magnitude of piezoresistive effect in p-type 4H-SiC

4.2 Orientation dependence of piezoresistive effect in (0001) plane of p-type 4H-SiC

5 Piezoresistive effect in p-type 4H-SiC at high temperature

6 4H-SiC based device development for mechanical sensing

6.1 P-type 4H-SiC van der Pauw strain sensor

6.2 P-type 4H-SiC pressure sensor at cryogenic and elevated temperatures
7 Conclusion and future work

7.1 Conclusion .............................................. 147

7.2 Future work and outlook ................................. 149
List of Figures

1.1 Technical requirements for sensors used in harsh environments. (Courtesy of Albert P. Pisano, University of California, San Diego) . . . . . . . . . 2


2.1 Si–C bonding in SiC . . . . . . . . . . . . . . . . . . . . . . . . . . . . 11

2.2 Crystal structure of three most common SiC polytypes: a) cubic 3C-SiC (β-SiC), b) hexagonal 4H-SiC, c) hexagonal 6H-SiC. Reprinted with permission from [2]. . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 11

2.3 The current status of SiC growth process available. Reprinted with permission from [2]. . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 13

2.4 Stress tensor components in the Cartesian coordinate . . . . . . . . . . 19

2.5 Two common configurations for the piezoresistive effect a) two-terminal (E∥J) and b) four-terminal (E⊥J) . . . . . . . . . . . . . . . . . . . . 20

2.6 Energy band structures of a) 4H-SiC and b) 6H-SiC. Courtesy of Kaeckell et al. [115]. . . . . . . . . . . . . . . . . . . . . . . . . . . . . 28

2.7 a) SiC piezoresistors placed on top of cantilever beam, b) Cross-sectional view of layers, and c) Measured longitudinal and transverse gauge factor of n-type 4H piezoresistors. Reprinted with permission from [96]. . . 29

2.8 Six conduction band minima at M points in the first Brillouin zone (six semi-ellipsoids and equivalent three full ellipsoids). Reprinted with permission from [118]. . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 30

2.9 Comparison of the GFs reported by Shor, Okojie and Toriyama. a) longitudinal b) transverse. Reprinted with permission from [118]. . . 31
2.10 a) Electromechanical characterization measurements, b–c) AFM and topography images of a p-type 6H-SiC nano wire on the graphite substrate, d) I–V curves at different applied forces across the nano wires, e) The relationship between the nano wire resistance and the applied forces, f–g) SEM and TEM images of p-type 6H-SiC nano wires. Reprinted with permission from [121].

2.11 a) SEM images of 3C-SiC NWS with released and non-released SiC NWs. b) A 5 fold-sensitivity enhancement with released NWs. Reprinted with permission from [127].

2.12 The Brillouin zone and carrier-electron distribution on the multi valley conduction band for the SiC(0001) nano sheet models: (a) 2H-SiC (0001) model and (b) 4H-, 6H-, and 3C-SiC (0001) models. The dashed and solid lines respectively represent the isotropic energy surfaces at the valleys for the strain-free and [1T00] tensile strain models. (c) Si_{12}C_{12}H_{2} unit super cells of three-dimensional periodic boundary models of the hexagonal 2H-, 4H-, 6H-, and 3C-SiC(0001) nanosheets. Courtesy of Nakamura et al. [132].

2.13 Variations in calculated longitudinal and transverse GFs with reciprocal of temperature of n-type 4H-, 6H-, and 3C-SiC (0001) nano-sheet models for the [1T00] tensile strain at 1×10^{19} cm^{-3} carrier concentration. Courtesy of Nakamura et al. [132].

2.14 Temperature dependence of the piezoresistive effect in 3C-SiC.

2.15 Requirements in various harsh environment applications. Reprinted with permission from [2].

2.16 a) Actual test fixture location. b) Test fixture for Si and SiC in-cylinder where SiC die are mounted to the tungsten disk with a high temperature ceramic adhesive. c) Silicon carbide sample, A-C: samples before engine testing; D-F: samples after 20, 40, and 60 mins. Courtesy of Wodin et al. [146].

2.17 High shock sensor with four longitudinal piezoresistors on the narrow beams and the four damping slots. Reprinted with permission from [147].

2.18 a) Schematic of a balanced mass double-ended tuning fork. b) Time-lapse photography of the shock event. c) Transient response curve of the sensor in dry steam. Reprinted with permission from [148].
3.1 a) First Brillouin zone of hexagonal SiC (e.g. 4H- and 6H-SiC) with high symmetry points in k-space. b) Representation of the constant energy surfaces for three highest valence bands in 4H-SiC. From top to bottom: Light hole, heavy hole, and spin orbit split-off (SOSO) bands, respectively. .................................................. 62

3.2 a) A general representation of two coordinates applied to a hexagonal crystal structure. b) Euler angles of the transformation from the principle coordinate to an arbitrary coordinate. ................. 67

3.3 a) Bending cantilever setup. b) 4H-SiC piezoresistor placed on top of cantilever. .................................................. 74

3.4 a) The bending beam mechanical model; b) Strain distribution in the cross-sectional of SiC beam. c) Strain transferred onto piezoresistors lying on the top surface of the beam. The strain induced is independent on the width of piezoresistors. ................................. 75

3.5 Characterization of the piezoresistive effect of 4H-SiC micro structure at high temperatures in a Linkam™hotplate. ......................... 76

3.6 4H-SiC wafer with two epitaxial layers on bulk SiC substrate ...... 77

3.7 Fabrication process of p-type 4H-SiC piezoresistors .................. 78

3.8 a) Laser scribing process after micromachining 4H-SiC. b) Alignment of laser scribing on the back side with respect to the pre-fabricated sensor on the front side. c) SEM image of the back side of the diaphragm after laser scribing. Scale bar, 100 µm. ................................. 80

3.9 a) Cross-sectional view of metallisation and SiC layers. b) I-V measurement of Ti/Al contact annealed up to 1000°C. ......................... 82

3.10 Current-voltage characteristics of the p-type piezoresistor with a voltage ranging from -5V to 5V at various temperatures from 23°C to 600°C, showing the excellent Ohmic characteristics of the Ti/Al contact to the p-type piezoresistors. Inset: Schematic sketch of the measurement of the current flowing in p-type layer and through the p-n junction. .......................................................... 83

3.11 Micro structures of SiC/metal interfaces of sample with Ti/Al electrodes before annealing (top) and after annealing (bottom). Reprinted with permission from [24]. ................................. 83
# List of Tables

2.1 Physical characteristics of silicon carbide and other wide band gap materials [2, 3, 7, 8] ................................................................. 12

2.2 Index conversion rule for the piezoresistance coefficient and the stress component subscripts \((ij \rightarrow i \text{ if } i = j \text{ and sum of three numbers equal to 9 if } i \neq j)\) ................................................................. 20

2.3 List of gauge factors (GF) of SiC reported in the literature ............ 27

2.4 Comparison of SiC based pressure sensors ................................. 37

3.1 Comparison of three arbitrary PZR coefficients in cubic lattices (e.g. Si, 3C-SiC) and a hexagonal lattice (i.e. 4H-SiC, this work) ............. 73

3.2 Fabrication process of 4H-SiC micro structures .......................... 79
## Abbreviations

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>AFM</td>
<td>Atomic Force Microscopy</td>
</tr>
<tr>
<td>APCVD</td>
<td>Atmospheric Pressure Chemical Vapor Deposition</td>
</tr>
<tr>
<td>CVD</td>
<td>Chemical Vapor Deposition</td>
</tr>
<tr>
<td>DFT</td>
<td>Density Functional Theory</td>
</tr>
<tr>
<td>DRIE</td>
<td>Deep Reactive Ion Etching</td>
</tr>
<tr>
<td>FEA</td>
<td>Finite Element Analysis</td>
</tr>
<tr>
<td>FIB</td>
<td>Focused Ion Beam</td>
</tr>
<tr>
<td>GF</td>
<td>Gauge Factor</td>
</tr>
<tr>
<td>HH</td>
<td>Heavy Hole</td>
</tr>
<tr>
<td>HMCVD</td>
<td>Hot Mesh Chemical Vapor Deposition</td>
</tr>
<tr>
<td>HTCVD</td>
<td>High Temperature Chemical Vapor Deposition</td>
</tr>
<tr>
<td>ICs</td>
<td>Integrated Circuits</td>
</tr>
<tr>
<td>ICP</td>
<td>Inductively Coupled Plasma</td>
</tr>
<tr>
<td>IPA</td>
<td>IsoPropyl Alcohol</td>
</tr>
<tr>
<td>LDA</td>
<td>Local Density Approximation</td>
</tr>
<tr>
<td>LH</td>
<td>Light Hole</td>
</tr>
<tr>
<td>LPCVD</td>
<td>Low Pressure Chemical Vapor Deposition</td>
</tr>
<tr>
<td>MEMS</td>
<td>Micro Electro Mechanical Systems</td>
</tr>
<tr>
<td>NEMS</td>
<td>Nano Electro Mechanical Systems</td>
</tr>
<tr>
<td>NW</td>
<td>Nano Wire</td>
</tr>
<tr>
<td>PECVD</td>
<td>Plasma Enhanced Chemical Vapor Deposition</td>
</tr>
<tr>
<td>PZR</td>
<td>Piezoresistive</td>
</tr>
<tr>
<td>RIE</td>
<td>Reactive Ion Etching</td>
</tr>
<tr>
<td>RTP</td>
<td>Rapid Thermal Annealing</td>
</tr>
<tr>
<td>SEG</td>
<td>Selective Epitaxy Growth</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Full Form</td>
</tr>
<tr>
<td>--------------</td>
<td>-----------</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscopy</td>
</tr>
<tr>
<td>SiC</td>
<td>Silicon Carbide</td>
</tr>
<tr>
<td>SHM</td>
<td>Structural Health Monitoring</td>
</tr>
<tr>
<td>SOI</td>
<td>Silicon On Insulator</td>
</tr>
<tr>
<td>SOSO</td>
<td>Spin Orbit Split Off</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission Electron Microscopy</td>
</tr>
</tbody>
</table>
## Physical Constants

<table>
<thead>
<tr>
<th>Constant</th>
<th>Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boltzmann constant $k_B$</td>
<td>$2.38 \times 10^{-23}$</td>
<td>$\text{m}^2\text{s}^{-2}\text{K}^{-1}$</td>
</tr>
<tr>
<td>Electron charge $e$</td>
<td>$1.602 \times 10^{-19}$</td>
<td>$\text{C}$</td>
</tr>
<tr>
<td>Electron mass $m_0$</td>
<td>$9.11 \times 10^{-31}$</td>
<td>$\text{kg}$</td>
</tr>
<tr>
<td>Planck constant $h$</td>
<td>$6.626 \times 10^{-34}$</td>
<td>$\text{m}^2\text{kgs}^{-1}$</td>
</tr>
<tr>
<td>Reduced Planck constant $\hbar$</td>
<td>$1.054 \times 10^{-34}$</td>
<td>$\text{m}^2\text{kgs}^{-1}$</td>
</tr>
</tbody>
</table>
## Symbols

<table>
<thead>
<tr>
<th>Notation</th>
<th>Meaning</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>$J$</td>
<td>electric current density</td>
<td>[Am$^{-2}$]</td>
</tr>
<tr>
<td>$\rho$</td>
<td>resistivity</td>
<td>[$\Omega m$]</td>
</tr>
<tr>
<td>$R$</td>
<td>resistance</td>
<td>[$\Omega$]</td>
</tr>
<tr>
<td>$\Delta R/R$</td>
<td>relative resistance change</td>
<td>[-]</td>
</tr>
<tr>
<td>$\mu_h$</td>
<td>hole mobility</td>
<td>[ms$^{-1}$]</td>
</tr>
<tr>
<td>$\mu_e$</td>
<td>electron mobility</td>
<td>[ms$^{-1}$]</td>
</tr>
<tr>
<td>$n_h$</td>
<td>hole concentration</td>
<td>[cm$^{-3}$]</td>
</tr>
<tr>
<td>$n_e$</td>
<td>electron concentration</td>
<td>[cm$^{-3}$]</td>
</tr>
<tr>
<td>$\nu$</td>
<td>Poisson's ratio</td>
<td>[-]</td>
</tr>
<tr>
<td>$\lambda$</td>
<td>electrical conductivity</td>
<td>[Sm$^{-1}$]</td>
</tr>
<tr>
<td>$m^*$</td>
<td>electron effective mass</td>
<td>[kg]</td>
</tr>
<tr>
<td>$v$</td>
<td>electron velocity</td>
<td>[ms$^{-1}$]</td>
</tr>
<tr>
<td>$k_x, k_y, k_z$</td>
<td>vectors in $k$-space</td>
<td>[-]</td>
</tr>
<tr>
<td>$N_v$</td>
<td>hole effective density of state</td>
<td>[-]</td>
</tr>
<tr>
<td>$F_{\pm1/2}$</td>
<td>Fermi–Dirac integral of order $\pm1/2$</td>
<td>[-]</td>
</tr>
<tr>
<td>$p_i$</td>
<td>number of holes per unit volume</td>
<td>[-]</td>
</tr>
<tr>
<td>$m_i$</td>
<td>effective mass of light hole or heavy hole</td>
<td>[kg]</td>
</tr>
<tr>
<td>$E_F$</td>
<td>Fermi energy</td>
<td>[eV]</td>
</tr>
<tr>
<td>$G_l$</td>
<td>longitudinal gauge factor</td>
<td>[-]</td>
</tr>
<tr>
<td>$G_t$</td>
<td>transverse gauge factor</td>
<td>[-]</td>
</tr>
<tr>
<td>$G_s$</td>
<td>shear gauge factor</td>
<td>[-]</td>
</tr>
<tr>
<td>$\varepsilon$</td>
<td>strain</td>
<td>[-]</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>stress</td>
<td>[MPa]</td>
</tr>
<tr>
<td>Symbol</td>
<td>Description</td>
<td>Unit</td>
</tr>
<tr>
<td>--------</td>
<td>-------------</td>
<td>------</td>
</tr>
<tr>
<td>$\Psi$</td>
<td>electrical field</td>
<td>[V]</td>
</tr>
<tr>
<td>$V(r)$</td>
<td>potential energy</td>
<td>[V]</td>
</tr>
<tr>
<td>$\phi_k(r)$</td>
<td>wave function</td>
<td>[-]</td>
</tr>
<tr>
<td>$E$</td>
<td>band energy</td>
<td>[eV]</td>
</tr>
<tr>
<td>$T$</td>
<td>temperature</td>
<td>[K]</td>
</tr>
<tr>
<td>$\pi$</td>
<td>piezoresistive coefficient tensor in principal coordinate</td>
<td>[-]</td>
</tr>
<tr>
<td>$\pi_{ij}$</td>
<td>piezoresistive coefficients in principal coordinate</td>
<td>[Pa$^{-1}$]</td>
</tr>
<tr>
<td>$\pi'$</td>
<td>piezoresistive coefficient tensor in arbitrary coordinate</td>
<td>[-]</td>
</tr>
<tr>
<td>$\pi'_{ij}$</td>
<td>piezoresistive coefficients in arbitrary coordinate</td>
<td>[Pa$^{-1}$]</td>
</tr>
</tbody>
</table>
To my lovely wife
Chapter 1

Introduction and research scopes

1.1 Introduction and research objectives

Micro Electro Mechanical Systems (MEMS) sensors have been extensively studied and developed for a wide range of applications thanks to the superior miniaturisation and integration capabilities \[1–3\]. Over the past five decades, Si and polymers have remained the most common materials for MEMS sensors owing to their wide availability and highly developed fabrication process. However, the intrinsic physical properties of these materials (e.g. the low energy band gap and plastic deformation at high temperatures) hinder the use of Si/polymer-based devices in harsh environment applications \[2, 4, 5\].

Harsh environments involve high temperature, high pressure, strong electric fields, extremely high shock, intense vibration and aggressive chemical conditions which occur in structural health monitoring (SHM), process control, oil/gas exploration, aerospace industries, and space exploration. Figure 1.1 presents harsh environment applications with associated technical requirements. Those applications demand the investigation and development of advanced materials which are not only highly compatible with those conditions but are able to adapt to the mature technology based on the silicon material. The current research on MEMS sensors has paid a great deal of attention to wide band gap materials such as zinc oxide (ZnO), diamond-like carbon (DLC), aluminium nitride (AlN), gallium nitride (GaN) and silicon carbide (SiC)
### Chapter 1: Introduction and research scopes

#### Figure 1.1: Technical requirements for sensors used in harsh environments. (Courtesy of Albert P. Pisano, University of California, San Diego)

<table>
<thead>
<tr>
<th>Applications</th>
<th>Oil/gas exploration</th>
<th>Automotive engines</th>
<th>Geothermal</th>
<th>Aircraft engines</th>
<th>Space exploration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Required temperatures</td>
<td>![Image] 275°C</td>
<td>![Image] 300°C</td>
<td>![Image] 375°C</td>
<td>![Image] 600°C</td>
<td>&gt;1000°C</td>
</tr>
<tr>
<td>for sensors</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Desired sensing parameters</th>
<th>Temperature</th>
<th>Pressure</th>
<th>Hydrocarbon</th>
<th>Flame speed</th>
<th>O₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oil/gas exploration</td>
<td>Temperature</td>
<td>Pressure</td>
<td>Hydrocarbon</td>
<td>Flame speed</td>
<td>O₂</td>
</tr>
<tr>
<td>Automotive engines</td>
<td>Temperature</td>
<td>Pressure</td>
<td>H₂S</td>
<td>Flame speed</td>
<td>O₂</td>
</tr>
<tr>
<td>Geothermal</td>
<td>Temperature</td>
<td>Pressure</td>
<td>H₂S</td>
<td>Strain</td>
<td></td>
</tr>
<tr>
<td>Aircraft engines</td>
<td>Temperature</td>
<td>Pressure</td>
<td>H₂S</td>
<td>Flame speed</td>
<td>O₂</td>
</tr>
<tr>
<td>Space exploration</td>
<td>Temperature</td>
<td>Pressure</td>
<td>Radiation</td>
<td>Strain</td>
<td></td>
</tr>
</tbody>
</table>

[6–10]. In terms of mechanical sensing, SiC is positioned as a promising candidate owing to the low dielectric constant (low noise), low leakage current, high thermal conductivity, and chemical inertness. The superior properties of SiC along with its stability at high temperatures offer new possibilities for developing MEMS devices for such challenging applications.

SiC exists in many different crystal structures, which are called polytypes. There are more than 200 SiC polytypes, but the majority of studies and developments have focused on three types: 3C-, 4H-, and 6H-SiC. Among these, the 4H-SiC polytype is more favourable for MEMS devices owing to its excellent properties [11]. The energy band gap of 4H-SiC is 3.23 eV, which is considerably higher than that of 3C (2.3 eV), 6H (3.0 eV) and Si (1.12 eV). The high potential barrier in SiC materials can effectively minimize the number of electron-hole pairs generated in high temperatures across the band gap, enabling the high temperature stability of SiC electronic devices and sensors [11–16].

The piezoresistive effect is one of the most significant sensing mechanisms used in MEMS/NEMS sensors. This sensing effect has been widely employed to detect mechanical signals, such as pressure, inertia, acceleration and deflection etc., enabling the miniaturization and integration capabilities of MEMS devices with low power requirements and simple read-out circuits. Studies on the piezoresistive effect of SiC based sensors at elevated temperatures have shown that the effect is relatively stable.
up to 400°C [17–27]. Although investigations of the piezoresistive effect in SiC have been intensively carried out for 3C-SiC and in a few studies of 6H-SiC, the piezoresistive effect in p-type 4H-SiC has not yet been elucidated. Being as all-SiC wafers, the use of 4H-SiC eliminates the thermal expansion mismatch between the sensing layer and the substrate, which exists in 3C-SiC, extending the working regime of the sensing devices. Moreover, 4H-SiC possesses the highest energy band gap among SiC polytypes, which potentially offers the most stable performances in high temperature operations. Since 4H-SiC is a wide band gap semiconductor, there exists a large difference of the work functions of metals and 4H-SiC, leading to a high Schottky barrier formed between the p-type and metal layers. This obstacle partly limits the understanding of the piezoresistive effect in p-type 4H-SiC and hinders the developments of all-SiC devices which can effectively work in extreme conditions. Therefore, this thesis initially aims at developing metal/SiC Ohmic contacts, which enables the electrical measurements for the characterisation of the piezoresistive effect in 4H-SiC. Subsequently, theoretical and experimental investigations of the piezoresistive effect in p-type 4H-SiC at room and high temperatures is conducted, utilising a simple but effective method (i.e. bending beam method). The success of this characterisation will demonstrate the potential for applications where the extreme temperature conditions are imperative. Furthermore, the device development is also implemented and demonstrated, including a highly sensitive van der Pauw strain sensor and a pressure sensor at cryogenic and elevated temperatures which utilised a top-down fabrication (i.e. laser engraving).

The main objectives of this thesis are

(i) Investigation of the piezoresistive effect and the fundamental piezoresistive coefficients of p-type 4H-SiC by discovering the physics underlying the effect and conducting experimental characterisations. The SiC/metal Ohmic contact is initially developed, enabling the electrical measurements.

(ii) Investigation of the temperature dependence of the piezoresistive effect in p-type 4H-SiC as well as the stability, linearity and reproducibility at high temperatures.

(iii) Fabrication and demonstration of micromachined p-type 4H-SiC based-devices for mechanical sensing devices for harsh conditions utilising the piezoresistive effect,
including van der Pauw devices and pressure sensors.

1.2 Research scope

In this thesis, the piezoresistive effect of p-type 4H-SiC and its piezoresistive coefficients in the (0001) plane are investigated. To further explore the capability in harsh environments, the piezoresistive effect at high temperatures of up to 600°C is also studied. Another important objective of this thesis is to demonstrate the device integration capability by developing 4H-SiC based sensing devices using the piezoresistive effect as the sensing mechanism.

1.3 Thesis structure

This thesis is organised into seven chapters as follows

Chapter 1 introduces the research objectives and the scope of this thesis.

Chapter 2 provides a thorough review on the SiC material processing, the piezoresistive effect in semiconductors and SiC polytypes as well as state-of-the-art SiC based mechanical sensing devices.

Chapter 3 presents the research methodology including theoretical and experimental methods. The theoretical analysis of the piezoresistive effect in p-type 4H-SiC is based on the hole transfer mechanism and the coordinate transformation. Subsequently, the micromachining processes to fabricate SiC micro structures are presented. The metallisation process to obtain Ohmic contact will also be carried out with high temperature annealing. The content of this chapter has been partly presented in published papers J.22, J.23, J.24, J.25 and J.26.

Chapter 4 presents the piezoresistive effect and the isotropic piezoresistance in the (0001) plane in p-type 4H-SiC. The obtained large gauge factors and the linear relationship between the resistance change versus induced strain demonstrate the potential of p-type 4H-SiC for mechanical sensing applications. The isotropy of the piezoresistance in the (0001) plane of p-type 4H-SiC is discovered, which is correlated
Chapter 1 Introduction and research scopes

to the isotropic hole energy shift under uniaxial strain. This interesting behaviour of the piezoresistive effect in p-type 4H-SiC is favourable for the design and fabrication of mechanical sensors since high sensitivity can be achieved regardless of the crystallographic orientation. The content of this chapter is presented in forms of: Published paper (J.22) – “Experimental investigation of piezoresistive effect in p-type 4H-SiC” & Published paper (J.24) – “Isotropic piezoresistance of p-type 4H-SiC in (0001) plane”.

Chapter 5 presents the temperature dependence of the piezoresistive effect in p-type 4H-SiC. The piezoresistive effect exhibits good linearity and high stability at high temperatures. This finding demonstrates the capability of p-type 4H-SiC for mechanical sensing in harsh environments. The content of this chapter is presented in forms of: Published paper (J.26) – “High-temperature tolerance of piezoresistive effect in p-4H-SiC for harsh environment sensing”.

Chapter 6 presents a p-type 4H-SiC van der Pauw strain sensor and a 4H-SiC piezoresistive pressure sensor utilising a laser scribing approach. The van der Pauw sensor exhibits an excellent repeatability and linearity with a significantly large output voltage, while the pressure sensor is able to operate at a temperature range from cryogenic to elevated temperatures with an excellent linearity and repeatability. The high sensitivity and good reliability at cryogenic and elevated temperatures are attributed to the profound piezoresistive effect in p-type 4H-SiC and the robust p-n junction, which prevents the current from leaking to the substrate. The content of this chapter is presented in forms of: Published paper (J.23) – “Highly sensitive p-type 4H-SiC van der Pauw strain sensor” & Published paper (J.25) – “Highly sensitive 4H-SiC pressure sensor at cryogenic and elevated temperatures”.

Chapter 7 draws the conclusion and discusses the future work and outlook.

The thesis structure is shown in Fig. 1.2.
Chapter 1 Introduction and research scopes

Research objectives
Piezoresistive effect in p-type 4H-SiC for mechanical sensing at high temperatures

Literature review
(1) Piezoresistive effect in semiconductors
(2) Piezoresistive effect in SiC
(3) Material aspects of SiC devices
(4) SiC based sensing devices utilising the piezoresistive effect for harsh environment applications

Research gap
(1) Limited understanding on piezoresistive effect of p-type 4H-SiC at room temperature and high temperature
(2) Limited 4H-SiC sensing devices due to difficulties in formation of good Ohmic contact and material processing

Theoretical analysis
(1) Piezoresistive effect of p-type 4H-SiC
(2) Piezoresistance coefficients in the (0001) plane using the coordinate transformation and analysis of strain induced effect in the crystal structure
(3) Methods for applying strain to thin films
(4) Temperature dependence of the piezoresistive effect of p-type 4H-SiC micro structures

Sample preparation
(1) Fabrication of p-type 4H-SiC micro structures: piezoresistors, four-terminal devices and pressure sensors using ICP etching
(2) Ohmic contact formation: metalisation and thermal annealing at high temperatures.
(3) Formation of 4H-SiC diaphragm using laser scribing
(4) Measurement setup for PZR effect measurement at high temperatures.

Characterization & results
(1) Piezoresistive effect of p-type 4H-SiC
(2) Piezoresistance coefficients of p-type 4H-SiC
(3) Temperature dependence of piezoresistive effect in p-type 4H-SiC
(4) 4H-SiC van der Pauw sensor utilising the piezoresistive effect
(5) 4H-SiC pressure sensor using laser scribing

Outcome
- Paper J.22 (EDL)
- Paper J.24 (APL)
- Paper J.26 (JMC)
- Paper J.23 (RSC)
- Paper J.25 (M&D)

Data analysis and evaluation
Dissertation

Figure 1.2: Flow chart of the thesis structure.
## Bibliography


Chapter 1 Introduction and research scopes


Chapter 1 Introduction and research scopes


Chapter 2

Research background and literature review

This chapter reviews the state-of-the-art investigations and applications of the piezoresistive effect in semiconductors and SiC materials. The research background of the SiC materials, analysis and physics of the piezoresistive effect are presented. Subsequently, studies on SiC piezoresistive effect and SiC based sensing devices are also outlined and discussed.

2.1 SiC material aspects

2.1.1 Crystallography of SiC

SiC exists in many different crystal structures, which are called polytypes. Amongst more than 200 polytypes, three most common SiC polytypes which are being developed for electronics and sensing devices, include cubic 3C-SiC(\(\beta\)-SiC), hexagonal 4H-SiC and 6H-SiC (\(\alpha\)-SiC), and rhombohedral 15R-SiC. A comprehensive introduction to SiC crystallography and polytypism can be found in [1–3]. The SiC polytypes can be distinguished by the stacking sequence of the Si-C bilayer and the orientation of adjacent layers [4]. Figure 2.2 shows the different planar arrangements of A, B and C, then ABCABC, ABCBA, CABCBA represent the stacking sequences of 3C, 4H and 6H-SiC, respectively. In the crystal structures of all SiC polytypes, each silicon
(or carbon) atom is bonded with four adjacent carbon atoms (or silicon atoms) in a tetrahedral structure, the distances between Si/C and C/C atoms are 1.89 Å and 3.08 Å, respectively. The Si-C pairs are placed in hexagonal bilayers where Si and C atoms alternately compose sub layers [2, 5].

![Si–C bonding in SiC](image)

**Figure 2.1:** Si–C bonding in SiC

![Crystal structure of three most common SiC polytypes](image)

**Figure 2.2:** Crystal structure of three most common SiC polytypes: a) cubic 3C-SiC (β-SiC), b) hexagonal 4H-SiC, c) hexagonal 6H-SiC. Reprinted with permission from [2].

### 2.1.2 Physical properties of SiC

Table 2.1 compares the fundamental properties of single crystalline SiC polytypes, including 3C-SiC, 4H-SiC, and 6H-SiC, with Si and other wide band gap materials such as GaN and diamond. It can be seen that SiC polytypes are wide energy band gap materials (e.g. 2.3 eV for 3C-SiC, 3.0 eV for 6H-SiC, and especially 3.4 eV for...
4H-SiC) with high carrier mobility and high breakdown voltage. SiC possesses a Mohs hardness of 9, and a wear resistance of 9.1, which are slightly below those of diamond, the hardest material [3]. The sublimed and melting temperatures of SiC are about 1800°C and 2830°C, respectively, which are significantly higher than those of Si. Comparing with gallium nitride (GaN), which is also widely used for MEMS applications, 4H-SiC possess equivalent energy band gap and break-down voltage (3.2 eV/2.4 MV/cm² vs. 3.4 eV/3.0 MV/cm²), higher electron mobility (1000 cm²/Vs⁻¹ vs. 900 cm²/Vs⁻¹), and significantly larger thermal conductivity and Young’s modulus (4.9 Wcm⁻¹K⁻¹/500 GPa vs. 1.3 Wcm⁻¹K⁻¹/300 GPa). Additionally, 4H-SiC has much higher chemical/oxidation resistance than that of GaN, which is crucial for devices working in harsh environments. SiC materials are also highly resistant and not attacked by most acids. Additionally, the good compatibility with micromachining processes of SiC enables the development of miniaturised SiC based MEMS devices. These promising properties positions SiC a good candidate for MEMS mechanical sensors in harsh environments [9, 10].

### 2.1.3 Growth process of SiC

The formation of SiC involves the reactions between Si and C atoms in specific conditions such as the deposition temperatures for amorphous SiC, polycrystalline SiC, and single crystallise SiC are approximately 700°C, 800°C, above 1000°C, respectively. Many deposition methods have been proposed and developed to grow good quality SiC films for a wide range of applications. As a result, single crystalline,
polycrystalline and amorphous SiC thin films can be manufactured and tailored to meet specific requirements. A comprehensive review of SiC growth processes can be found elsewhere [11].

![Image](image.png)

**Figure 2.3:** The current status of SiC growth process available. Reprinted with permission from [2].

### 2.1.3.1 Vapour phase growth

SiC substrates are commonly grown by vapour phase in the sublimation of SiC at temperature above 1800°C. As a result, various elemental and molecular species are also formed apart from SiC sublimation from solid to gas phases, because of the dissociation of SiC (Eq. 2.1) [12].

\[
\begin{align*}
\text{SiC}(\text{solid}) & \rightarrow \text{Si}(\text{gas}) + \text{C}(\text{solid}) \\
2\text{SiC}(\text{solid}) & \rightarrow \text{Si}_2\text{C}_2(\text{gas}) + \text{Si}(\text{gas}) \\
2\text{SiC}(\text{solid}) & \rightarrow \text{C}(\text{solid}) + \text{Si}_2\text{C}(\text{gas})
\end{align*}
\]

**a. Atmospheric pressure chemical vapour deposition**

Atmospheric pressure chemical vapour deposition (APCVD) was the first SiC deposition approach to epitaxially grow single crystalline and polycrystalline SiC films. While single crystalline SiC can only be grown on Si, polycrystalline SiC films are able to be deposited on various substrates (e.g. Si\textsubscript{3}N\textsubscript{4}, SiO\textsubscript{2}). A detailed growth process
for APCVD can be found elsewhere [3]. A significant advantage of this method is that since the APCVD systems requires less temperature sensitive components than low pressure configurations, the temperatures of the substrate can easily be kept at as high as 1300°C with relatively low expenses. This method has been widely utilised for the growth of 3C- and 6H-SiC.

b. High temperature chemical vapour deposition

High temperature chemical vapour deposition (HTCVD) was introduced and extensively developed for bulk-SiC growth [12]. With significant improvements, this method is widely utilised to manufacture commercial SiC wafers. In HTCVD, silicon and carbon precursors are used, commonly silane (SiH$_4$) (Si source) and methane or ethane or propane (C sources). The precursors are kept in coaxial tubes in which the silicon precursor is contained in the most inner-tube. In order to facilitate the dissociation of silane and formation of Si clusters, high flow rate and high pressure are maintained. Subsequently, the Si clusters react with the hydrocarbon precursor, forming Si$_x$C$_y$ clusters. The sublimation of Si$_x$C$_y$ clusters forms Si and C containing species prior to reaching the seed crystal. There are several advantages of this method over the APCVD method. As such, the Si/C ratio can be directly controlled by supplying the precursors gases flow rates. Another advantage is to grow p-type substrates with precise dopant control [13]. However, further improvements are required to grow of large-scale wafers for the commercial perspective.

c. Plasma enhanced chemical vapour deposition

Plasma enhanced chemical vapour deposition (PECVD) allows the deposition of SiC films on numerous substrates as well as lowers the required deposition temperatures which are typically in between 200 to 400°C. PECVD normally produces amorphous SiC, but the films can be crystallized by annealing. A significant issue emerging in the PECVD is the existence of high residual stresses SiC films which highly depends on the deposition parameters. The film stresses can be either compressive or tensile [14]. However, the annealing process can reduce the film stresses. The PECVD SiC layers are typically utilised as coating materials which can protect the function layers from volatile chemical substances.

d. Low pressure chemical vapour deposition
LPCVD is widely employed to manufacture high quality SiC thin films including $\beta$-SiC and poly-SiC with various precursors. Thanks to the low pressure used in the growth process, the film uniformity is ensured in large-scale wafers which allows mass productions of SiC devices. However, slower growth rates of LPCVD in comparison to that of APCVD is needed to be addressed. LPCVD were typically conducted at relatively high temperatures (e.g. above 1000$^\circ$C) to improve the quality of SiC films, using either hot-wall or cold-wall reactors with the heating sources from induction or resistive heating [15].

### 2.1.3.2 Solution phase growth

A number of efforts have utilised the solubility of SiC and C in melting Si to grow SiC from high temperature solutions. In this approach, the growth of SiC occurred in the liquid phase by solutions containing Si and C [16]. The recent interests of solution phase growth of SiC are to grow SiC crystals with low dislocation densities. It was found that at conditions near to the thermal equilibrium of solutions, high quality crystals can be achieved in the growth process with controllable polytypes [17]. A significant advantage of this method is the low temperature requirement which is suitable for the 3C-SiC polytype.

### 2.2 Piezoresistive effect

#### 2.2.1 Piezoresistive effect in semiconductors

Piezoresistivity is a property of solid materials that change their electrical resistance with elongation by external strain. The effect has been utilised in a vast number of devices for mechanical sensing applications owing to the miniaturization, low power consumption, and the simple read-out circuitry requirements [18–27]. Piezoresistive sensors possess several advantages in comparison to capacitive counterparts such as the easiness of design configuration and the wider linearity range [15, 28–36]. Over the past four decades, Si remains the most important material used for pressure sensors owing to its wide availability and mature fabrication process. However, its intrinsic physical properties, such as the low energy band gap and plastic deformation,
have limited its usage in harsh environments. In contrast, the use of wide band gap materials (e.g. SiC, GaN, aluminium nitride (AlN), diamond like carbon (DLC), and zinc selenide (ZnSe)) for mechanical sensors can eliminate the thermal expansion mismatch between the sensing layer and the substrate and extend the working regime of the sensing devices [8, 37–44]. Moreover, the high energy band gap significantly reduces the number of thermal activated electron-hole pairs, improving the reliability and stability of sensing devices in extreme conditions.

The superior mechanical strength, excellent corrosive/shock resistance and high stability at high temperatures position silicon carbide a promising material for extreme condition sensing. In fact, numerous SiC based mechanical sensors have been reported including capacitive, piezoelectric, and piezoresistive sensors [45–58].

The piezoresistive effect in metals was introduced in the middle of the nineteenth century by Thomson and Kelvin, followed by a vast number of studies. In the 1930s, Allen discovered this property in single crystals of bismuth, antimony, cadmium, zinc and tin [59–62]. However, the applications of the piezoresistive effect in metals are very limited due to the fact that their resistance changes are insignificant. In 1950, Bardeen and Shockley proposed the first theoretical model to explain the large resistivity change using the deformation of the crystal structure [63], followed by the discovery of Smith of a high piezoresistive shear coefficient in silicon and germanium [64]. The semiconductor strain gauges, in which the sensitivity has been found to be two or three orders of magnitude higher than that of metals, were employed for highly sensitive mechanical sensing including displacement, force, and torque measurements [65].

Large-scale manufacturing of piezoresistive based sensors were then further developed along with the rapid advances in micro fabrication technologies [66]. Tremendous developments in the miniaturisation of piezoresistive devices enabled the integration of piezoresistive elements within sensing objects [67, 68]. Subsequently, piezoresistive sensors were commercially manufactured in the 1980’s. Moreover, advances in the fabrication of integrated circuits (ICs), such as doping, etching, and depositing processes, have greatly enhanced the sensitivity, resolution, and miniaturization of piezoresistive based MEMS devices.
2.2.2 Theoretical analysis of piezoresistive effect

The piezoresistive effect is positioned as the main transduction mechanism for mechanical sensing such as strain/stress, pressure, and acceleration sensors, in which significant variations of the electrical resistance can be recorded when applying a bias voltage or a current through the piezoresistor under strain. The resistance varies with the application of mechanical loads, which induces stress/strain onto resistors. The basic equation for the resistance of a wire (assuming a cylindrical wire for simplicity) is given as \[ R = \rho \frac{L}{A} \] (2.2)

where \( \rho \) is the resistivity of the wire material, \( L \) is the wire’s length, and \( A \) is the cross-sectional area. From Eq. 2.2, one can obtain

\[
\frac{\partial R}{R} = \frac{\partial L}{L} + \frac{\partial \rho}{\rho} + \frac{\rho}{A} \frac{\partial A}{A} - \frac{\rho L}{A^2} \frac{\partial A}{A^2} \] (2.3)

Dividing by Eq. 2.2, we have

\[
\frac{\partial R}{R} = \frac{\partial L}{L} + \frac{\partial \rho}{\rho} - \frac{\partial A}{A} \] (2.4)

with a note that \( \frac{\partial A}{A} = 2 \frac{\partial D}{D} \) and \( \frac{\partial D}{D} = -\nu \left( \frac{\partial L}{L} \right) \), where \( D \) is the wire’s diameter and \( \nu \) is the Poisson’s ratio of the wire material. Substituting these into the Eq. 2.4, the variation of the resistance, which depends on the dimensions and the resistivity changes, is derived as

\[
\frac{\partial R}{R} = \varepsilon_l (1 + 2\nu) + \frac{\partial \rho}{\rho} \] (2.5)

where \( \varepsilon_l = \frac{\partial L}{L} \) is the longitudinal strain induced to the resistor. The piezoresistive effect can be represented by the gauge factor (GF), which is defined by the fractional change in the resistance per unit strain \[70, 71\]

\[
GF = \frac{\partial R}{R} \frac{1}{\varepsilon_l} = (1 + 2\nu) + \frac{1}{\varepsilon_l} \frac{\partial \rho}{\rho} \] (2.6)
The geometric factor ($\nu$) and the fractional change in resistivity ($\Delta \rho/\rho$) contribute to the variation of the resistance of materials with an applied strain. Most solid materials have Poisson’s ratios $\nu$ around 0-0.5 [72], while the resistivity’s variation of metals under strain is negligible [70]. In semiconductor materials (e.g. Si), the geometric factor provides a GF of approximately 1 to 2, whereas the change of resistivity is a hundred of times larger. Since the Poisson’s ratio of semiconductors is typically below 1, it can be concluded that the GF is mainly attributed to the variation of the resistivity due to the stress-induced phenomenon. Subsequently, Eq. 2.6 can be expressed as

$$\text{GF} = \frac{1}{\varepsilon} \frac{\partial \rho}{\rho} = -\frac{1}{\varepsilon} \frac{\partial \lambda}{\lambda} \quad (2.7)$$

where $\rho$ and $\lambda$ are the resistivity and conductivity of semiconductor materials varying with induced stress/strain, respectively. This phenomenon is correlated to the variation of the carrier density and mobility, leading to a dramatic change of the resistivity or conductivity. In semiconductors, the piezoresistivity exhibits a large anisotropy, which depends on the stress/strain direction and the field directions (e.g. voltages and currents). Typically, the piezoresistivities of p-type and n-type show opposite trends in terms of the variation in the resistance magnitude under stress/strain. There are various factors that regulate the magnitude of the piezoresistive effect such as the carrier concentration, ambient temperature, crystallographic orientation, and the direction of applied voltage/current versus stress/strain.

### 2.2.2.1 Tensor representation of piezoresistive effect

**Resistivity tensor**

In single crystal materials (e.g. silicon, silicon carbide), the relationship between electric field $E$ and electric current density $J$ is given as [73]

$$E = \rho J \quad (2.8)$$
where $\rho$ is resistivity tensors. In the Cartesian coordinate system, we have

$$
\begin{bmatrix}
E_x \\
E_y \\
E_z
\end{bmatrix} =
\begin{bmatrix}
\rho_{xx} & \rho_{xy} & \rho_{xz} \\
\rho_{yx} & \rho_{yy} & \rho_{yz} \\
\rho_{zx} & \rho_{zy} & \rho_{zz}
\end{bmatrix}
\begin{bmatrix}
J_x \\
J_y \\
J_z
\end{bmatrix}
$$

(2.9)

The resistivity tensors is symmetrical (i.e. $\rho_{ij} = \rho_{ji}$), thus there are only six independent components including $\rho_{xx}$, $\rho_{yy}$, $\rho_{zz}$, $\rho_{yz}$, $\rho_{zx}$, and $\rho_{xy}$.

Piezoresistive coefficient tensor

From the theory of elasticity, the stress tensor is a second rank tensor and symmetrical, given as

$$
\sigma =
\begin{bmatrix}
\sigma_{xx} & \sigma_{xy} & \sigma_{xz} \\
\sigma_{yx} & \sigma_{yy} & \sigma_{yz} \\
\sigma_{zx} & \sigma_{zy} & \sigma_{zz}
\end{bmatrix}
$$

(2.10)

where $\sigma_{ii}$ denotes the stress normal to the principle faces, whereas $\sigma_{ij}$ ($i \neq j$) indicates shear stresses on these faces, as shown in Fig. 2.4. In general, the relationship between stress versus strain induced is [70]

$$
\sigma_{ij} = C_{ijkl} \times \varepsilon_{kl}
$$

(2.11)

where $C_{ijkl}$ is the fourth-rank elastic stiffness tensor, with its inverse matrix $S_{ijkl}$ is the compliance tensor.
Chapter 2 Research background and literature review

Table 2.2: Index conversion rule for the piezoresistance coefficient and the stress component subscripts ($ij \rightarrow i$ if $i = j$ and sum of three numbers equal to 9 if $i \neq j$)

<table>
<thead>
<tr>
<th>Tensor indices</th>
<th>11</th>
<th>22</th>
<th>33</th>
<th>23&amp;32</th>
<th>13&amp;31</th>
<th>12&amp;21</th>
</tr>
</thead>
<tbody>
<tr>
<td>Simplified indices</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
</tr>
</tbody>
</table>

The relationship of the resistivity tensor and the stress tensor can be given

$$
\frac{\delta \rho_{ij}}{\rho_{ij}} = \pi_{ijkl} \times \sigma_{kl}
$$

(2.12)

where $\pi_{ijkl}$ is the fourth-rank piezoresistive coefficient tensor.

![Figure 2.5: Two common configurations for the piezoresistive effect a) two-terminal ($E \parallel J$) and b) four-terminal ($E \perp J$)](image)

Each piezoresistive component $\pi_{ijkl}$ has four subscripts which related two second-rank stress and resistivity tensors. The first subscript $i$ denotes the electric field component, the second $j$ relates to the current density, and $k$ and $l$ subscripts refers to the stress components. According to Mason [65, 74], the four subscripts can be simplified as $1111 \rightarrow 11$, $1122 \rightarrow 12$, $2323 \rightarrow 44$ etc. (the completed index conversion can be found in Table 2.2). Therefore, the number of independent components in the piezoresistive coefficient tensor can be deducted to 36. The number of independent piezoresistive coefficients can be further reduced by applying the crystal symmetry [75]. Subsequently, in the case of a fixed direction electric filed and a varying-orientation current, a general equation for the relative resistance change versus the stress tensor is given as [75–77]

$$
\frac{\Delta \rho(x)}{\rho} = \sum_{i=1}^{6} \pi_{(x)i} \sigma_i
$$

(2.13)
where $\chi = 1$ ($ij = 11$) or 6 ($ij = 12$) represents the electric field parallel (two-terminal configurations) or perpendicular to the current flow (four-terminal configurations). The Eq. 2.13 is very useful to determine the anisotropic variation of resistance versus applied stress. In micromachined sensing devices, a thin film is typically used so that the out-of-plane stress components (i.e. $\sigma_3$, $\sigma_4$, and $\sigma_5$) will be vanished. Thus, Eq. 2.13 can be further simplified into

$$\frac{\Delta \rho(1)}{\rho} = \pi_{11}\sigma_1 + \pi_{12}\sigma_2 + \pi_{16}\sigma_6$$
$$\frac{\Delta \rho(6)}{\rho} = \pi_{61}\sigma_1 + \pi_{62}\sigma_2 + \pi_{66}\sigma_6$$

(2.14)

where $\pi_{11}$, $\pi_{12}$, and $\pi_{16}$ are the longitudinal, transverse, and shear piezoresistive coefficients in two-terminal configurations, respectively; while $\pi_{61}$, $\pi_{62}$, and $\pi_{66}$ are the longitudinal, transverse, and shear piezoresistive coefficients in four-terminal configurations, respectively. In four-terminal devices, the fraction $\Delta \rho(6)/\rho = V_{out}/V_{in}$ implies a pseudo resistance change; therefore four-terminal configurations are sometimes called the “pseudo-Hall” devices.

2.2.2.2 Physics of piezoresistive effect

In 1960, Keynes proposed an elastic strain model to explain the change of the electrical conductance (and resistivity) of solid-state materials [78]. The piezoresistive effect in semiconductors was subsequently explained by the electron transfer phenomenon occurring in the energy band structure of a semiconductor by Smith [64]. The stress accounted for the change in the number of charge carriers by shifting the energy band gap. Bardeen and Shockley developed the deformation potentials and mobilities theory of the crystal structure under strain. In this model, the energy band is modified by stress, leading to the re-distribution of the charge carriers across the energy bands. The difference in the charge carrier effective masses greatly changes the carrier mobility and conductivity of non-polar crystals (e.g. silicon, germanium and tellurium) [63]. This finding inspired by many subsequent studies to explain the piezoresistivity of semiconductor materials [79–82]. The piezoresistance in p-type silicon was calculated using k-p Hamiltonian model in the relaxation-time approximation [83]. Perlaers investigated the strain induced effect on the band structure
and effective masses in MoS$_2$ by the density functional theory [84]. The work suggested that a small uniaxial stress can lead to large changes in the hole effective mass. Toriyama and Suzuki derived an approximation for piezoresistance in p-type silicon using the valence band model of Bir and Pikus with the spin-orbit interaction [85, 86]. In the work, the piezoresistance effect was attributed to the hole transfer and conduction mass shift due to stress. The anisotropy of mobility in the piezoresistance in p-type silicon has been reported [87]. A systematic study on the strain-induced effect to the crystal symmetry was introduced by Son et al. [88]. In order to explain the variation of the electrical conductance under stress, the energy band diagram of individual semiconductors needs to be figured out. The energy momentum $E - k$ relationship which can be achieved by solving the Schrödinger equation in the single electron problem. Using Bloch theorem for the periodic potential energy $V(r)$ in a periodic crystal lattice, the Schrödinger equation can be given as

$$\left[ -\frac{\hbar^2}{2m^*} \nabla^2 + V(r) \right] \phi_k(r) = E_k \phi_k(r) \quad (2.15)$$

where $m^*$ is the electron effective mass, $V(r)$ is the potential energy in the direct lattice, and $\phi_k(r)$ is the wave function of the electron.

**Effective mass**

In the case of the electron problem with an external force $F_e$ (e.g. obtained from an electric field $E$), the energy that the electron receives from the external source is

$$\frac{dE}{dt} = F_e \frac{dr}{dt} = F_e v \quad (2.16)$$

where $v$ is the electron velocity in the 3-D space. Applying the wave theory for the electron problem, noting that $v = \frac{d\omega}{dk}$ and $E = \hbar \omega$, where $\omega$ is the angular frequency, we have

$$v = \frac{1}{\hbar} \frac{dE}{dk} \quad (2.17)$$

Combining with Eq. 2.16, one obtains

$$F_e = \frac{d(\hbar k)}{dt} \quad (2.18)$$
This relationship is similar to a classical mechanics problem, where $\hbar k$ is equivalent to the momentum of an external force that makes the electron move. Therefore, the acceleration can be derived in the time dependent domain as

$$a = \frac{dv}{dt} = \frac{1}{\hbar} \frac{d^2E}{dk^2} \frac{d(hk)}{dt} = \left[ \frac{1}{\hbar} \frac{d^2E}{dk^2} \right] F_e$$  \hspace{1cm} (2.19)

Now the quantum problem can be considered as the form of the classical mechanics of motion. Let

$$m^* = \left[ \frac{1}{\hbar} \frac{d^2E}{dk^2} \right]^{-1}$$  \hspace{1cm} (2.20)

be the effective mass which depends on the crystal structure of semiconductors. This relationship indicates that the effective mass can be calculated if the band structure of a specific semiconductor is given. It also can be seen that at the bottom of the conduction band the effective mass is positive. In the valence band, the hole energy increases in the downwards direction of the $E - k$ diagram, indicating that the hole effective mass is also positive in the vicinity of the band extrema.

The band extrema in the $E - k$ diagram are important since the parabolic approximation can yield a constant effective mass. The kinetic equation for either electron and hole is expressed as

$$E - E_0 = \frac{\hbar^2 k^2}{2m^*}$$  \hspace{1cm} (2.21)

The band structure of Si is a good example for the analysis of the band structure. For the case of the electron effective mass, we have the conduction band approximation

$$E - E_0^e = \frac{\hbar^2}{2} \sum_{i=1}^{3} \frac{k_i^2}{m_i^*}$$  \hspace{1cm} (2.22)

where $i$ denotes $x$, $y$, and $z$ axes in 3-D space.

The approximation for the valence band is as

$$E - E_0^v = \frac{\hbar^2}{2m_0} \left[ Ak^2 + \sqrt{B^2k^4 + C^2(k_x^2k_y^2 + k_y^2k_z^2 + k_z^2k_x^2)} \right]$$  \hspace{1cm} (2.23)

where the “+” and “−” signs apply to the light hole and heavy hole bands, respectively; $m_0$ is the free-electron mass; $A$, $B$, and $C$ are the mass parameters.
Variation of electrical conductance in a semiconductor

The electrical conductance of a semiconductor is determined by the charge carrier including the electron occupied in the conduction band and the hole in the valence band as

$$\lambda = e(\mu_e n_e + \mu_h n_h)$$

(2.24)

where $\mu_e$ and $\mu_h$ are the mobilities of the electrons and holes, respectively; $n_e$ and $n_h$ are the carrier concentrations of the electrons and holes, respectively. In terms of p-type semiconductors, the majority of the charge carriers are the holes located in the valence band, then we can simplify Eq. 2.17 into

$$\lambda_p = q\mu_h n_h$$

(2.25)

With reduced Fermi energy $n_F = -(E_F - E_V)/k_B T$, the hole concentration is obeyed the Boltzmann distribution as

$$n_h = N_v F_{1/2} \left( -\frac{E_F - E_V}{k_B T} \right)$$

(2.26)

where $T$ is the temperature (K), $N_v$ is the effective density of state of the holes, $F_{1/2}$ is the Fermi–Dirac integral of order 1/2, $E_F$ and $E_V$ are the energy in Fermi level and the top valence band, respectively.

The hole mobility is

$$\mu_h = \frac{q}{m^*_h} \tau_h$$

(2.27)

where $\tau_h$ is the isotropic relaxation time. Substituting Eq. 2.20, Eq. 2.26 and Eq. 2.27 into Eq. 2.25, we have

$$\lambda_p = \frac{e^2 \tau_h}{m^*_h} N_v F_{1/2} exp \left( -\frac{E_F - E_V}{k_B T} \right)$$

(2.28)

Eq. 2.28 shows that the electrical conductivity of a p-type semiconductor generally depends on the material property (i.e. $E_F$ and $E_V$), the temperature and the orientation (i.e. $m^*_h$). In the case of Si, the hole conductivity comprises of the holes in the valence band.
Chapter 2 Research background and literature review

heavy hole and light hole bands, we have

\[ \lambda_p = \sum_{i=1}^{2} \lambda_i = \sum_{i=1}^{2} \left( e^2 \tau_h \frac{p_i}{m_i} \right) \]  \hspace{1cm} (2.29)

where \( i = 1, 2 \) denotes the heavy hole and light hole bands, respectively; \( p_i \) is the number of holes per unit volume defined as \([89, 90]\)

\[ p_i = N_{vi} F_{1/2}(n_F) \]  \hspace{1cm} (2.30)

where \( N_{vi} = 2(2\pi m_i^* k_B T / h^2)^{3/2} \) denotes the effective density of states in the heavy and light hole bands. Considering the interaction of the spin-orbit split-off band to the two bands is weak, the change of the conductivity is due to the hole transfer mechanism between the heavy hole and light hole bands under stress \([85]\)

\[ \Delta \lambda_i = e^2 \tau_h \left( \frac{\Delta p_i}{m_i} + p_i \frac{\Delta}{m_i} \right) \]  \hspace{1cm} (2.31)

The hole transfer between the heavy hole and light hole bands are

\[ \Delta p_i = - \frac{N_{vi} \Delta(E_{v1} - E_F)}{k_B T} F_{-1/2}(n_F) \]  \hspace{1cm} (2.32)

with a note that the total number of hole remains the same during transferring (i.e. \( \Delta p_1 + \Delta p_2 = 0 \)). The Fermi energy shift can be calculated as

\[ \Delta E_F = \frac{\Delta E_{v1} N_{v1} + \Delta E_{v2} N_{v2}}{N_{v1} + N_{v2}} F_{-1/2}(n_F) \]  \hspace{1cm} (2.33)

From Eq. 2.32 and Eq. 2.33, we have

\[ \Delta p_i = \frac{\Delta E_{v1}(N_{v1} + N_{v2}) - (\Delta E_{v1} N_{v1} + \Delta E_{v2} N_{v2})}{k_B T(N_{v1} + N_{v2})} F_{-1/2}(n_F) \]  \hspace{1cm} (2.34)

Dividing Eq. 2.34 to Eq. 2.30, we have the fractional change of the hole transferred in the two bands as

\[ \frac{\Delta p_i}{p_i} = \frac{\Delta E_{v1}(N_{v1} + N_{v2}) - (\Delta E_{v1} N_{v1} + \Delta E_{v2} N_{v2})}{k_B T(N_{v1} + N_{v2})^2} F_{-1/2}(n_F) \]  \hspace{1cm} (2.35)

where \( F_{-1/2}(n_F)/F_{1/2}(n_F) \) represents the degeneracy in the hole transfer \([91]\).
2.2.3 Piezoresistive effect in SiC

A great deal of effort has been made toward the piezoresistive effect in SiC materials including single crystalline $\alpha$-SiC, $\beta$-SiC, poly crystalline and amorphous SiC (Table 2.3). There are several types of wafers available, such as commercial bulk $\alpha$-SiC wafers fabricated by bulk growth processes, single crystalline $\beta$-SiC, poly crystalline, and amorphous SiC films hetero-epitaxially grown by CVD films on Si substrates [92, 93]. At room temperature, the GF of a single crystalline SiC was measured to be relatively smaller than Si [94, 95], ranging from approximately -30 to 32 and -18 to 25 at elevated temperatures. However, SiC, in turn, is able to maintain its GF at high temperature. Although a high GF is crucial to the sensitivity of SiC devices, their thermal stability is also important to minimize distortion of the output and the requirement for temperature compensation [2]. Despite having a smaller GF compared to that of single crystalline SiC, poly crystalline SiC is favourable for MEMS devices because of lower deposition temperatures required.

In terms of strain engineering, the piezoresistive effect of SiC is typically characterised by three-point or four-point bending beam, cantilever bending and pressurising diaphragm methods. In the first three techniques, a point load is applied to beam-shaped samples with supported points so that a uniaxial strain is induced to the sensing layer. In contrast, pressurising methods yield a biaxial strain which is typically maximized in the vicinity of the diaphragm edges.

2.2.3.1 Piezoresistive effect in SiC micro structures

a. The piezoresistive effect in single crystalline $\beta$-SiC/3C-SiC

Since 3C-SiC can be epitaxially grown on large Si wafers, the cost of 3C-SiC devices can be significantly reduced. 3C-SiC-on-Si platforms are also highly compatible with MEMS processes [14].

A comprehensive comparison of the piezoresistive effect in $\beta$-SiC can be found in [111]. Shor et al. reported the first study on the piezoresistive effect in n-type single crystalline 3C-SiC grown on a Si substrate by APCVD [93]. In the work, a negative GF of a $10^{16}$-$10^{17}$ cm$^{-3}$ doped 3C-SiC was found to be -31.8. Several growth
Table 2.3: List of gauge factors (GF) of SiC reported in the literature

<table>
<thead>
<tr>
<th>Polytype</th>
<th>Type/Dopant</th>
<th>Growth</th>
<th>Carrier concentration</th>
<th>GF Room temp.</th>
<th>GF High temp.</th>
<th>Orientation</th>
<th>Stress</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single 4H-SiC [96]</td>
<td>n/N</td>
<td>-</td>
<td>$1.5 \times 10^{19}$</td>
<td>20.8</td>
<td>-</td>
<td>(0001)</td>
<td>uniaxial</td>
</tr>
<tr>
<td>Single 6H-SiC [97]</td>
<td>n/N</td>
<td>-</td>
<td>$3.8 \times 10^{18}$</td>
<td>-29.4</td>
<td>-17 (250°C)</td>
<td>(0001)</td>
<td>uniaxial</td>
</tr>
<tr>
<td>Single 6H-SiC [98]</td>
<td>n/N</td>
<td>-</td>
<td>$2 \times 10^{19}$</td>
<td>-22</td>
<td>-11 (250°C)</td>
<td>(0001)</td>
<td>uniaxial</td>
</tr>
<tr>
<td>Single 6H-SiC [98]</td>
<td>p/Al</td>
<td>-</td>
<td>$2 \times 10^{19}$</td>
<td>27</td>
<td>12 (250°C)</td>
<td>(0001)</td>
<td>uniaxial</td>
</tr>
<tr>
<td>Single 3C-SiC [93]</td>
<td>n/N</td>
<td>APCVD</td>
<td>$10^{18}$</td>
<td>-31.8</td>
<td>-18 (450°C)</td>
<td>[100]</td>
<td>uniaxial</td>
</tr>
<tr>
<td>Single 3C-SiC [99]</td>
<td>n/N</td>
<td>HMCVD</td>
<td>$10^{18}$</td>
<td>-27</td>
<td>-</td>
<td>[100]</td>
<td>uniaxial</td>
</tr>
<tr>
<td>Single 3C-SiC [100]</td>
<td>n/N</td>
<td>APCVD</td>
<td>-</td>
<td>-18</td>
<td>-7 (400°C)</td>
<td>[100]</td>
<td>biaxial</td>
</tr>
<tr>
<td>Single 3C-SiC [101]</td>
<td>n/N</td>
<td>LPCVD</td>
<td>$0.4 - 2 \times 10^{19}$</td>
<td>-24.8</td>
<td>-11 (450°C)</td>
<td>[100]</td>
<td>biaxial</td>
</tr>
<tr>
<td>Single 3C-SiC [102]</td>
<td>n/N</td>
<td>APCVD</td>
<td>highly doped</td>
<td>-16</td>
<td>-12.5 (400°C)</td>
<td>[100]</td>
<td>uniaxial</td>
</tr>
<tr>
<td>Single 3C-SiC [103]</td>
<td>p/Al</td>
<td>LPCVD</td>
<td>$5 \times 10^{18}$</td>
<td>30.3</td>
<td>-</td>
<td>[110]</td>
<td>uniaxial</td>
</tr>
<tr>
<td>Single 3C-SiC [104]</td>
<td>p/Al</td>
<td>LPCVD</td>
<td>$1.3 - 10 \times 10^{18}$</td>
<td>20-30</td>
<td>-</td>
<td>[110]</td>
<td>uniaxial</td>
</tr>
<tr>
<td>Single 3C-SiC [105]</td>
<td>p/Al</td>
<td>LPCVD</td>
<td>highly doped</td>
<td>28</td>
<td>25 (300°C)</td>
<td>[110]</td>
<td>uniaxial</td>
</tr>
<tr>
<td>Poly 3C-SiC [106]</td>
<td>n/N</td>
<td>LPCVD</td>
<td>low doped</td>
<td>-10</td>
<td>-</td>
<td>-</td>
<td>biaxial</td>
</tr>
<tr>
<td>Poly 3C-SiC [100]</td>
<td>n/N</td>
<td>LPCVD</td>
<td>-</td>
<td>-2.1</td>
<td>-</td>
<td>-</td>
<td>biaxial</td>
</tr>
<tr>
<td>Poly 3C-SiC [107]</td>
<td>p/Bo</td>
<td>LPCVD</td>
<td>$10^{18} - 10^{20}$</td>
<td>10</td>
<td>7 (200°C)</td>
<td>-</td>
<td>uniaxial</td>
</tr>
<tr>
<td>Nanocrystalline SiC [108]</td>
<td>p/Al</td>
<td>LPCVD</td>
<td>$2 \times 10^{18}$</td>
<td>14.5</td>
<td>-</td>
<td>-</td>
<td>uniaxial</td>
</tr>
<tr>
<td>Amorphous SiC [109, 110]</td>
<td>n/N</td>
<td>PECVD</td>
<td>-</td>
<td>49</td>
<td>-</td>
<td>-</td>
<td>uniaxial</td>
</tr>
<tr>
<td></td>
<td>n/N</td>
<td>Sputtering</td>
<td>-</td>
<td>31</td>
<td>-</td>
<td>-</td>
<td>uniaxial</td>
</tr>
</tbody>
</table>

N=Nitrogen  
Al=Aluminum  
Bo=Boron
processes had been carried out to develop 3C-SiC, including a selective deposition of cubic silicon carbide on SOI substrates [112], low temperature LPCVD [113] and hot mesh chemical vapour deposition [99]. The GF of the SEG on Si wafer was found to be -18 [112] while Yasui reported a GF of -27 for n-type 3C-SiC [99]. To eliminate the current leakage between SiC and Si layers, 3C-SiC thin films were transferred to or grown on insulating substrates, such as an unintentionally doped 3C-SiC transferred to SiO$_2$/Si with a GF of -18 [100] and 3C-SiC fabricated by a bonding-free method with a GF of -17.8 [114]. In general, at temperatures above 300°C, the GFs of n-type $\beta$-SiC were found to be less dependent on the temperature change [93].

Phan et al. reported GFs of p-type 3C-SiC of 25 to 28 at the temperature range of 27°C to 300°C using an in situ measurement method [105]. It was concluded that a combination of the piezoresistive and thermostressive effects possibly increases the GF of p-type 3C-SiC to approximately 20% in high temperatures.

b. The piezoresistive effect in single crystalline $\alpha$-SiC

![Energy band structures of 4H- and 6H- SiC](image)

**Figure 2.6**: Energy band structures of a) 4H-SiC and b) 6H-SiC. Courtesy of Kaeckell et al. [115].

Figure 2.6 shows the band structures of 4H- and 6H- SiC calculated using the density functional theory (DFT) and the local density approximation (LDA). Subsequently, the solution for the electron-electron interaction can be derived [115, 116]. Commercial bulk $\alpha$-SiC wafers have been made widely available with mass productions from CREE Inc. [117]. The primary advantage of using commercial bulk $\alpha$-SiC wafers is
that the thermal mismatch between layers is completely eliminated since the functioning layer and substrate are made from the same material. Following are reports on the piezoresistive effect in $\alpha$-SiC, including 4H-SiC and 6H-SiC.

The piezoresistive effect in 4H-SiC

![Diagram of SiC piezoresistors](image)

Figure 2.7: a) SiC piezoresistors placed on top of cantilever beam, b) Cross-sectional view of layers, and c) Measured longitudinal and transverse gauge factor of n-type 4H piezoresistors. Reprinted with permission from [96].

There are very limited studies and applications of 4H-SiC utilising the piezoresistive effect due to the wafer availability and difficulties in the metallisation for good Ohmic contact. Akiyama et al. fabricated n-type 4H-SiC piezoresistors from a 4° off-cut 4H-SiC wafer with a sheet resistance of 0.02 $\Omega$cm and a 295 $\mu$m-thick substrate [96]. The layer structure is illustrated in Fig. 2.7(b) with three epitaxial layers with different doping concentrations. The piezoresistors were formed by patterning the top n-type epitaxial 1 $\mu$m-thick layer with a doping concentration $N_d = 1.5 \times 10^{19}$ cm$^{-3}$. The subsequent layer was 1 $\mu$m-thick p-type with a doping concentration $N_a = 5.4 \times 10^{14}$ cm$^{-3}$. An n-type 2 $\mu$m-thick buffer layer had a doping concentration $N_d = 2.1 \times 10^{19}$ cm$^{-3}$. The electrical isolation of the piezoresistors was generated by the p–n junction.
between two top layers. The results showed the highest GF of 20.8 in a single element piezoresistor in transverse orientation and -10 for the longitudinal orientation with a clustered piezoresistor.

The piezoresistive effect in 6H-SiC

\[
\begin{align*}
G_l &= 1 + 2\nu_{\text{gauge}} - \frac{3D_1 + D_2}{24k_BT} \frac{m_1 - m_2}{m_1 + m_2} (1 + \nu_{\text{sub}})f \\
G_t &= 1 + 2\nu_{\text{gauge}} + \frac{3D_1 + D_2}{24k_BT} \frac{m_1 - m_2}{m_1 + m_2} (1 + \nu_{\text{sub}})f \\
G_s &= -\frac{3D_1 + D_2}{24k_BT} \frac{m_1 - m_2}{m_1 + m_2} (1 + \nu_{\text{sub}})f
\end{align*}
\]

Figure 2.8: Six conduction band minima at M points in the first Brillouin zone (six semi-ellipsoids and equivalent three full ellipsoids). Reprinted with permission from [118].

The piezoresistive effect in n-type 6H-SiC is attributed to the electron transfer and mobility shift in the temperature range of from 300K to 773K, with an impurity concentration of from 2 to \(3.3 \times 10^{19} \text{ cm}^{-3}\) [118, 119]. Figure 2.9 compares theoretical and experimental results of the GFs in 6H-SiC. Three important GFs including the longitudinal, transverse and shear GFs were calculated using band parameters
\[ f \equiv \frac{F_{s-1/2}}{F_{s-1/2}} \]  

(2.39)

where \( \nu_{\text{gauge}} \) and \( \nu_{\text{sub}} \) are Poisson’s ratios of the gauge and the substrate, and \( 0 \leq C \leq 1 \) is the gauge shape factor [120]. In n-type 6H-SiC, the energy extrema are located in between \( M \) and \( L \) along \( \equiv U \) axis of the Brillouin zone [116]. Therefore, a semi-ellipsoidal six-valley model was given to represent the band energy surface, as shown in Fig. 2.8. At an energy level below 12 meV, the constant energy surface contains double-well-like minima, when the energy level higher than 12 meV, the minima became ellipsoidal shapes with a centre at \( M \) point [116]. In the temperature range from 298K to 773K, the corresponding energy level \( 3/2k_B T \) is in a range from 38.5 to 100 meV (far above 12 meV) [118]. The ellipsoidal approximation represents the energy surface of the extrema at \( M \) points and three anisotropic mass components \( m_1, m_2 \) and \( m_3 \), with the \( C_{\text{v}2} \) symmetry at \( M \) points in the wurtzite crystals.

![Figure 2.9: Comparison of the GFs reported by Shor, Okojie and Toriyama. a) longitudinal b) transverse. Reprinted with permission from [118].](image)

The piezoresistive effect of a low doped n-type 6H-SiC grown homo-epitaxially on p-type 6H-SiC were characterized by Shor et al. [97]. At room temperature, the longitudinal GFs of n-type 6H-SiC, with the doping concentrations of \( 1.8 \times 10^{17} \text{ cm}^{-3} \) and \( 3.3 \times 10^{18} \text{ cm}^{-3} \), were found to be -35 and -29.4, respectively. These GFs decreased by roughly 43% at 250°C. At room temperature, the transverse GF was found to be 20 then dropped to 12 at 250°C. The piezoresistive effect in n-type 6H-SiC was found to be decreased with increasing temperature and doping level, which was in good agreement with the multi-valley theory. For example, increasing doping level from
1.8 to $3 \times 10^{17}$ cm$^{-3}$ led to the reduction of the GF by 20%, while an increasing temperature from 25 to 250°C reduced GF by approximately 40%.

The piezoresistive effect in highly doped (i.e. $2 \times 10^{19}$ cm$^{-3}$) n-type and p-type 6H-SiC were investigated by Okojie et al. [98]. The GF of n-type 6H-SiC was found to be 22 at room temperature, followed by a decrease of 52% at 250°C, while the GF of p-type 6H-SiC was 27 at room temperature, then decreased 55% at 250°C. The experiments of Shor and Okojie agreed that the magnitude of the GFs in n- and p-type 6H-SiC decreases when increasing the carrier concentration and temperature.

### 2.2.3.2 Piezoresistive effect in SiC nano structures

Advancements in fabrication processes of nano-scales allowed more studies on SiC NWs (one dimension) and nano-thin films (two dimensions) [122–127]. He et al. reported Si NWs with an unusually large piezoresistive coefficient along the $<111>$

![Figure 2.10](image)

**Figure 2.10:** a) Electromechanical characterization measurements, b–c) AFM and topography images of a p-type 6H-SiC nano wire on the graphite substrate, d) I–V curves at different applied forces across the nano wires, e) The relationship between the nano wire resistance and the applied forces, f–g) SEM and TEM images of p-type 6H-SiC nano wires. Reprinted with permission from [121].
direction, reaching $-3.550 \times 10^{-11} \text{ Pa}^{-1}$ for the p type nanowires with diameters ranging from 50 to 350 nm and the resistivities of from 0.003 to 10 $\text{O} \text{cm}$ [128]. This result was much higher than that of bulk Si which is only $-94 \times 10^{-11} \text{ Pa}^{-1}$. It was hypothesized that the strain-induced carrier mobility change and surface modifications had a significant impact on the piezoresistive coefficients. Based on this interesting result, a number of studies on the piezoresistive effect in SiC nano structures have been carried out towards the harsh environment sensing [129, 130].

Bottom-up growth methods are typically used to fabricate 3C-SiC NWs. To investigate the piezoresistive effect, mechanical stresses were locally induced to 3C-SiC NWs using either piezoelectric or electrostatic actuators and the applied strain was estimated using SEM [129, 130]. However, the obtained GFs, which were only -6.9 and 14.1, are relatively small in comparison to that of bulk 3C-SiC. In another work, an AFM tip was used to induce a strain onto an n-type 3C-SiC NW [121], as shown in Fig. 2.10. The transverse GF of the NW was found to be from 4.5 to 46.2. It was assumed that the piezoresistance was attributed to the band structure modification and variation of surface states in SiC NW. Gao et al. characterized the piezoresistivity of p-type 6H-SiC NWs with high piezoresistive coefficients from 51.2 to $159.5 \times 10^{-11} \text{ Pa}^{-1}$ [131].

![Figure 2.11: a) SEM images of 3C-SiC NWS with released and non-released SiC NWs. b) A 5 fold-sensitivity enhancement with released NWs. Reprinted with permission from [127].](image)

Phan et al. reported the piezoresistive effect of top-down fabricated 3C-SiC NWs using FIB [127]. A p-type 3C-SiC NWs was patterned from a 3C-SiC thin film epitaxially grown on a Si substrate with a carrier concentration of $5 \times 10^{18} \text{ cm}^{-3}$. 

33
By applying tensile strains varying from 0 to 280 ppm, a large GF of 35 of the p-type 3C-SiC NWs was obtained. When the 3C-SiC NWs was etched back to form the suspended structure Fig. 2.11(a), the magnitude of the piezoresistive in the NW based strain was amplified by a factor of 6 in comparison to that of conventional SiC micro and nano structures (Fig. 2.11(b)). As shown in Fig. 2.11, the effective GF of the 380 nm and 470 nm SiC NW was measured to be 150 and 124, respectively, which were about 5 fold-increase from bulk 3C-SiC.

A giant piezoresistive effect in p-type 3C-SiC NWs has been reported [133]. The measured negative piezoresistive coefficient \( \pi_{\{110\}} \) of the nano wires was in a range of -8.83 to -103.42 \times 10^{-11} \text{ Pa}^{-1} with the applied forces ranged from 51.7 to 181.0 nN. A giant GF was reported for a NW with a diameter of 540 nm as high as 620.5, which was more than 10 times compared to the other reports for SiC nano structures. Additionally, simulation models have been also established to investigate piezoresistivity of SiC nano structures [126, 132, 134, 135]. The simulated piezoresistivity of n-type \( \alpha \)- and \( \beta \)-SiC nano sheets were reported using the density functional theory by Nakamura et al. (Fig. 2.12). The results showed that at room temperature the GF of SiC

![Figure 2.12](image-url)
Chapter 2 Research background and literature review

Figure 2.13: Variations in calculated longitudinal and transverse GFs with reciprocal of temperature of n-type 4H-, 6H-, and 3C-SiC (0001) nano-sheet models for the [1100] tensile strain at $1 \times 10^{19} \text{ cm}^{-3}$ carrier concentration. Courtesy of Naka-
mura et al. [132].

Nano thin films varies from 30 to 60, followed by a decrease of approximately 50% at 500°C, as shown in Fig. 2.13. It was also suggested that piezoresistivity of nano thin films should be on a par with that of bulk SiC wafers, and a giant piezoresistive effect should be very limited in SiC NWs due to a quantum confinement.

2.2.3.3 Carrier concentration, orientation and temperature dependences of piezoresistive effect in SiC

A number of studies focused on the dependence of the GF on carrier concentration and orientation in order to obtain higher GFs. Shor and Eickhoff found that the GF decreases with increasing carrier concentration [93, 100]. The results also indicated that the thermal stability of the GF of 3C-SiC can be enhanced by increasing doping concentration. The first characterization of the orientation dependence of the n-type 3C-SiC was reported in various directions [93]. The result showed that the highest GF of n-type 3C-SiC was in [100] direction of (100) plane. Dao et al. reported the
piezoresistive effect in p-type single crystalline 3C–SiC which was grown on a Si (111) substrate. The GF in the (111) plane was found to be 11, which is smaller than that in p-type 3C–SiC (100) [136]. This was attributed to the defects occurring in the as-grown 3C–SiC film in the (111) direction.

A comparison of the temperature dependence of the GF in 3C-SiC is illustrated in Fig. 2.14. It can be seen that the GF of highly doped n-type 3C-SiC is more temperature stable than that of low doped n-type 3C-SiC. For highly doped n-type 3C-SiC, the GF only decreased 20% while that of low doped 3C-SiC decreased approximately 50% at temperatures above 400°C. At temperatures above 200°C, the magnitude of the GF of 3C-SiC is typically reported in a range of 10 to 18.

2.3 SiC based sensors for harsh environments

In order to work properly in harsh conditions, mechanical sensors are required to retain their characteristics in one or more of followings conditions: large temperature variations or high temperatures, highly corrosive, intense radiation exposure, and high shock. These conditions are usually found in combustion engines, energy and chemical processes, military control, and space exploration, as shown in Fig. 2.15.
Chapter 2 Research background and literature review

In combustion monitoring, the efficiency of automotive engines and gas turbines is not only dependent on manufacturing processes but also the degradation of components and fuel and intake air qualities [144, 145]. In order to optimize the efficiency and reduce emission, working parameters, such as temperature, pressure, air-to-fuel ratio, are needed to be accurately monitored [2]. Conventional monitoring methods passively observe events in the operation of engines, limiting the control efficiencies. This obstacle can be overcome by utilising real-time monitoring [145].

<table>
<thead>
<tr>
<th>Requirement</th>
<th>Combustion monitoring</th>
<th>Process Monitoring</th>
</tr>
</thead>
<tbody>
<tr>
<td>Application</td>
<td>Structural Health Monitoring</td>
<td>Navigation</td>
</tr>
<tr>
<td></td>
<td>Space Exploration</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Requirement</th>
<th>High shock &amp; Vibration</th>
<th>High temperature</th>
<th>Corrosive media</th>
<th>Intense radiation</th>
</tr>
</thead>
</table>

| Figure 2.15: Requirements in various harsh environment applications. Reprinted with permission from [2]. |

<table>
<thead>
<tr>
<th>Table 2.4: Comparison of SiC based pressure sensors</th>
</tr>
</thead>
<tbody>
<tr>
<td>Work of</td>
</tr>
<tr>
<td>--------------------------------------------------</td>
</tr>
<tr>
<td>Okojie [137]</td>
</tr>
<tr>
<td>Okojie [138]</td>
</tr>
<tr>
<td>Akiyama [139]</td>
</tr>
<tr>
<td>Okojie [98]</td>
</tr>
<tr>
<td>Wieczorek [140]</td>
</tr>
<tr>
<td>Shor [93]</td>
</tr>
<tr>
<td>Eickhoff [112]</td>
</tr>
<tr>
<td>Wu [100]</td>
</tr>
<tr>
<td>Zierrmann [141]</td>
</tr>
<tr>
<td>Berg [142]</td>
</tr>
<tr>
<td>Chung [143]</td>
</tr>
<tr>
<td>Fraga [109]</td>
</tr>
</tbody>
</table>

* Single crystalline
* Poly crystalline
* Amorphous
Chapter 2 Research background and literature review

The piezoresistive effect of SiC has been extensively studied for pressure sensors at high temperatures as summarized in Table 2.4. SiC based pressure sensors have been commonly in diaphragm forms which were fabricated by various techniques. A 4H-SiC pressure sensor with piezoresistive transducers for high temperature and corrosive chemical environments was also reported [139]. As such, a bulk single crystal SiC wafer was used to fabricate a 4H-SiC pressure sensor, which had a diameter of 1 mm and a thickness of 50 µm, by a milling process. Piezoresistors in both transverse and longitudinal directions were fabricated on an n-type SiC diaphragm. It was shown that an Ohmic contact was acquired by depositing triple metal layers (i.e. Ta/Ni/Pt) for the electrical contact then annealing at 1000°C in 20 minutes. The 4H-SiC sensors were also measured under hydrostatic pressures of up to 60 bar with a sensitivity of 268 µV/V/bar. The devices exhibited a good thermal stability in a high temperature up to 600°C for 165 hours.

Figure 2.16: a) Actual test fixture location. b) Test fixture for Si and SiC in-cylinder where SiC die are mounted to the tungsten disk with a high temperature ceramic adhesive. c) Silicon carbide sample, A-C: samples before engine testing; D-F: samples after 20, 40, and 60 mins. Courtesy of Wodin et al. [146].

The majority of studies on SiC based pressure sensors focus on the proof-of-concepts for pressure sensing at elevated temperatures. However, for real applications, further developments needed to be conducted to optimize the resolution, temperature and long-term stabilities. For instance, an unstable offset voltage of 4H-SiC pressure sensors during a long-term work at 600°C was reported due to an agglomeration electrodes and SiC [137]. In a 4H-SiC based pressure sensor at 800°C, the full-scale output (FSO) voltage gradually decreased with increasing temperature from 23°C to
Chapter 2 Research background and literature review

400°C. Interestingly, the sensitivity of the sensors increases in temperatures above 400°C [138].

In terms of combustion engines, the in-cylinder monitoring is vital to improve the engine efficiency and the fuel flexibility. The use of MEMS sensors in the cylinders assist the monitoring and control of combustion events in order to enhance the fuel consumption efficiency and minimise gas emissions (Fig. 2.16). SiC based MEMS sensors are good candidates in the combustion engine monitoring owing to the capability of high-temperatures and high-corrosive tolerances. Wodin et al. developed an amorphous silicon carbide (α-SiC) coated die for a combustion engine with an exhaust temperature of 800°C. It was found that no measurable oxide was grown during the long-term operation, indicating the robustness of the SiC sensor for the in-cylinder monitoring [146].

![Figure 2.17: High shock sensor with four longitudinal piezoresistors on the narrow beams and the four damping slots. Reprinted with permission from [147].](image-url)

SiC is also a promising material for high shock and intense vibration sensing devices. For instance, an extreme impact of up to 40,000 g ($g=9.81 \text{ ms}^{-2}$) was measured by a 6H-SiC accelerometer placed on a diaphragm as the piezoresistive sensing element (Fig. 2.17) [147]. In this work, several shapes accelerometers with resonant frequencies in a range of 200 – 800 kHz were designed and the obtained sensitivities were
in a range from 40 to 250 nV/g. An amorphous SiC strain sensor produced by RF magnetron sputtering for monitoring the mechanical strain in hot zones of combustion chambers was reported [110]. The GF of the sensor was found to be 48 with a very low thermal coefficient of resistance.

Figure 2.18 shows a silicon carbide tuning fork developed by Myers et al., which could withstand harsh environments and retain the strain sensitivity and resolution bandwidth [148]. The sensor exhibited good performance in the operation at 600°C with the existence of dry steam and at a high acceleration of 64,000 g. The device yielded a high strain sensitivity of 66 Hz/µε with a resolution of 0.045 µε in the 10 kHz bandwidth.
2.4 Micromachining of SiC materials

2.4.1 SiC etching

Despite the chemical resistance of SiC, silicon-compatible etching processes have been developed for SiC, including wet etching and dry etching [14]. Recently, dry etching of SiC is being widely employed to selectively etch SiC. These processes are based on plasma-based RIE, providing an accurate control of the width, side wall, and surface profile. SiC is removed in RIE via physical and chemical processes. In the sputtering process, surfaces are bombarded by energetic particles from the plasma, while in chemical etching, there are reactions of active chemical elements and surface particles. It is important to precisely control these two processes in order to obtain an etch profile with the expected slop angles or surface finish. There are a number of etching species employed in plasma etching processes, including CF$_4$, SF$_6$, and NF$_3$ [149–151]. Among these compounds, SF$_6$/O$_2$ is a favourable combination because it has been widely developed for Si etching [149]. The required etching depth depends on device types, such as in the sub-micron range for electronics devices, few microns for MEMS devices, and hundreds of microns for resonant SiC devices [152]. The dry etching rate of SiC is significantly slower than that of Si. However, a fast etching rate of 1 $\mu$m/min has been reported, enabling the dry etching for thick SiC films [153].

Wet chemical etching methods have been also reported in a number of studies. The wet etching of SiC includes the oxidation of the SiC surface and following dissolution of oxides. There are various methods for wet etching SiC. For example, SiC was etched using hot molten KOH [154] and orthophosphoric acid [155]. However, the etching rates were slow and required a high temperature condition. A photo electrical technique developed by Shor can be used to pattern the epilayer of bulk $\alpha$-SiC wafers, in which the top SiC layer can be selectively etched, excluding the SiC layer underneath with a different dopant [156]. In the process, ultraviolet illumination in HF solution was used to selectively remove the top n-type 3C-SiC on p-type 3C-SiC [156]. The main advantage of wet etching methods is the ability of selective etching of amorphous SiC in single crystalline SiC or dopant selective etching. Because the etch process takes place via the oxidation of surfaces, the micro structures of the surface and dopant type are very crucial for the SiC properties [155].
2.4.2 SiC thin film transferring

A poly silicon bonding technique was conducted to obtain single crystalline 3C-SiC-on-SiO$_2$ films. In the technique, two wafers were bonded by their poly silicon layers [157]. Another approach was to form 3C-SiC layer on SiO$_2$ films by anodically bonding 3C-SiC to a glass substrate [158]. In this process, a SiO$_2$ layer was deposited on a Si wafer by a high temperature oxidation. Subsequently, a multi-layer of SiO$_2$, SiC and Si was bonded onto an aluminosilicate glass substrate. The 3C-SiC-on-glass structure was then obtained by the removal of the Si layer by RIE and wet etching. A combination of wafer bonding and smart cut techniques were used to fabricate single crystal 6H-SiC MEMS devices [159] and 6H-SiC micro disk torsional resonators [160]. A single crystalline $\beta$-SiC thin film was formed on SOI substrates for high temperature applications which exhibited good electrical insulation of the SiC layer from the Si substrate in a temperature range of up to 723K [161].

2.4.3 Bulk micromachining

In bulk micromachining processes, suspended structures are obtained by selective removal of underneath substrates. This process is necessary for the fabrication of bulk SiC wafers and SiC on Si wafers [162], in which SiC is patterned by plasma etching or chemical wet etching. Released SiC structures can be formed by etching top or bottom surface of substrates. A wide range of MEMS structures, such as cantilever beams, suspended bridges, and membranes of epitaxially grown 3C-SiC, can be fabricated by this technique. Thin SiC films were etched using wet etching (KOH) or dry etching (RIE, ICP) on Si, SiO$_2$ or SOI layers [158, 161, 163], while in bulk SiC wafers, SiC layers were etched by using electrochemical etching or RIE. The etching rate of SiC (hundreds of nano-meter-per-min [153, 164]) was much slower than that of Si (few micro-meter-per-min [165]). Several approaches were introduced to increase the etching rate of SiC, including laser micromachining and wafer milling [139, 140].
2.5 Summary of literature review and research goals

In summary, SiC materials are superior over Si in mechanical sensing in harsh environments. The GFs of SiC vary with temperature, the impurity concentrations, and the quality/growth processes of SiC films. The dopant type (n or p) typically dictates the sign of the GFs (i.e. “-” sign for longitudinal n-type or transverse p-type piezoresistors and “+” sign for transverse n-type or longitudinal p-type piezoresistors). Moreover, the GF normally decreases with increasing the carrier concentration and with increasing temperature [120]. The positive (or negative) values of the GF indicate the conductivity of the SiC piezoresistors decrease (or increase) with increasing tensile stress/strain. In terms of sensing devices, only the magnitude of the GF is significant not the sign. However, in the case of mobility enhancement, it is possible to significantly increase the electron mobility in p-type/n-type SiC by utilizing the transverse/longitudinal tensile strains (with negative GF).

Although a great effort has been carried out for the investigation of the piezoresistive effect in SiC materials, there are research gaps which need to be addressed. The piezoresistive effect in 4H-SiC as well as 4H-SiC based mechanical sensors have been rarely reported due to the lack of availability of 4H-SiC wafers and the difficulties in obtaining the good Ohmic contact for 4H-SiC devices. Recent rapid developments in the 4H-SiC wafer manufacturing enable the use of this polytype in practical applications. However, the limited insight of the piezoresistive effect in 4H-SiC at room and high temperatures is hindering developments of the 4H-SiC material for mechanical sensing in harsh environments. Therefore, this thesis aims at the following goals to bring 4H-SiC a further step towards practical applications in harsh environments:

i) Understanding the piezoresistive effect in p-type 4H-SiC by theoretical and experimental studies and developing the good Ohmic contact between metal and SiC,

ii) Investigating the piezoresistive coefficients of p-type 4H-SiC in the (0001) plane,

iii) Investigating the temperature dependence of the PZR effect in p-type 4H-SiC,

iv) Developing 4H-SiC based van der Pauw strain sensor,
v) Developing 4H-SiC based pressure sensor utilizing the piezoresistive effect in the p-type 4H-SiC.
Chapter 2 Research background and literature review

Bibliography


Chapter 2  Research background and literature review


Chapter 2 Research background and literature review


Chapter 2 Research background and literature review


Chapter 2 Research background and literature review


Chapter 2 Research background and literature review


Chapter 2 Research background and literature review


Chapter 2 Research background and literature review


Chapter 2 Research background and literature review


Chapter 3

Methodology

In this chapter, the general methodology for all the works in this thesis, including theoretical analyses and experimental characterisations, will be presented. The detailed methods specified to each published work are elaborated within respective papers. To investigate the piezoresistive effect in p-type 4H-SiC and the piezoresistive coefficients in the (0001) plane of p-type 4H-SiC, analytical calculations based on the hole transfer mechanism and a coordinate transformation were conducted. In the experiment work, SiC micro structures were fabricated using micro-fabrication processes, followed by a metallisation process to obtain Ohmic contact with high temperature annealing. The temperature dependence of the piezoresistive effect in p-type 4H-SiC at varying temperatures of up to 600°C was investigated. The device integration aspect of 4H-SiC was demonstrated by the fabrication and characterisation of a 4H-SiC based strain sensor and a 4H-SiC pressure sensor, which employed the piezoresistive effect as the sensing mechanism.
3.1 Investigation on piezoresistive effect in 4H-SiC

3.1.1 Theoretical methods

3.1.1.1 Analysis of strain induced effect to the electrical conductance of p-type 4H-SiC

The variation of the electrical conductance in 4H-SiC is dictated by the modification in the top valence bands, namely the heavy hole (HH), light hole (LH), and spin-orbit split-off (SOSO) bands. Figure 3.1(a) illustrates the first Brillouin zone of 4H-SiC with the highest symmetry points on the axes and on the edge of the hexagonal prism. The piezoresistive effect in 4H-SiC can be clarified by considering the hole energy shift in the strained crystal structure of its wurtzite lattice in the space group $C_{6v}^4$. The piezoresistance of p-type 4H-SiC is correlated to the hole transfer by means of the deformation of the top valence bands under strain [1]. As such, a uniaxial stress could raise the light hole (LH) bands and simultaneously lower the heavy hole (HH) bands parallel to the stress orientation because of the change in the overlap between the atomic orbitals. Figure 3.1(b) shows the constant energy surfaces for three topmost valence bands without strain in 4H-SiC, showing ellipsoidal shapes [2].

![Figure 3.1: a) First Brillouin zone of hexagonal SiC (e.g. 4H- and 6H-SiC) with high symmetry points in $\mathbf{k}$-space. b) Representation of the constant energy surfaces for three highest valence bands in 4H-SiC. From top to bottom: Light hole, heavy hole, and spin orbit split-off (SOSO) bands, respectively.](image-url)
Chapter 3 Methodology

Hole occupation in valence subbands of 4H-SiC

The orientation dependence of the piezoresistive effect in 4H-SiC can be determined by the hole energy shift of the valence bands at \( k = 0 \) under strain. According to Bir [3], the energy of the three topmost valence bands at \( k = 0 \) in the unstrained 4H-SiC crystal are

\[
E_i^0 = \Delta_i + \Delta_2
\]

(3.1)

where \( \Delta_i (i = 1, 2, 3) \) are the spinor representations for the symmetry points in the Brillouin zone. The first order approximation for the strained band energy \( E(k, \epsilon) \) are

\[
E_i^s = F
\]

\[
E_{2,3}^s = \frac{\Delta_1 - \Delta_2}{2} \pm \sqrt{\left( \frac{\Delta_1 - \Delta_2}{2} \right)^2 + 2\Delta_3^2}
\]

(3.2)

where \( \Delta = \sqrt{2}\Delta_3; F = \Delta_1 + \Delta_2 + \lambda + \theta; G = \Delta_1 - \Delta_2 + \lambda + \theta; \lambda = A_1k_x^2 + A_2(k_x^2 + k_y^2) + D_1\epsilon_{zz} + D_2(\epsilon_{xx} + \epsilon_{yy}); \theta = A_3k_z^2 + A_4(k_x^2 + k_y^2) + D_3\epsilon_{zz} + D_4(\epsilon_{xx} + \epsilon_{yy}); \epsilon \) is the strain component; \( k_x, k_y, \) and \( k_z \) are the vectors in the \( k \)-space, \( A_i \) are the components of the Hamiltonian matrix of the 4H-SiC crystal. Substituting into Eq. 3.2, we have

\[
E_i^s = \Delta_1 + \Delta_2 + \lambda + \theta
\]

(3.3)

\[
E_{2,3}^s = \frac{\Delta_1 - \Delta_2 + 2\lambda + \theta}{2} \pm \sqrt{\left( \frac{\Delta_1 - \Delta_2 + \theta}{2} \right)^2 + \Delta^2}
\]

In the case \( 2\Delta \ll \Delta_1 - \Delta_2 + \theta \)

\[
E_2^s = \frac{\Delta_1 - \Delta_2 + 2\lambda + \theta}{2} + \frac{\Delta_1 - \Delta_2 + \theta}{2} \sqrt{\left( \frac{\Delta_1 - \Delta_2 + \theta}{2} \right)^2 + \Delta^2}
\]

\[
\approx \Delta_1 - \Delta_2 + \lambda + \theta + \frac{\Delta^2}{\Delta_1 - \Delta_2 + \theta}
\]

(3.4)

\[
E_1^s - E_2^s = 2\Delta_2 - \frac{\Delta^2}{\Delta_1 - \Delta_2 + \theta}
\]
If $2\Delta \gg \Delta_1 - \Delta_2 + \theta$

$$E_2^s = \frac{\Delta_1 - \Delta_2 + 2\lambda + \theta}{2} + \Delta \sqrt{\left(\frac{\Delta_1 - \Delta_2 + \theta}{2\Delta}\right)^2 + 1}$$

$$\approx \frac{\Delta_1 - \Delta_2 + 2\lambda + \theta}{2} + \Delta + \frac{(\Delta_1 - \Delta_2 + \theta)^2}{8\Delta} \quad (3.5)$$

$$E_1^s - E_2^s = \frac{\Delta^2}{\Delta_1 - \Delta_2 + \theta}$$

Hole occupation for the highest valence subband is given as

$$P_1 = 2\int \left[ e^{-\frac{E_1^s - E_F}{k_BT}} + 1 \right]^{-1} dk$$

$$\approx 2\int \frac{E_1^s - E_F}{k_BT} dk$$

$$= 2\int e^{-\frac{\Delta_1 + \Delta_2 + \lambda + \theta - E_F}{k_BT}} dk$$

$$= 2\int e^{-\frac{\Delta_1 + \Delta_2 + D_\lambda(\epsilon) + D_\theta(\epsilon) - E_F}{k_BT}} \times \int e^{\frac{A_2 + A_4}{k_BT}k_x^2} dk_x \int e^{\frac{A_2 + A_4}{k_BT}k_y^2} dk_y$$

$$\times \int e^{\frac{A_1 + A_3}{k_BT}k_z^2} dk_z \quad (3.6)$$

where

$$D_\lambda(\epsilon) \equiv D_1\epsilon_{zz} + D_2(\epsilon_{xx} + \epsilon_{yy})$$

$$D_\theta(\epsilon) \equiv D_3\epsilon_{zz} + D_4(\epsilon_{xx} + \epsilon_{yy}) \quad (3.7)$$

with either $A_1 + A_3$ and $A_2 + A_4$ are negative.

Subsequently, we derive

$$P_1 = 2e \frac{\Delta_1 + \Delta_2 + D_\lambda(\epsilon) + D_\theta(\epsilon) - E_F}{k_BT} \frac{\pi k_BT}{-3/2} \sqrt{-\frac{1}{(A_1 + A_3)(A_2 + A_4)^2}} \quad (3.8)$$
Using first order approximation for the second highest valence subband, we have

\[
P_2 = 2e \frac{\Delta_1 + \Delta_2 + D_\lambda(\epsilon) + D_\theta(\epsilon) + \Delta^2/(\Delta_1 - \Delta_2 + \theta) - E_F}{k_B T} \frac{(\pi k_B T)^{3/2}}{\sqrt{- (A_1 + A_3)(A_2 + A_4)^2}}
\]

(3.9)

From Eq. 3.8 and Eq. 3.9, we have

\[
\frac{P_1}{P_2} = \frac{2\Delta_2 - \Delta^2/(\Delta_1 - \Delta_2 + \theta)}{k_B T}
\]

(3.10)

\[
= \frac{E_1^s - E_2^s}{k_B T}
\]

Conducting the same procedure, we also have

\[
\frac{P_1}{P_3} = \frac{E_1^s - E_3^s}{k_B T}
\]

(3.11)

According to Persson [4], \(\Delta E_{1-2} = 0.15 \text{ eV}\) and \(\Delta E_{1-3} = 0.8 \text{ eV}\), substitute \(k_B = 1.38 \times 10^{-23} \text{JK}^{-1}\) and \(T = 298 \text{K}\), we have

\[
\frac{P_1}{P_2} = \frac{0.15 \times 1.62 \times 10^{-19}}{298 \times 1.38 \times 10^{-23}} = e^{5.9} \approx 365
\]

(3.12)

\[
\frac{P_1}{P_3} = \frac{0.8 \times 1.62 \times 10^{-19}}{298 \times 1.38 \times 10^{-23}} = e^{31.5} \approx 4.83 \times 10^{13}
\]

These results indicate that the number of hole occupied in the \(E_1\) subband is much greater than the other two subbands. Therefore, it can be concluded that the contribution of the hole transfer in \(E_2\) and \(E_3\) subbands to the piezoresistive effect is negligible.

**Strain induced effect on valence band energy**

Taking the second derivative of \(E_1^s\) and \(E_2^s\) from Eq. 3.2 at strain-free state, we have
In \( \Gamma A \) direction
\[
\frac{\partial^2 E_1^0}{\partial k_z^2} = 2(A_1 + A_3)
\]
\[
\frac{\partial^2 E_2^0}{\partial k_z^2} = 2(A_1 + A_3 \frac{E_2^0}{E_2^0 - E_3^0})
\]
(3.13)

In \( \Gamma M \) and \( \Gamma K \) directions
\[
\frac{\partial^2 E_1^0}{\partial k_z^2} = \frac{\partial^2 E_2^0}{\partial k_z^2} = 2(A_2 + A_4)
\]
(3.14)

It should be noted that \( A_i \) (\( i=1,2,3,4 \)) and \( E_3^0 < 0 \), we have \( \partial^2 E_1^0 / \partial k_j^2 > \partial^2 E_2^0 / \partial k_i^2 \) with \( j=(x,y,z) \), or in other words, the holes occupied in the subband \( E_1 \) has small effective mass and high mobility while the holes in the subband \( E_2 \) has large effective mass and low mobility.

At strained state, we also have

in \( \Gamma A \) direction
\[
\frac{\partial^2 E_1^s}{\partial k_z^2} = \frac{\partial^2 E_1^0}{\partial k_z^2} = 2(A_1 + A_3)
\]
\[
\frac{\partial^2 E_2^s}{\partial k_z^2} = 2A_1 + A_3 + A_3 \frac{\Delta_1 - \Delta_2 + D_0(\epsilon)}{2 \sqrt{(\frac{\Delta_1 - \Delta_2 + D_0(\epsilon)}{2})^2 + \Delta^2}}
\]
(3.15)

in \( \Gamma M \) and \( \Gamma K \) directions
\[
\frac{\partial^2 E_1^s}{\partial k_z^2} = \frac{\partial^2 E_1^0}{\partial k_z^2} = 2(A_2 + A_4)
\]
\[
\frac{\partial^2 E_2^s}{\partial k_z^2} = 2A_2 + A_4 + A_4 \frac{\Delta_1 - \Delta_2 + D_0(\epsilon)}{2 \sqrt{(\frac{\Delta_1 - \Delta_2 + D_0(\epsilon)}{2})^2 + \Delta^2}}
\]
(3.16)

Eq. 3.15 and Eq. 3.16 indicate that the variation of the subband \( E_1 \) is independent on the strain direction.
In the case where $2\Delta \ll \Delta_1 - \Delta_2 + D_\theta(\epsilon)$ with $\theta = A_3k_z^2 + A_4(k_z^2 + k_y^2) + D_3\epsilon_{zz} + D_\theta(\epsilon)$, we have

$$\frac{\partial^2 E_2^z}{\partial k_z^2} \approx \frac{\partial^2 E_2^0}{\partial k_z^2}$$

$$\frac{\partial^2 E_2^x}{\partial k_z^2} \approx \frac{\partial^2 E_2^0}{\partial k_z^2}$$

$$\frac{\partial^2 E_2^y}{\partial k_y^2} \approx \frac{\partial^2 E_2^0}{\partial k_y^2}$$

Therefore, the subband $E_2$ is also independent on the strain orientation.

### 3.1.1.2 Graphical representation of piezoresistive coefficients

**Coordinate transformation**

![Coordinate transformation diagram](image)

**Figure 3.2**: a) A general representation of two coordinates applied to a hexagonal crystal structure. b) Euler angles of the transformation from the principle coordinate to an arbitrary coordinate.

The relationship between $O(X^*,Y^*,Z^*)$ and $O(X,Y,Z)$ is related to the coordinate transformation of two first-rank tensors as

$$r' = Rr \quad \text{or} \quad \begin{bmatrix} x' \\ y' \\ z' \end{bmatrix} = \begin{bmatrix} l_1 & m_1 & n_1 \\ l_2 & m_2 & n_2 \\ l_3 & m_3 & n_3 \end{bmatrix} \begin{bmatrix} x \\ y \\ z \end{bmatrix}$$

(3.18)
where \( R \) is the rotational matrix from \( O(X,Y,Z) \) to \( O(X^*,Y^*,Z^*) \) and \( l_i, m_i, \) and \( n_i \) satisfy \( l_i^2 + m_i^2 + n_i^2 = 1 \) and \( l_i l_j + m_i m_j + n_i n_j = 0 \) \((i \neq j)\). The three Euler’s angles \( \phi, \theta, \) and \( \psi \) are typically used for the coordinate transformation, as shown in Fig. 3.2.

The transformation is as follows:

1) Rotating \( O(X,Y,Z) \) counter-clockwise an angle \( \phi \) to a new coordinate \( O(X',Y',Z') \);
2) Rotating \( O(X',Y',Z') \) an angle \( \theta \) to \( O(X'',Y'',Z'') \);
3) Rotating \( O(X'',Y'',Z'') \) an angle \( \psi \) to \( O(X^*,Y^*,Z^*) \). Thus, the rotational matrix is

\[
\begin{bmatrix}
  l_1 & m_1 & n_1 \\
  l_2 & m_2 & n_2 \\
  l_3 & m_3 & n_3 \\
\end{bmatrix}
= \begin{bmatrix}
  \cos\phi\cos\theta\cos\psi - \sin\phi\sin\theta \cos\psi + \sin\phi\cos\phi & -\cos\phi\sin\theta \\
  -\sin\phi\cos\phi - \cos\phi\cos\psi \sin\phi + \sin\phi\cos\phi & \sin\phi\sin\theta \\
  \sin\phi\cos\phi & \sin\phi\sin\phi & \cos\phi \\
\end{bmatrix}
\]

(3.19)

From the relationship of the electric field tensor \( E \) versus the electric current density tensor \( J \) and resistivity tensor \( \rho \) in the principle coordinate, \( E = \rho J \) and in an arbitrary coordinate, \( E' = \rho' J' \), then conducting the coordinate transformation using the Euler’s angles: \( E \rightarrow E' \) and \( J \rightarrow J' \), we have

\[
\rho' = R\rho R^t
\]

(3.20)

Since the resistivity tensor is second-rank and symmetrical, there are only six independent components \( \rho_{xx} \equiv \rho_1, \rho_{yy} \equiv \rho_2, \rho_{zz} \equiv \rho_3, \rho_{xy} \equiv \rho_6, \rho_{yz} \equiv \rho_4, \) and \( \rho_{zx} \equiv \rho_5 \). Eq. 3.20 becomes

\[
\begin{bmatrix}
\rho_1 \\
\rho_2 \\
\rho_3 \\
\rho_4 \\
\rho_5 \\
\rho_6 \\
\end{bmatrix}
= \begin{bmatrix}
l_1^2 & m_1^2 & n_1^2 & 2m_1n_1 & 2n_1l_1 & 2l_1m_1 \\
l_2^2 & m_2^2 & n_2^2 & 2m_2n_2 & 2n_2l_2 & 2l_2m_2 \\
l_3^2 & m_3^2 & n_3^2 & 2m_3n_3 & 2n_3l_3 & 2l_3m_3 \\
l_2l_3 & m_2m_3 & n_2n_3 & m_2n_3 + m_3n_2 & n_2l_3 + n_3l_2 & m_2l_3 + m_3l_2 \\
l_3l_1 & m_3m_1 & n_3n_1 & m_3n_1 + m_1n_3 & n_3l_1 + n_1l_3 & m_3l_1 + m_1l_3 \\
l_1l_2 & m_1m_2 & n_1n_2 & m_1n_2 + m_2n_1 & n_1l_2 + n_2l_1 & m_1l_2 + m_2l_1 \\
\end{bmatrix}
\begin{bmatrix}
\rho_1 \\
\rho_2 \\
\rho_3 \\
\rho_4 \\
\rho_5 \\
\rho_6 \\
\end{bmatrix}
\]

(3.21)
Chapter 3 Methodology

Let

\[ \alpha = \begin{bmatrix}
  l_1^2 & m_1^2 & n_1^2 & 2m_1n_1 & 2n_1l_1 & 2l_1m_1 \\
  l_2^2 & m_2^2 & n_2^2 & 2m_2n_2 & 2n_2l_2 & 2l_2m_2 \\
  l_3^2 & m_3^2 & n_3^2 & 2m_3n_3 & 2n_3l_3 & 2l_3m_3 \\
  l_2l_3 & m_2m_3 & n_2n_3 & m_2n_3 + m_3n_2 & n_2l_3 + n_3l_2 & m_2l_3 + m_3l_2 \\
  l_3l_1 & m_3m_1 & n_3n_1 & m_3n_1 + m_1n_3 & n_3l_1 + n_1l_3 & m_3l_1 + m_1l_3 \\
  l_1l_2 & m_1m_2 & n_1n_2 & m_1n_2 + m_2n_1 & n_1l_2 + n_2l_1 & m_1l_2 + m_2l_1 
\end{bmatrix} \]  

be transformation tensor of the resistivity from the principle coordinate \(O(X,Y,Z)\) to an arbitrary coordinate \(O(X^*,Y^*,Z^*)\), then Eq. 3.20 becomes

\[ \rho' = \alpha \rho \]  

(3.23)

As described in the Eq. 2.12, the relationship between \(\rho\) and the piezoresistance tensor \(\pi\) and stress tensor \(\sigma\) is given by

\[ \rho = \rho_0(1 + \pi \sigma) = \rho_0(1 + \Xi) \]  

(3.24)

where

\[ \Xi = \frac{\delta \rho_{ij}}{\rho_{ij}} = \pi \sigma \]  

(3.25)

is relative variant component of the resistivity tensor under strain. Transforming the coordinate system to an arbitrary coordinate, one obtains

\[ \Xi' = \pi' \sigma' \Rightarrow \alpha \Xi = \pi' \alpha \sigma \Rightarrow \Xi = \alpha^{-1} \pi' \alpha \sigma \]  

(3.26)

Then comparing Eq. 3.25 and Eq. 3.26 we derive the relationship between the piezoresistance tensors in the principle and arbitrary coordinates as

\[ \pi = \alpha^{-1} \pi' \alpha \quad \text{or} \quad \pi' = \alpha \pi \alpha^{-1} \]  

(3.27)

where \(\alpha^{-1}\) is the inverse matrix of the transformation tensor of the resistivity. For the sake of simplicity, when only the rotation along the Z-axis is considered \((\phi \neq 0, \phi \neq 2\pi)\),
\( \psi = 0, \theta = 0 \), then the "lmn" matrix is

\[
\begin{bmatrix}
l_1 & m_1 & n_1 \\
l_2 & m_2 & n_2 \\
l_3 & m_3 & n_3
\end{bmatrix} = \begin{bmatrix}
cos \phi & sin \phi & 0 \\
-sin \phi & cos \phi & 0 \\
0 & 0 & 1
\end{bmatrix}
\]

(3.28)

The transformation matrix \( \alpha \) and its inverse \( \alpha^{-1} \) would be

\[
\alpha = \begin{bmatrix}
cos^2 \phi & sin^2 \phi & 0 & 0 & 0 & 2sin \phi cos \phi \\
sin^2 \phi & cos^2 \phi & 0 & 0 & 0 & -2sin \phi cos \phi \\
0 & 0 & 1 & 0 & 0 & 0 \\
0 & 0 & 0 & cos \phi & -sin \phi & 0 \\
0 & 0 & 0 & sin \phi & cos \phi & 0 \\
-sin \phi cos \phi & sin \phi cos \phi & 0 & 0 & 0 & cos^2 \phi - sin^2 \phi
\end{bmatrix}
\]

(3.29)

\[
\alpha^{-1} = \begin{bmatrix}
cos^2 \phi & sin^2 \phi & 0 & 0 & 0 & -2sin \phi cos \phi \\
sin^2 \phi & cos^2 \phi & 0 & 0 & 0 & 2sin \phi cos \phi \\
0 & 0 & 1 & 0 & 0 & 0 \\
0 & 0 & 0 & cos \phi & sin \phi & 0 \\
0 & 0 & 0 & -sin \phi & cos \phi & 0 \\
-sin \phi cos \phi & -sin \phi cos \phi & 0 & 0 & 0 & cos^2 \phi - sin^2 \phi
\end{bmatrix}
\]

(3.30)

Substituting Eq. 3.29 and Eq. 3.30 into Eq. 3.27, the piezoresistance coefficients in an arbitrary coordinate is

\[
\pi'_{ij} = \sum_{k,l=1}^{6} \alpha_{ik} \pi_{kl} \alpha^{-1}_{lj}
\]

(3.31)
According to Toriyama [5], the piezoresistive coefficient tensor for hexagonal SiC polytypes in the principle coordinate is

\[
\Pi = \begin{bmatrix}
\pi_{11} & \pi_{12} & \pi_{13} & 0 & 0 & 0 \\
\pi_{12} & \pi_{11} & \pi_{13} & 0 & 0 & 0 \\
\pi_{31} & \pi_{31} & \pi_{33} & 0 & 0 & 0 \\
0 & 0 & 0 & \pi_{44} & 0 & 0 \\
0 & 0 & 0 & 0 & \pi_{44} & 0 \\
0 & 0 & 0 & 0 & 0 & \pi_{11} - \pi_{12}
\end{bmatrix}
\] (3.32)

It can be seen that, the piezoresistive coefficient tensor has five independent components (i.e. \(\pi_{11}, \pi_{12}, \pi_{13}, \pi_{31}, \) and \(\pi_{33}\)). Subsequently, each component \(\pi'_{ij}\) of the \(\pi'\) matrix including the longitudinal, transverse, and shear piezoresistive coefficients in an arbitrary coordinate can be given as

**Longitudinal piezoresistive coefficient**

\[
\pi_l = \pi'_{11} = \sum_{i,j=1}^{6} \alpha_{1i}\Pi_{ij}\alpha_{j1}^{-1}
\]

\[
= \pi_{11}(\alpha_{11}\alpha_{11}^{-1} + \alpha_{12}\alpha_{21}^{-1} + \alpha_{16}\alpha_{61}^{-1}) + \pi_{12}(\alpha_{11}\alpha_{21}^{-1} + \alpha_{12}\alpha_{11}^{-1} - \alpha_{16}\alpha_{61}^{-1})
\]

\[
+ \pi_{13}(\alpha_{11}\alpha_{31}^{-1} + \alpha_{12}\alpha_{31}^{-1} + \alpha_{13}\alpha_{11}^{-1} + \alpha_{13}\alpha_{21}^{-1} + \alpha_{33}\alpha_{13}^{-1} + \alpha_{33}\alpha_{33}^{-1} + \pi_{44}(\alpha_{14}\alpha_{41}^{-1} + \alpha_{15}\alpha_{51}^{-1})
\]

\[
= \pi_{11}(l_1^4 + m_1^4 + 2l_1^2m_1^2 + \pi_{12}(l_1^2m_1^2 + l_1^2m_1^2 - 2l_1^2m_1^2) + 2\pi_{13}(l_1^2n_1^2 + m_1^2n_1^2)
\]

\[
+ \pi_{33}n_1^4 + 2\pi_{44}(m_1^2n_1^2 + n_1^2m_1^2)
\]

\[
= \pi_{11}(\sin^2\phi + \cos^2\phi)^2
\]

\[
= \pi_{11}
\] (3.33)
Table 3.1 indicates the isotropic piezoresistance in the (0001) plane of 4H-SiC, which is useful for the design and fabrication of sensing devices. In contrast to the anisotropy
## Table 3.1: Comparison of three arbitrary PZR coefficients in cubic lattices (e.g. Si, 3C-SiC) and a hexagonal lattice (i.e. 4H-SiC, this work).

<table>
<thead>
<tr>
<th></th>
<th>Cubic (Si, 3C-SiC) [6, 7]</th>
<th>Hexagonal (4H-SiC) (this work)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\pi_{11}'$</td>
<td>$\pi_{11} - 2(\pi_{11} - \pi_{12} - \pi_{44})(l_1^2m_1^2 + l_1^2n_1^2 + m_1^2n_1^2)$</td>
<td>$\pi_{11}$</td>
</tr>
<tr>
<td>$\pi_{12}'$</td>
<td>$\pi_{12} + (\pi_{11} - \pi_{12} - \pi_{44})(l_1^2l_2^2 + m_1^2m_2^2 + n_1^2n_2^2)$</td>
<td>$\pi_{12}$</td>
</tr>
<tr>
<td>$\pi_{16}'$</td>
<td>$2(\pi_{11} - \pi_{12} - \pi_{44})(l_1^2l_2 + m_1^2m_2 + n_1^2n_2)$</td>
<td>0</td>
</tr>
</tbody>
</table>

of the PZR effect in several common semiconductors (e.g. Si, 3C-SiC), the piezoresistance in 4H-SiC exhibit an isotropic property, which is more favourable for the design and fabrication of mechanical sensors, as the need for choosing the optimal orientation is no longer needed. As a result, high sensitivity can be achieved regardless of the arrangement of devices in wafers as well as the number of devices per wafer can be significantly increased then the cost of sensing devices can be reduced.

### 3.1.2 Experimental methods

#### 3.1.2.1 Strain inducing method

To investigate the piezoresistive effect in p-type 4H-SiC, a simple and well-established bending beam method to induce uniaxial tensile strain into the piezoresistors was utilised. This method has been used in a considerably large number of studies charactering the piezoresistive effect of semiconductors [7–18]. The configuration of the bending cantilever method is illustrated in Fig. 3.3. In this experimental setup, one end of the SiC cantilever is fixed by a clamp, while the other end is deflected downward by varying weights. When applying a weight at the free end, a uniaxial strain is induced into the SiC resistors lying on the top surface of the substrate. By conducting a basic mechanical analysis, the optimal position of SiC resistors is near the clamped end of the cantilever in order to maximize induced strain onto SiC piezoresistors. This will be clarified in the following section.

The bi-layered bending beam model is used to estimate the induced strain onto 4H-SiC layer. The schematic sketch of the bi-layers SiC is illustrated in Fig. 3.3(b) where
the lateral strain of the SiC piezoresistor can be given by [7, 19, 20]:

$$\varepsilon_{4H}(x) = -\frac{F}{wD_1}(L_1 + L_2 - x)t_n$$  \hspace{1cm} (3.36)

where \(F\) is the applied force; \(t_n\) is the distance from neutral axis to the SiC layer; \(w\) and \(L_1\) are the width and length of the cantilever, respectively; \(L_2\) is the distance between two ends of the cantilever and the SiC piezoresistor; the bending modulus per unit width \(D_1\) is given by:

$$D_1 = \frac{E_1^2t_1^4 + E_2^2t_2^4 + 2E_1E_2t_1t_2(2t_1^2 + 2t_2^2 + t_1t_2)}{12(E_1t_1 + E_2t_2)}$$  \hspace{1cm} (3.37)

where \(E_1\) is the Young modulus of the SiC substrate, and \(E_2\) is the Young modulus of the top 4H-SiC layer; \(t_1\) and \(t_2\) are the thickness of the SiC cantilever and piezoresistor.
Figure 3.4: a) The bending beam mechanical model; b) Strain distribution in the cross-sectional of SiC beam. c) Strain transferred onto piezoresistors lying on the top surface of the beam. The strain induced is independent on the width of piezoresistors.
The strain induced finite element analysis (FEA) using COMSOL™ Multiphysics was conducted to verify the calculation, as shown in Fig. 3.4. As such, the strain was effectively transferred onto the 1 µm thin 4H-SiC film lying on the top surface of the SiC beam. The strain transferred onto piezoresistors with different widths were found almost identical and uniform (Fig. 3.4(c)). Therefore, it can be concluded that the strain induced to the piezoresistors is independent on the width, which is in the tens-of-µm range.

3.1.2.2 Measurement of piezoresistive effect at high temperatures

The piezoresistance of p-type 4H-SiC at high temperatures was investigated by the experimental setup shown in Fig. 3.5. The 4H-SiC beams were placed in a temperature-controlled stage in an enclosed chamber (i.e. Linkam™ HFS600E-PB2). The temperature of the probe chamber was precisely regulated with a tolerance of ±0.1°C, which minimizes the deviation of the resistance change by the temperature variation. Figure 3.5 depicts the bending experiment in which one end of the 4H-SiC beam was fixed on the hot plate by clamps while the other end was deflected by a static force.

Figure 3.5: Characterization of the piezoresistive effect of 4H-SiC micro structure at high temperatures in a Linkam™ hotplate.
3.2 Device development

3.2.1 Fabrication process

3.2.1.1 Micromachining

To develop bulk 4H-SiC wafers, high-quality epitaxial SiC layers have to be homoepitaxially grown on bulk SiC substrates [21]. In terms of large-scaled manufacturing, 4H-SiC wafers are typically cut perpendicular to the \( c \)-axis with a slight off-cut angle of up to 8\(^\circ\) towards [11\( \bar{2} \)0] or [1\( \bar{1} \)00] orientations, in order to reduce basal plane dislocations in the SiC epitaxial layers. For 4H-SiC wafers with a large diameter of up to 100 mm, the chosen off-cut angle in the wafer manufacturing is 4\(^\circ\) to reduce the material loss.

The 4H-SiC (0001) wafers used in this thesis was purchased from Ascatron™. The wafers have 4\(^\circ\) off-cut surface from the basal plane (0001) (or perpendicular to the \( c \)-axis) towards the [1\( \bar{1} \)20] orientation. The wafer’s diameter is 100 mm with a 1-\( \mu \)m-p-type 4H-SiC epitaxial layer lying on a 1-\( \mu \)m-n-type layer and the substrate is low doped 4H-SiC with a thickness of 350 \( \mu \)m, as shown in Fig. 3.6(a). The doping concentrations for p-type and n-type epitaxial layers are both \( 1 \times 10^{18} \) \( \text{cm}^{-3} \). A n-type buffer layer with a thickness of 0.8 \( \mu \)m was also grown prior to the growth of p and n layers.
The inductive coupled plasma (ICP) etching and lithography processes were performed at Queensland micro- and nano technology centre. Figure 3.7 summarizes the fabrication process of the p-type 4H-SiC devices used in this thesis. The process started with a standard wafer cleaning procedure then spin coating/developing 4.3 \( \mu \text{m} \)-thick AZ9245 photoresist by a standard lithography process to create a protective mask for the subsequent etching of the p-type layer (step (2) & (3)). The ICP etching was performed to etch p-type 4H-SiC using a STS™ etcher (step (4)). The plasma etching employed HCl at a low pressure of 2 mTorr and the wafer was continuously cooled down by back-side gas cooling in the etching chamber. The etch rate was measured at approximately 100 nm/min and the final etched depth of 1.3 \( \mu \text{m} \) was achieved. This ensures that the 1 \( \mu \text{m} \) p-type layer was thoroughly etched in the designated area; then mesa square-shape structures were formed on the p-type layer. Titanium (Ti) and aluminium (Al) metal layers were subsequently deposited.
to form a contact for the sensor, using a SNS™ sputterer with the thickness of each layer being 100 nm (step 5). Next, a second photoresist mask was formed on top of the as-etched p-type 4H-SiC patterns (step 6), followed by a wet etching of Ti/Al to create the designated metal contact for subsequent electrical measurements (step 7). An annealing process at high temperature up to 1000°C was conducted to obtain a good ohmic contact. Finally, the wafer was diced into 30 mm × 3 mm × 0.35 mm beams for the bending beam measurement (step 8).

Table 3.2: Fabrication process of 4H-SiC micro structures

<table>
<thead>
<tr>
<th>Step</th>
<th>Task</th>
<th>Process</th>
</tr>
</thead>
</table>
| 1 | SiC resistor | - Spin coating photoresist (AZ9245)  
- Baking photoresist at 110°C for 60 sec  
- Aligning the first mask  
- Post bake the photoresist at 120°C for 120 sec  
- Photoresist development  
- Plasma etching SiC by HCl gas (flow rate 500 sccm at 2 mTorr)  
- Removing the photoresist by acetone and IPA |
| 2 | Metallisation | - Sputtering titanium & aluminium  
(∼100 nm each)  
- Spin coating photoresist (AZ6612)  
- Baking photoresist at 100°C for 60 sec  
- Aligning the second mask  
- Post bake the photoresist at 110°C for 60 sec  
- Photoresist development  
- Etching Al by H₃PO₄ and Ti by HF & H₂O₂ mixture  
- Removing the photoresist by acetone and IPA |
| 3 | Dicing | - Spin coating another photoresist for protection  
- Dicing SiC wafer into designated trip  
- Removing the photoresist by acetone and IPA |

Table 3.2 summary the specified conditions used in the fabrication of 4H-SiC micro structures including patterning SiC resistors, patterning metal contacts, and dicing into beam-shaped samples.
The laser scribing process of the 4H-SiC pressure sensor was carried out at Stanford University. The square-shaped diaphragms were formed by scribing the back side of each chip (step 4) by a diode-pumped Nd/YVO₄ laser with a peak power of up to 1.5kW and the average scribing power was 1-3W (Fig. 3.8). The details of laser scribing process can be found elsewhere [22]. First, the 4H-SiC sample (with the prefabricated piezoresistor) was placed in the chamber to align the laser focus. Subsequently, the scribing process was conducted on the back of the 4H-SiC chip (engraving on the side without the piezoresistor). The material was ablated layer-by-layer using the laser beam with the cross-hatch patterning until the desired depth is achieved. The total time for the laser scribing procedure was just approximately 25 min. The scribing time is far less than that of other etching processes for bulk SiC materials (e.g. inductive plasma etching (ICP), deep reactive-ion etching (DRIE)) which could take many hours or even impractical to conduct for the etch depth of hundreds of micrometres. It should be noted that owing to the transparency of the SiC wafer, the alignment of back side scribing to the pre-fabricated piezoresistor in
the front side was made straightforward. Figure 3.8(c) shows the side-wall and surface roughness of the back side of the diaphragm after the laser scribing process. The final dimensions and thickness of the diaphragms were measured to be $5 \times 5 \text{ mm}^2$ and 70 $\mu$m, respectively.

3.2.2 Metallisation and contact characterisation

3.2.2.1 Metallisation

Studies on the metal Ohmic contacts to 4H-SiC showed that the thickness of Ti/Al deposition layer is crucial for the Ohmic contacts characteristic of p-type 4H-SiC samples [23–28]. The following metallisation process was conducted to obtain good Ohmic contacts to p-type 4H-SiC. To form the metal contact, 100-nm-thick Ti and Al layers were sputtered in a SNS™ Sputterer. Another photolithography step was performed to create the contact patterned mask prior to the wet etching of Al and Ti by an Al etchant (i.e. $\text{H}_3\text{PO}_4$) and a mixture of hydrogen fluoride (HF) and hydrogen peroxide ($\text{H}_2\text{O}_2$), respectively. Subsequently, the wafer was thoroughly cleaned using acetone then deionized water.

3.2.2.2 Ohmic contact and current leakage

Since 4H-SiC is a considerably wide band gap semiconductor, it is expected that after depositing metal contacts, a Schottky barrier would be formed between the p-type and metal layers. This is attributed to the large difference of the work functions of metals and wide band gap semiconductors [27]. Initially, the contact between the Ti/Al and p-type 4H-SiC layers exhibited a rectifying behaviour. After the metallisation, the wafer was annealed by a rapid thermal annealing (RTP) process at $1000^\circ\text{C}$ in nitrogen atmosphere. In the annealing process, the temperature was kept at $1000^\circ\text{C}$ for 3 minutes and the total duration, including heating and cooling, was approximately 1 hour.

Electrical characterisations before and after annealing were performed by current–voltage (I–V) measurements. The I-V characteristic Ti/Al contact annealed to $800^\circ\text{C}$ exhibited a semi-rectifying behaviour. In the I-V curves shown in Fig. 3.9, a linear I-V
Chapter 3 Methodology

characteristic of the sample with Ti/Al contact annealed to 1000°C indicates that good Ohmic contacts were obtained. This means that the required temperature for annealing multi-layer metal contacts of p-type 4H-SiC samples is 1000°C. The measured sheet resistances of 1-µm p-type layer was approximately 20 kΩ/□.

Figure 3.10 shows the current-voltage characteristics versus the current leak through the p-n junction at various temperature from 23°C to 600°C. The measured leakage current was at least three orders of magnitudes smaller then the current flowing in the p-type sensing layer. For example, at an applied voltage of 5V at 600°C, the current in the p-type was approximately 460µA while the leakage current was measured below 10mA. It should also be pointed out that, since p-type 4H-SiC was epitaxially grown on n-type 4H-SiC, the back-to-back p-n diodes would prevent the current from leaking into the substrate, which eliminates the contribution of the n-type 4H-SiC to the measurement of the piezoresistance in p-type 4H-SiC. The very small current leaking to the substrate is a significant advantage of 4H-SiC over other low band gap materials for mechanical sensing at elevated temperatures.
Figure 3.10: Current-voltage characteristics of the p-type piezoresistor with a voltage ranging from -5V to 5V at various temperatures from 23°C to 600°C, showing the excellent Ohmic characteristics of the Ti/Al contact to the p-type piezoresistors. Inset: Schematic sketch of the measurement of the current flowing in p-type layer and through the p-n junction.

Figure 3.11: Micro structures of SiC/metal interfaces of sample with Ti/Al electrodes before annealing (top) and after annealing (bottom). Reprinted with permission from [24].

The mechanism for the Ohmic contact formation in the thermal annealing at 1000°C can be explained as follows. The early stages of annealing of these contacts at 1000°C resulted in the reaction of Ti, Al to form TiAl₂, TiAl₃ and Ti₃Al leaving unreacted C atoms at the Ti/SiC interfaces, and the later stages of annealing resulted in the
reaction of the Ti, SiC and C to form a ternary Ti$_3$SiC$_2$ compound, as shown in Fig. 3.11. The formation of the ternary compound prevented the formation of unreacted C at the SiC/metal interface [24].
Chapter 3 Methodology

Summary

In this chapter, the methodology for the investigation of the piezoresistive effect in p-type 4H-SiC was proposed, including the theoretical analyses and experimental characterisation. The isotropic piezoresistive coefficients in the (0001) plane of p-type 4H-SiC was analytically confirmed using the calculation of hole energy shift under strain and a coordinate transformation. Additionally, in the experimental work, the micro-fabrication process of SiC micro structures and metallisation process to obtain good Ohmic contact were carried out. The experimental setup for the characterisation of the temperature dependence of the piezoresistive effect in p-type 4H-SiC up to 600°C and a laser scribing process to fabricate 4H-SiC diaphragm for pressure sensing were presented.
Chapter 3 Methodology

Bibliography


86
Chapter 3 Methodology


unimorph cantilevers of different nonpiezoelectric/piezoelectric length ratios,”


phology for epitaxial growth on 4° off-axis 4H-SiC substrates,” *J. Cryst. Growth*,

[22] E. H. Ransom, K. M. Dowling, D. Rocca-Bejar, J. W. Palko, and D. G. Senesky,
“High-throughput pulsed laser manufacturing etch process for complex and re-
leased structures from bulk 4H-SiC,” in *2017 IEEE 30th Inter. Conf. on Micro

[23] B. J. Johnson and M. A. Capano, “Mechanism of ohmic behavior of Al/Ti con-
tacts top-type 4H-SiC after annealing,” *J. Appl. Phys.*, vol. 95, pp. 5616–5620,
2004.

[24] R. Konishi, R. Yasukuchi, O. Nakatsuka, Y. Koide, M. Moriyama, and M. Mur-
akami, “Development of Ni/Al and Ni/Ti/Al ohmic contact materials for p-type

J.-M. Bluet, S. Scharnholz, and D. Planson, “Ni–Al ohmic contact to p-type

“The role of nickel and titanium in the formation of ohmic contacts on p-type

[27] L. Fursin, J. Zhao, and M. Weiner, “Nickel ohmic contacts to p- and n-type

on simultaneous formation of ohmic contacts on p- and n- type 4H-SiC using
Ni/Ti/Al ternary system,” in *12th IEEE Inter. Conf. Solid-State Integrated Cir-
cuit Tech. (ICSICT)*, 2014.
Chapter 4

Piezoresistive effect and piezoresistive coefficients in (0001) plane of p-type 4H-SiC

This chapter presents the piezoresistive effect and the isotropic piezoresistance in the (0001) plane in p-type 4H-SiC. The large gauge factors, attributed to the change of valance energy bands upon application of mechanical strain, and the linear relationship between the resistance change versus induced strain demonstrate the potential of p-type 4H-SiC for mechanical sensing applications. The isotropy of the piezoresistance in the basal plane of p-type 4H-SiC is correlated to the isotropic hole energy shift under uniaxial strain. This interesting phenomenon in p-type 4H-SiC is promising for the design and fabrication of mechanical sensors and strain-engineered electronics since high sensitivity and consistent performance can be achieved regardless of the crystallographic orientation. Additionally, it is still useful for the characterisation of the piezo coefficients in other planes.

Statement of contribution to co-authored published papers

This chapter is presented in the form of two peer-reviewed published papers. The bibliographic details of the published papers, including all authors, are:

(J.22) Tuan-Khoa Nguyen, Hoang-Phuoug Phan, Toan Dinh, Jisheng Han, Sima Dimitrijev, Philip Tanner, Abu Riduan Md Foisal, Yong Zhu, Nam-Trung Nguyen

My contribution to the published paper involved:

- Design and fabrication of the samples
- Experimental task
- Data acquisition and analysis
- Writing manuscripts

(Sign) ____________________________ (Date) 30/07/2018

**Tuan-Khoa Nguyen** (First and corresponding author)

(Sign) ____________________________ (Date) 30/07/2018

**Assoc. Prof. Dzung Viet Dao** (Principle supervisor)

(Sign) ____________ (Date) 30/07/2018

**Dr. Yong Zhu** (Principle supervisor)


My contribution to the published paper involved:

- Design and fabrication of the samples
- Experimental task
- Data acquisition and analysis
Chapter 4 Piezoresistive effect and piezoresistive coefficients in (0001) plane of p-type 4H-SiC

- Writing manuscripts

(Sign) ___________________ (Date) 30/07/2018

Tuan-Khoa Nguyen (First and corresponding author)

(Sign) ___________________ (Date) 30/07/2018

Assoc. Prof. Dzung Viet Dao (Principle supervisor)

Appropriate acknowledgements have been stated and included in the papers.

Reuse permission: The contents are reused from


Copyright permission from the publishers to reuse theses papers in this thesis are attached in the following pages.
4.1 Magnitude of piezoresistive effect in p-type 4H-SiC
Title: Experimental Investigation of Piezoresistive Effect in p-Type 4H-SiC
Author: Tuan-Khoa Nguyen
Publication: IEEE Electron Device Letters
Publisher: IEEE
Date: July 2017

Thesis / Dissertation Reuse

The IEEE does not require individuals working on a thesis to obtain a formal reuse license, however, you may print out this statement to be used as a permission grant:

Requirements to be followed when using any portion (e.g., figure, graph, table, or textual material) of an IEEE copyrighted paper in a thesis:

1) In the case of textual material (e.g., using short quotes or referring to the work within these papers) users must give full credit to the original source (author, paper, publication) followed by the IEEE copyright line © 2011 IEEE.
2) In the case of illustrations or tabular material, we require that the copyright line © [Year of original publication] IEEE appear prominently with each reprinted figure and/or table.
3) If a substantial portion of the original paper is to be used, and if you are not the senior author, also obtain the senior author's approval.

Requirements to be followed when using an entire IEEE copyrighted paper in a thesis:

1) The following IEEE copyright/credit notice should be placed prominently in the references: © [year of original publication] IEEE. Reprinted, with permission, from [author names, paper title, IEEE publication title, and month/year of publication]
2) Only the accepted version of an IEEE copyrighted paper can be used when posting the paper or your thesis on-line.
3) In placing the thesis on the author's university website, please display the following message in a prominent place on the website: In reference to IEEE copyrighted material which is used with permission in this thesis, the IEEE does not endorse any of [university/educational entity's name goes here]'s products or services. Internal or personal use of this material is permitted. If interested in reprinting/republishing IEEE copyrighted material for advertising or promotional purposes or for creating new collective works for resale or redistribution, please go to http://www.ieee.org/publications_standards/publications/rights/rights_link.html to learn how to obtain a License from RightsLink.

If applicable, University Microfilms and/or ProQuest Library, or the Archives of Canada may supply single copies of the dissertation.
Experimental Investigation of Piezoresistive Effect in p-Type 4H–SiC

Tuan-Khoa Nguyen, Hoang-Phuong Phan, Toan Dinh, Jisheng Han, Sima Dimitrijev, Philip Tanner, Abu Riduan Md Faisal, Yong Zhu, Nam-Trung Nguyen, and Dzung Viet Dao

Abstract—This letter presents for the first time the piezoresistive effect in p-type 4H–SiC. Longitudinal and transverse p-type 4H–SiC piezoresistors with a doping concentration of $10^{18}\text{cm}^{-3}$ were fabricated along [110] directions. Ni/Al electrodes annealed at 1000 °C showed a good ohmic contact, and then, the longitudinal and transverse gauge factors were found to be as high as 31.5 and −27.3, respectively. The large gauge factors, attributed to the change of valance energy bands upon application of mechanical strain, and the linear relationship between the resistance change versus induced strain demonstrate the potential of p-type 4H–SiC for mechanical sensing applications.

Index Terms—4H-SiC, MEMS mechanical sensor, piezoresistive effect, silicon carbide.

I. INTRODUCTION

O

VER the past four decades, silicon (Si) has been one of the main materials used in MEMS sensors [1], [2]. However, Si based sensors are not suitable for a range of applications where harsh conditions, including high temperature, high pressure, strong electric fields, extremely high shock or intense vibration and aggressive chemicals exist [3], [4]. On the other hand, the superb mechanical properties of silicon carbide (SiC), along with its electrical stability at high temperatures, offer new possibilities of developing MEMS sensors for such applications [5], [6]. 4H-SiC is one of the most favorable polytypes for MEMS devices owing to its excellent properties [7] and commercial availability.

The piezoresistive effect in SiC has been extensively investigated for mechanical sensing applications [8], [9]. A cost-effective approach to develop SiC piezoresistive devices is to epitaxially grow 3C-SiC on Si wafer ($\beta$-SiC) in which SiC would be the sensing layer, while Si acts as the substrate. This can take advantage of the wide availability and low cost of Si wafers. An early study on the piezoresistive effect in n-type 3C-SiC was conducted by Shor et al. with a negative gauge factor (GF) of −31.8 for a $10^{16}$-$10^{17}$ cm$^{-3}$ doping concentration [10]. Phan et al. reported a high GF of 30.3 in $5 \times 10^{18}$ cm$^{-3}$ doped p-type 3C-SiC [11]. Furthermore, the GFs were found to be stable at the temperature ranging from 300 to 573°K [12]. In addition, hexagonal SiC polytypes ($\alpha$-SiC), such as 4H-SiC and 6H-SiC, have attracted an increasing research interest since the use of these polytypes offers the possibility of making SiC-only devices. A GF of $3.3 \times 10^{18}$ cm$^{-3}$ doped 6H-SiC has been reported as high as −29.4, and the device could be used up to 250°C [13]. By scaling down the sizes of piezoresistors to nanometer scale, giant piezoresistances have been realized owing to the quantum confinement effect in SiC nanostructures [14], [15]. The piezoresistive effect of 4H-SiC is also of interest since this polytype can be integrated into electronic components in the same platforms. Akiyama et al. reported the piezoresistive effect in a highly doped of $1.5 \times 10^{19}$ cm$^{-3}$ n-type 4H-SiC with longitudinal and transverse GFs of −10 and 20.8, respectively [16]. A comprehensive review of the piezoresistive effect in different SiC polytypes can be found elsewhere [4].

Although the piezoresistive effect of other SiC polytypes has been reported in a large number of studies, such effect in p-type 4H-SiC, an indispensable material for MEMS and power electronics devices, has not been elucidated yet. This letter presents the first experimental investigation of the piezoresistive effect in p-type 4H-SiC with significantly large GFs. The three-point bending method was utilized to obtain the Young’s modulus of 4H-SiC, while thermally annealed Ni/Al thin film was used to form Ohmic contact for 4H-SiC piezoresistors. The experimental results show that under uniaxial strains along [1100] orientation, the GFs of p-type 4H-SiC were found to be 31.5 and −27.3 for the longitudinal and transverse [1100] directions, respectively. The piezoresistance of p-type 4H-SiC also exhibited an excellent linearity in the induced strain range of 0 to nearly 200 με. These results indicate that p-type 4H-SiC is a promising candidate for mechanical sensors.

II. RESULTS AND DISCUSSION

The 4H-SiC wafer used in this study was purchased from Ascatron™ with a 1-μm p-type epitaxial layer grown on top of a 1-μm n-type epitaxial layer and n-type SiC substrate. A cross-sectional view of the epitaxial p-type 4H-SiC layer on n-type 4H-SiC substrate is illustrated in Fig. 1 (a). The p-type layer, with aluminum dopant, and n-type layer, with nitrogen dopant, both have a carrier concentration of $10^{18}$ cm$^{-3}$. 50-nm-thick nickel and 300-nm-thick aluminum layers were...
deposited on the top of the p-type layer. Figure 1 (b) shows the SEM image of an array of 200 μm-long and 1000 μm-wide SiC resistors. Since 4H-SiC is a considerably wide band gap semiconductor, it is expected that after depositing metal contacts, a Schottky barrier would be formed between the p-type and metal layers. This is attributed to the large difference of the work functions of metals and wide band gap semiconductors [17]. Initially, the contact between the Ni/Al and p-type 4H-SiC layers exhibited a rectifying behavior. However, by conducting a high temperature thermal annealing in nitrogen atmosphere, an Ohmic contact was formed in the annealed samples (Fig. 1 (c)). In the annealing process, the temperature was kept at 1000°C for 2 minutes and the total duration, including heating and cooling, was approximately 1 hour. The measured sheet resistances of 1-μm p-type layer was 22.9 kΩ/□ and the contact resistance was approximately 10⁻³ Ωcm². The measurement of the leakage current from p-type layer to the substrate, shown in Fig. 1 (d), indicates that the leakage current was less than 0.05% of the current flowing in the SiC resistor at an applied DC voltage of up to 2V. It should also be pointed out that, since p-type 4H-SiC was epitaxially grown on n-type 4H-SiC, the back-to-back p-n diodes would prevent the current from leaking into the substrate, which eliminates the contribution of the n-type 4H-SiC to the measurement of the piezoresistance in p-type 4H-SiC. The finite element analysis (FEA), shown in Fig. 1 (e), demonstrates the distribution of electric field between two parallel electrodes. Evidently, the electric field mainly distributes between the two electrodes (more than 97%). Consequently, the influence of the currents flowing in different directions other than [1100] direction can be negligible.

The Young’s modulus of the 4H-SiC was characterized using three-point bending experiment shown in Fig. 2 (a)-(b). From the relationship between the deflection δ of the SiC beam and the applied force F, the Young’s modulus E of 4H-SiC can be calculated by [18]: 
$$E = \frac{FL^3}{4wt^3\delta},$$
where L is the distance between two supporting points, w and t are the width and thickness of the beam, respectively. Accordingly, the Young’s modulus was found to be 503.7 GPa. To investigate the piezoresistive effect in p-type 4H-SiC, we utilized a simple and well-established bending beam method to induce uniaxial tensile strain into the piezoresistors as shown Fig. 3 (a). This method has been used in a considerably large number of studies characterizing the piezoresistive effect of semiconductors [11], [13], [16], [19]–[21]. 4H-SiC cantilevers, which are 45-mm-long, 5-mm-wide, and 0.35-mm-thick, were fixed at one end using an electrically insulated clamp, while the other end was deflected downwards by static forces. Let M be the bending moment generated by an external force, then the strain induced into the 4H-SiC piezoresistor is given by: 
$$\epsilon = (Ml)/(EI),$$
where l and t are the inertial moment and thickness of the SiC cantilever, respectively. From the obtained Young’s modulus, for applied forces varying from 0 to 0.3 N, the strain induced into the 4H-SiC piezoresistor ranged from 0 to 182 με. This was in good agreement with the FEA result using COMSOL® Multiphysics (Fig. 2 (c)).

The GF of a p-type 4H-SiC piezoresistor is given by: 
$$GF = (AR/R)/\epsilon,$$
where AR is the resistance change of the unstrained resistance R. Figure 3 (b) plots the relationship between the resistance change of 4H-SiC piezoresistors versus applied strain using the four-point probe measurement. Evidently, the measured resistance of the 100 μm-long 4H-SiC piezoresistors exhibited a good linearity under induced strain varying from 0 to 182 με with linear regressions of 31.5 and −27.3 for longitudinal and transverse GFs, respectively. Table I lists the longitudinal and transverse GFs in [1100] direction with the length ranging from 100 to 300 μm. It can be seen that these measured GFs are comparable with the previous results of 3C-SiC [10]–[12], 6H-SiC [13], and n-type 4H-SiC [16]. The similarity of the
Fig. 3. (a) Schematic sketch of the bending experiment to apply uniaxial strain to the 4H-SiC piezoresistors. The distance from the piezoresistors to the free end of the SiC cantilever is 38 mm. (b) The linear relationship between the relative resistance change ($\Delta R/R_o$ of 100 $\mu$m-long piezoresistors versus induced strain, varying from 0 to 182 $\mu$ε. The unstrained resistance was 2.08 k$\Omega$. (c) The output voltage versus strain varying from 0 to 182 $\mu$ε (the red line) and ON-OFF constant strain at an induced strain of 60.6 $\mu$ε (the blue line). Inset: Wheatstone bridge circuit.

measured GFs in SiC piezoresistors with different dimensions, listed in Table I, indicates that the strain induced on the top surface of the substrate was effectively transferred to the p-type layer. This was also consistent with the FEA of the strain induced to the p-type layer under bending (Fig.2 (c)).

To demonstrate the real-time data acquisition, the resistance change was converted to the output voltage of the Wheatstone bridge circuit (Fig.3 (c): Inset). In this measurement, the DC bias input voltage was set to be 0.1V, therefore the Joule heating effect in the piezoresistor can be negligible [22]. Initially, the strain was increased from 0 to 182 $\mu$ε, showing an increase in the output voltage of the p-type 4H-SiC resistor (Fig.3 (c)). The output voltage decreased when decreasing the applied strain, then returned to 0 when the load was completely removed. A constant strain was also repeatedly applied then the piezoresistance of p-type 4H-SiC exhibited a good repeatability. These results remained almost the same in repeated measurements on different days. The repeatability and linearity of the piezoresistance of the as-fabricate p-type 4H-SiC piezoresistors indicate the potential for strain sensing applications.

The positive longitudinal GF in [1100] direction exhibits a decrease in electrical conductance of p-type 4H-SiC under tensile strain, while the negative value of the transverse GF in this crystallographic orientation shows a positive trend. The electrical conductivity of p-type 4H-SiC can be simplified by [23], [24]:

$$\sigma = N_{hh} q \times \mu_{hh} + N_{lh} q \times \mu_{lh}$$

where $N$ is the hole concentration, $q$ is the unit charge, $\mu$ is the hole mobility, and the “$hh$” and “$lh$” subscripts stand for heavy holes and light holes, respectively. Under strain, the energy levels of heavy and light holes change, leading to the re-population of holes between these two bands. The decrease in the conductance of p-type 4H-SiC, under tensile strain along [1100] orientation, indicates that more charge carriers moved from the light hole band (with higher mobility) to the heavy hole band (with lower mobility). This also implies that the heavy hole band shifts upward to lower energy level, while the light hole band moves downward to higher energy level. On the other hand, the increasing conductance in the transverse direction under tensile strain shows that the heavy hole band moves downwards while the light hole band moves upwards. Therefore, the mobility of p-type 4H-SiC can be significantly enhanced by utilizing the transverse strain.

### III. Conclusion

The piezoresistive effect of p-type 4H-SiC in [1100] direction was characterized in which the longitudinal GF has a positive value of 31.5 while the transverse GF has a negative value of $-27.3$. The significantly high GFs found in p-type 4H-SiC demonstrate the potential for mechanical sensing applications. Additionally, it is confirmed that the mobility of p-type 4H-SiC based devices can be improved by employing strain engineering in the transverse direction.

### ACKNOWLEDGMENT

The authors would like to thank Mr. Khanlou (Griffith University) for the assistance in the Young’s modulus measurement and Asst. Prof. Minh Le (Tohoku University, Japan) for the helpful discussion about the thermal annealing process.
REFERENCES


4.2 Orientation dependence of piezoresistive effect in (0001) plane of p-type 4H-SiC
Isotropic piezoresistance of p-type 4H-SiC in (0001) plane

Tuan-Khoa Nguyen,1,a) Hoang-Phuong Phan,1 Toan Dinh,1 Toshiyuki Torigaya,2 Koichi Nakamura,3,4 Abu Riduan Md Foisal,1 Nam-Trung Nguyen,1 and Dzung Viet Dao1,5

1Queensland Micro- and Nanotechnology Centre, Griffith University, 170 Kessels Road, Brisbane, Queensland 4111, Australia
2Department of Mechanical Engineering, College of Science and Engineering, Ritsumeikan University, 1-1-1 Noji-higashi, Kusatsu, Shiga 525-8577, Japan
3Center for the Promotion of Interdisciplinary Education and Research, Kyoto University, Kyoto 615-8540, Japan
4Department of Materials Science and Engineering, Egypt-Japan University of Science and Technology, New Borg El-Arab, Alexandria 21934, Egypt
5School of Engineering and Built Environment, Griffith University, Parklands Drive, Gold Coast, Queensland 4215, Australia

(Received 25 April 2018; accepted 17 June 2018; published online 3 July 2018)

In this work, the isotropic piezoresistance in the (0001) plane of p-type 4H-SiC was discovered by means of the hole energy shift calculation and the coordinate transformation. These results were also confirmed by the measurement of the piezoresistance using a bending beam method. The fundamental longitudinal and transverse piezoresistive coefficients $\pi_{11}$ and $\pi_{12}$ were found to be $6.43 \times 10^{-11}$ Pa$^{-1}$ and $5.12 \times 10^{-11}$ Pa$^{-1}$, respectively. The isotropy of the piezoresistance in the basal plane of p-type 4H-SiC is attributed to the isotropic hole energy shift under uniaxial strain. This interesting phenomenon in p-type 4H-SiC is promising for the design and fabrication of mechanical sensors and strain-engineered electronics since high sensitivity and consistent performance can be achieved regardless of the crystallographic orientation. Published by AIP Publishing.

https://doi.org/10.1063/1.5037545

Since the pioneering discovery by Smith,1 the large change in resistivity upon applied stress/strain in semiconductors, namely, the piezoresistive (PZR) effect, has attracted great attention, reflecting in a vast number of studies and applications for mechanical sensing and strain engineering.2–6 Such an effect could also be magnified in nanoscale devices,7–9 which has been employed in highly sensitive and miniaturized Si-based devices [e.g., accelerometers, pressure sensors, and atomic force microscopy (AFM) probes]. Si remains the dominant material for the PZR effect in mechanical sensing. However, Si exhibits several limitations due to its intrinsic physical properties, such as the plastic deformation at a temperature above 500 °C and the instability of electrical conduction in high temperature operations.10 To push the sensing devices beyond their current limits, recent advances in the growth and fabrication technologies enable the development of SiC-based devices, which can inherit the current readiness of the Si technology and be utilized in harsh environmental applications. The strong covalent Si-C bond and wide energy bandgap of SiC lead to superior properties such as high mechanical strength, high shock resistance, chemical inertness, and thermal/electronic stability.11,12 Furthermore, its good compatibility with micro-machined fabrication processes positions SiC as a promising material for mechanical sensing in harsh environments.13,14

The PZR effect and strain-induced mobility enhancement of Si have been well established.15 As such, the piezoresistance of Si was graphically presented in the works of Kanda.16,17 in which an anisotropic characteristic has been realized. Subsequently, numerous studies have been dedicated to the understanding of the phenomenon in different SiC polytypes. For instance, the fundamental PZR coefficients in arbitrary crystallographic orientations of 3C-SiC have been revealed with a relatively high shear PZR coefficient.18 For hexagonal lattices, a high piezoresistance was realized in highly doped n-type and p-type 6H-SiC.19 By conducting the tensor transformation in n-type 6H-SiC, a wurtzite crystal, it was suggested that in the (0001) plane, the anisotropic part of the piezoresistance vanishes, whereas only the isotropic component remains.20 The piezoresistivity of n-type 4H-SiC was theoretically investigated by the first principles simulation, indicating the significant gauge factor (GF) at either room or high temperature.21

In contrast to heterostructure devices, the use of bulk 4H-SiC can eliminate the thermal expansion mismatch between the sensing layer and the substrate (which are made of different materials), pushing the temperature limit of the sensing devices which can work in extreme environments. Moreover, 4H-SiC possesses a very high energy bandgap of 3.23 eV (three times higher than that of Si), which significantly minimizes the generation of thermally activated electron-hole pairs, contributing to the good reliability of 4H-SiC based devices in harsh environments. Although many efforts have been made for the investigation of the PZR effect in SiC, to date, the fundamental PZR coefficients of 4H-SiC, one of the most important SiC polytypes for mechanical sensors and high-power electronics devices, have not been elucidated in terms of either theoretical analysis or experimental verification. Understanding such a phenomenon in 4H-SiC not only optimizes the sensors’ sensitivity but also enhances the devices’ mobility by means of strain engineering. Therefore, this paper aims to theoretically and experimentally investigate the
fundamental PZR coefficients of 4H-SiC in the (0001) plane. As such, the longitudinal and transverse PZR coefficients were found to be $6.43 \times 10^{-11}$ Pa$^{-1}$ and $-5.12 \times 10^{-11}$ Pa$^{-1}$, respectively. Additionally, the theoretical analysis by calculating hole energy shift and coordinate transformation is in good agreement with the experimental result of the isotropic and large piezoresistances in the (0001) plane of p-type 4H-SiC. This finding is useful for the design and fabrication of mechanical sensors since the anisotropy and complexity of the PZR effect are eliminated.

Figure 1(a) shows a scanning electron microscopy (SEM) image of p-type 4H-SiC piezoresistors aligned in the (01 10) orientation. The piezoresistors were located on numerous rectangular samples which were arranged in various orientations in a 350 $\mu$m-thick 4-in. epitaxial 4H-SiC (0001) wafer, as shown in Fig. 1(b). The wafer has a 1-$\mu$m-p-type epitaxial layer (with the hole concentration of $10^{18}$ cm$^{-3}$) lying on a $10^{18}$ cm$^{-3}$ doped n-type 4H-SiC layer to form a p-n junction. The substrate was low doped 4H-SiC. The mesa p-type piezoresistors were fabricated by conventional lithography and inductive plasma etching (ICP). It should be noted that the final etch depth of the p-type layer was 1.3 $\mu$m, which ensures that the designated areas in the layer are completely etched. To form the contact, titanium and aluminum layers, with the same thickness of 100 nm, were subsequently sputtered on the wafer followed by wet etching using Al and Ti etchants. The piezoresistors have the length and width varying from 20 $\mu$m to 200 $\mu$m and 5 $\mu$m to 80 $\mu$m, respectively. High temperature annealing was performed to obtain a good Ohmic contact from Ti/Al to p-type 4H-SiC, showing linear current-voltage characteristics in a voltage range from $-2$ V to 2 V. The detailed annealing condition was reported elsewhere in which the metal contact was annealed up to 1000$^\circ$C in a nitrogen atmosphere. Finally, the wafer was diced into 30 $\times$ 3 mm$^2$ beams for the bending experiment.

The current leakage through the p-n junction between p-type and n-type layers was measured and found to be four orders of magnitude smaller than the current flowing in the p-type piezoresistors. This is attributed to the robust p-n junction acting as a back-to-back diode which prevents the electric current leaking to the n-type layer. Therefore, it can be concluded that only the p-type layer contributed to the PZR measurement.

The straining configuration using a bending beam method to induce uniaxial strain/stress to the piezoresistors is illustrated in Fig. 2(a). The piezoresistors were designed in the vicinity of the clamping area to maximize the induced strain. Since the p-type layer is much thinner than the total thickness of the beam (i.e., 1 $\mu$m vs 350 $\mu$m), the uniaxial strain would be effectively transferred to the top p-type layer. Let $F$ be the applied force at the free end of the beam, and then, the induced stress can be deduced as $\sigma = 6Fl/bt^2$, where, $l$, $b$, and $t$ are the length, width, and thickness of the 4H-SiC beam, respectively. Additionally, the uniformity of strain induced into the top p-type layer was confirmed using a finite element analysis, which is independent of the width and length of the piezoresistor (see Sec. 1 in the supplementary material).

Figure 2(b) shows the measurement results of the PZR effect of p-type 4H-SiC aligned in various orientations in the (0001) plane at room temperature (i.e., 298 K). The longitudinal and transverse piezoresistors were fabricated in all beams in order to investigate the piezoresistive effect in different orientations in the plane. Subsequently, the relative resistance changes were measured against a varying induced strain from 0 to 557 ppm. The variation of the resistance in either the longitudinal or the transverse piezoresistor is proportional to the increasing strain, indicating a good linearity of the PZR effect in 4H-SiC. The consistent gauge factor (GF) in different orientations exhibits the isotropic piezoresistance of p-type 4H-SiC in the (0001) plane. The small tolerance among these results is attributed to the fact that the source wafer has the 4$^\circ$ off-cut surface towards the (1120) orientation from the ideal basal plane.

The PZR coefficients, typically represented in an arbitrarily oriented coordinate system, can be calculated from a tensor transformation. The resistivity tensor $\rho$ of 4H-SiC is given in the relationship between the electric potential $E$ and current density $J$ in the principal coordinate (i.e., O-xyz) as $E = \rho J$, where $E$ and $J$ are the first rank tensors and $\rho$ is a second rank tensor. By applying a coordinate transformation
from the principal coordinate system to an arbitrary coordinate (i.e., O-x₀y₀z₀) with the rotational matrix \( R \), the relationship between the two resistivity tensors is

\[
q_{0} = \frac{Rq_{1}R}{C_{0}} = aq,
\]

where \( R/C_{0} \) is the inverse of the rotational matrix \( R \) and \( a \) is the transformation matrix of the resistivity tensors from the principal to an arbitrary coordinate. The longitudinal GF in the principal coordinate can be deduced as

\[
G_{ij} = \frac{\Xi_{ij}}{\epsilon} = \pi_{ij}E,
\]

where \( \epsilon \) is the induced uniaxial strain into the piezoresistor and \( E \) is the Young’s modulus of 4H-SiC. The longitudinal GF in the principal coordinate can be deduced as

\[
G_{ij} = \frac{\Xi_{ij}}{\epsilon} = \pi_{ij}E,
\]

where \( \epsilon \) is the induced uniaxial strain into the piezoresistor and \( E \) is the Young’s modulus of 4H-SiC. The longitudinal GF in the principal coordinate can be deduced as

\[
G_{ij} = \frac{\Xi_{ij}}{\epsilon} = \pi_{ij}E,
\]

where \( \epsilon \) is the induced uniaxial strain into the piezoresistor and \( E \) is the Young’s modulus of 4H-SiC.22 The Young’s modulus in the various orientations in the (0001) plane was measured and found to be almost isotropic (see Sec. 2 in the supplementary material).

Figure 3 shows the graphical representation of the arbitrary piezoresistance coefficients \( p_{0}^{11} \) and \( p_{0}^{12} \) in the (0001) plane. It should be noted that these PZR coefficients are almost isotropic in different orientations, indicating the isotropic property of the PZR effect in this plane of 4H-SiC.

To theoretically analyze this interesting property, the coordinate transformation was conducted to deduce the relationship between the PZR coefficients in the principal and arbitrary coordinates, as shown in Table I (see Sec. 3 in the supplementary material for the detailed calculation). The obtained result indicated the isotropic piezoresistance in the (0001) plane of 4H-SiC, which is useful for the design and fabrication of the sensing devices [Eq. (1)].

Figure 4(a) illustrates the first Brillouin zone of 4H-SiC with the highest symmetry points on the axis and on the edge of the hexagonal prism. The piezoresistive effect in 4H-SiC can be further clarified by considering the hole energy shift in the strained crystal structure of its wurtzite lattice in the space group \( C_{6v}^{4} \). The piezoresistance of p-type 4H-SiC is correlated with the hole transfer and conduction mass shift by means of the deformation of the top valence bands under strain.25 As such, a uniaxial stress could raise the light hole (LH) bands

![FIG. 2. (a) Schematic sketch of the bending beam experiment. (b) Fractional change of the resistance versus applied strain in different orientations in the (0001) plane. Inset: Configuration of longitudinal (top) and transverse (bottom) piezoresistors.](image)

![FIG. 3. Graphical representation of the longitudinal and transverse PZR coefficients in the (0001) plane of p-type 4H-SiC. The longitudinal PZR coefficients are positive, whereas the transverse PZR coefficients are negative.](image)

<table>
<thead>
<tr>
<th>TABLE I. Comparison of three arbitrary PZR coefficients in the cubic lattice (e.g., Si and 3C-SiC) and a hexagonal lattice (i.e., 4H-SiC, this work). See Sec. 3 in the supplementary material for ( l_{i}, m_{i}, ) and ( n_{i} (i = 1, 2, \text{and } 3) ).</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cubic (Si, 3C-SiC)²⁵,²⁶,²⁸</td>
</tr>
<tr>
<td>Hexagonal (4H-SiC) (this work)</td>
</tr>
<tr>
<td>( \pi'_{11} )</td>
</tr>
<tr>
<td>( \pi'_{12} )</td>
</tr>
<tr>
<td>( \pi'_{16} )</td>
</tr>
</tbody>
</table>


and simultaneously lower the heavy hole (HH) bands parallel to the stress orientation because of the change in the overlap between the atomic orbitals. Figure 4(b) shows the constant energy surfaces for the three highest valence bands in 4H-SiC.\textsuperscript{24} From top to bottom: Light hole, heavy hole, and spin orbit split-off (SOSO) bands, respectively.

The energy surfaces for three topmost valence bands without strain in 4H-SiC, showing ellipsoidal shapes.\textsuperscript{24} The isotropic piezoresponse can be determined by the hole energy shift of the valence bands at $k = 0$ under strain. According to Bir,\textsuperscript{26} the energy of the three topmost valence bands at $k = 0$ in the unstrained 4H-SiC crystal is

$$E_1^0 = \Delta_1 + \Delta_2,$$

$$E_{2,3}^0 = \frac{\Delta_1 - \Delta_2}{2} \pm \sqrt{\left(\frac{\Delta_1 - \Delta_2}{2}\right)^2 + 2\Delta_3^2},$$

where $\Delta_i (i=1,2,3)$ are the spinor representations for the symmetry points in the Brillouin zone. The first order approximation for the strained band energy $E(k, \epsilon)$ is

$$E_1 = F,$$

$$E_{2,3} = \frac{G + \lambda}{2} \pm \sqrt{\left(\frac{G - \lambda}{2}\right)^2 + \Delta^2},$$

where $\lambda = \sqrt{2}\Delta_1; F = \Delta_1 + \Delta_2 + \lambda + \theta; G = \Delta_1 - \Delta_2 + \lambda + \theta; \lambda = A_1k_x^2 + A_2(k_y^2 + k_z^2) + D_1\epsilon_{xx} + D_2(\epsilon_{xx} + \epsilon_{yy}); \theta = A_3k_z^2 + A_4(k_y^2 + k_z^2) + D_3\epsilon_{zz} + D_4(\epsilon_{xx} + \epsilon_{yy}); \epsilon$ is the strain component; $k_x, k_y,$ and $k_z$ are the vectors in the $k$-space; and $A_i$ are the components of the Hamiltonian matrix of the 4H-SiC crystal. The four deformation potential constants $D_1, D_2, D_3,$ and $D_4$ determine the complete band warpage due to the elastic deformation. The components $A_1k_x^2 + A_2(k_y^2 + k_z^2)$ and $A_3k_z^2 + A_4(k_y^2 + k_z^2)$ indicate that the constant energy surface is the ellipsoid of the revolution around the $c$-axis without strain [Fig. 4(b)].

The ratios of holes occupied in the top valence subband ($E_1$) versus the second highest subband $E_2$ ($P_1/P_2$) and the spin orbit split-off (SOSO) subband $E_3$ ($P_1/P_3$) are $e^{\Delta_1-\lambda/\hbar sT}$ and $e^{\Delta_1-\lambda/\hbar sT}$, respectively (see Sec. 4 in the supplementary material for the explanation). From the band structure of 4H-SiC,\textsuperscript{27} we derive $\Delta E_{1-2} = 0.15$ eV and $\Delta E_{1-3} = 0.8$ eV. Substituting $k_B = 1.38 \times 10^{-23}$ m$^2$ kg s$^{-2}$ K$^{-1}$ and $T = 298$ K, we have $P_1/P_2 = 365$ and $P_1/P_3 = 4.83 \times 10^{13}$. This indicates that the number of holes in the highest subband $E_3$ is dominant over the other two subbands $E_2$ and $E_1$. Therefore, the piezoresistance of 4H-SiC is solely dictated by the hole redistribution in $E_1$ under strain (see Sec. 4 in the supplementary material). The piezoresistance in 4H-SiC is promising for mechanical sensing and strain engineering since a high sensitivity can be achieved regardless of the sensor orientation.

In contrast to the anisotropy of the PZR effect in some common semiconductors (e.g., Si and 3C-SiC), the piezoresponse in 4H-SiC exhibits an isotropic property, which is more favorable for the design and fabrication of mechanical sensors, as the need for choosing the optimal orientation is no longer needed. As a result, high sensitivity can be achieved regardless of the arrangement of devices in wafers, and the number of devices per wafer can be significantly increased, reducing the cost for the sensing device.

In conclusion, the isotropic piezoresponse in the (0001) plane of p-type 4H-SiC was discovered by the calculations of the hole energy shift in the top valence bands and the coordinate transformation. The interesting phenomenon was also verified by the experimental results using a bending beam method where a uniaxial strain was applied to the piezoresistor. The longitudinal and transverse PZR coefficients were found to be $6.43 \times 10^{-11}$ Pa$^{-1}$ and $-5.12 \times 10^{-11}$ Pa$^{-1}$, respectively, which are independent of the strain orientation in the (0001) plane. The isotropic piezoresponse of p-type 4H-SiC is attributed to the isotropic hole energy shift under uniaxial stress strain in the basal plane. For calculating the parameters for each subband in bulk p-type 4H-SiC, more effort is required, and the data will be presented in our future work. The relatively high and isotropic piezoresponse of p-type 4H-SiC is promising for mechanical sensing and strain engineering since a high sensitivity can be achieved regardless of the sensor orientation.

See supplementary material for the details of the strain distribution in the bending beam configuration, the measured Young’s modulus in various orientations in the (0001) plane, the matrix transformation from the principal coordinate to an arbitrary coordinate, the calculation of the hole occupation in
the valence subbands, and the valence subband modification under strain.

This work was partially supported by Australian Research Council Grant Nos. LP150100153 and LP160101553. This work was also supported by the Queensland node of the Australian National Fabrication Facility, a company established under the National Collaborative Research Infrastructure Strategy to provide nano- and micro-fabrication facilities for Australian researchers.

Isotropic piezoresistance of p-type 4H-SiC in (0001) plane

Tuan-Khoa Nguyen,*1 Hoang-Phuong Phan,1 Toan Dinh,1 Toshiyuki Toriyama,2 Koichi Nakamura,3,4 Abu Riduan Md Foisal,1 Nam-Trung Nguyen,1 and Dzung Viet Dao1,5

1 Queensland Micro- and Nanotechnology Centre, Griffith University, 170 Kessels Road, Brisbane, Queensland 4111, Australia
2 College of Science and Engineering, Department of Mechanical Engineering, Ritsumeikan University, 1-1-1 Noji-higashi, Kusatsu, Shiga 525-8577, Japan
3 Center for the Promotion of Interdisciplinary Education and Research, Kyoto University, Kyoto 615-8540, Japan
4 Department of Materials Science and Engineering, Egypt-Japan University of Science and Technology, New Borg El-Arab, Alexandria 21934, Egypt
5 School of Engineering and Built Environment, Griffith University, Parklands Drive, Gold Coast, Queensland 4215, Australia

1 Strain distribution

Figure S1: Finite element analysis of the strain distribution on piezoresistors with different dimensions using COMSOL™.

S-1
To confirm the strain transferred to the top layer with different dimensions, a finite element analysis (FEA) was conducted as can be seen in Fig. S1. The piezoresistors have various width ranging from 10 µm to 80 µm and the strain induced these piezoresistors are almost identical and uniform. Therefore, it can be concluded that the strain induced to the piezoresistors is independent on their size, which are in the tens of µm range.

2 Young’s modulus $E$ in (0001) plane

The Young’s modulus of the 4H-SiC in various orientations was characterized using the same configuration shown in Fig. 2(a)-(b) [1]. The Young’s modulus was found almost isotropic as shown in Table 1.

Table 1: Measured Young’s modulus in various orientations in the (0001) plane of 4H-SiC wafer

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Young’s modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1100</td>
<td>503</td>
</tr>
<tr>
<td>2110</td>
<td>481</td>
</tr>
<tr>
<td>1010</td>
<td>493</td>
</tr>
<tr>
<td>1120</td>
<td>497</td>
</tr>
<tr>
<td>1100</td>
<td>503</td>
</tr>
<tr>
<td>2110</td>
<td>481</td>
</tr>
<tr>
<td>1010</td>
<td>493</td>
</tr>
<tr>
<td>1120</td>
<td>497</td>
</tr>
</tbody>
</table>

3 Coordinate transformation

The relationship between the piezoresistance tensors in the principle and arbitrary coordinates is given by

$$\pi' = \alpha \pi \alpha^{-1}$$  \hspace{1cm} (S1)

where $\alpha$ and $\alpha^{-1}$ are transformation matrix and its inverse of elasto-resistance tensor from the principle coordinate O-xyz to an arbitrary coordinate O-x’y’z’. The two matrices are composed of $l_i$, $m_j$, $n_k$ ($i,j,k=1,2,3$) which are [2]

$$
\begin{bmatrix}
  l_1 & m_1 & n_1 \\
  l_2 & m_2 & n_2 \\
  l_3 & m_3 & n_3 
\end{bmatrix} = 
\begin{bmatrix}
  \cos \psi \cos \theta \cos \phi - \sin \psi \sin \phi & \cos \psi \cos \theta \sin \phi + \sin \psi \cos \phi & -\cos \psi \sin \phi \\
  -\sin \psi \cos \theta \cos \phi - \cos \psi \sin \phi & -\sin \psi \cos \theta \sin \phi + \cos \psi \cos \phi & \sin \psi \sin \phi \\
  \sin \theta \cos \phi & \sin \theta \sin \phi & \cos \theta 
\end{bmatrix}
$$  \hspace{1cm} (S2)
Figure S2: Euler angles of the transformation from principle to arbitrary piezoresistances

For a simple case where only the rotation along the Z-axis is executed ($\phi \neq 0, \psi = 0$), then the "lmm" matrix becomes

$$
\begin{bmatrix}
  l_1 & m_1 & n_1 \\
  l_2 & m_2 & n_2 \\
  l_3 & m_3 & n_3 \\
\end{bmatrix}
= 
\begin{bmatrix}
  \cos \phi & \sin \phi & 0 \\
  -\sin \phi & \cos \phi & 0 \\
  0 & 0 & 1 \\
\end{bmatrix}
$$

(S3)

The transformation matrix $\alpha$ and its inverse $\alpha^{-1}$ would be

$$
\alpha = 
\begin{bmatrix}
  \cos^2 \phi & \sin \phi \cos \phi & 0 & 0 & 0 & 0 & 2 \sin \phi \cos \phi \\
  \sin^2 \phi & \cos^2 \phi & 0 & 0 & 0 & -2 \sin \phi \cos \phi \\
  0 & 0 & 1 & 1 & 0 & 0 & 0 \\
  0 & 0 & 0 & \cos \phi & -\sin \phi & 0 \\
  0 & 0 & 0 & \sin \phi & \cos \phi & 0 \\
  -\sin \phi \cos \phi & \sin \phi \cos \phi & 0 & 0 & 0 & \cos^2 \phi - \sin^2 \phi \\
\end{bmatrix}
$$

(S4)

$$
\alpha^{-1} = 
\begin{bmatrix}
  \cos^2 \phi & \sin \phi \cos \phi & 0 & 0 & 0 & 0 & -2 \sin \phi \cos \phi \\
  \sin^2 \phi & \cos^2 \phi & 0 & 0 & 0 & 2 \sin \phi \cos \phi \\
  0 & 0 & 1 & 1 & 0 & 0 & 0 \\
  0 & 0 & 0 & \cos \phi & \sin \phi & 0 \\
  0 & 0 & 0 & -\sin \phi & \cos \phi & 0 \\
  \sin \phi \cos \phi & -\sin \phi \cos \phi & 0 & 0 & 0 & \cos^2 \phi - \sin^2 \phi \\
\end{bmatrix}
$$

(S5)
The relationship between $\rho$ and the piezoresistance tensor $\pi$ and stress tensor $\sigma$ is given by

$$\rho = \rho_0(1 + \pi \sigma) = \rho_0(1 + \Xi) \quad \text{(S6)}$$

where $\Xi$ is relative variant component of the resistivity tensor under strain. Transforming the coordinate system to an arbitrary coordinate, one obtains

$$\Xi' = \pi' \sigma' \Rightarrow \alpha \Xi = \pi' \alpha \sigma \Rightarrow \Xi = \alpha^{-1} \pi' \alpha \sigma \quad \text{(S7)}$$

Combining Eq. S6 and Eq. S7, the piezoresistance tensors in an arbitrary coordinate is

$$\pi'_{ij} = \sum_{k,l=1}^{6} \alpha_{ik} \pi_{kl} \alpha_{lj}^{-1} \quad \text{(S8)}$$

According to Toriyama [5], the piezoresistive coefficient tensor in the principle coordinate is

$$\Pi = \begin{bmatrix} \pi_{11} & \pi_{12} & \pi_{13} & 0 & 0 & 0 \\ \pi_{12} & \pi_{11} & \pi_{13} & 0 & 0 & 0 \\ \pi_{31} & \pi_{31} & \pi_{33} & 0 & 0 & 0 \\ 0 & 0 & 0 & \pi_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & \pi_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & \pi_{11} - \pi_{12} \end{bmatrix} \quad \text{(S9)}$$

It can be seen that, the piezoresistive coefficient tensor of 4H-SiC has five independent components (i.e. $\pi_{11}$, $\pi_{12}$, $\pi_{13}$, $\pi_{31}$, and $\pi_{33}$). Now, we can do the calculation for each component $\pi'_{ij}$ of the $\pi'$ matrix including the longitudinal, transverse, and shear piezoresistive coefficients, respectively;

$$\pi_l = \pi'_{11} = \sum_{i,j=1}^{6} \alpha_{1i} \Pi_{ij} \alpha_{j1}^{-1}$$

$$= \pi_{11} (\alpha_{11}^{-1} + \alpha_{12}^{-1} \alpha_{21}^{-1} + \alpha_{16}^{-1} \alpha_{61}^{-1}) + \pi_{12} (\alpha_{11}^{-1} \alpha_{21}^{-1} + \alpha_{12}^{-1} \alpha_{11}^{-1} - \alpha_{16}^{-1} \alpha_{61}^{-1}) + \pi_{13} (\alpha_{11}^{-1} \alpha_{31}^{-1} + \alpha_{12}^{-1} \alpha_{31}^{-1} + \alpha_{13}^{-1} \alpha_{13}^{-1} + \alpha_{13}^{-1} \alpha_{21}^{-1} + \pi_{33} \alpha_{33}^{-1} + \pi_{44} (\alpha_{14}^{-1} \alpha_{41}^{-1} + \alpha_{15}^{-1} \alpha_{31}^{-1})) + \pi_{11} (i_1^4 + m_1^4 + 2l_1^2 m_1^2) + \pi_{12} (l_1^2 m_1^2 + l_1^2 m_1^2 - 2l_1^2 m_1^2) + 2\pi_{13} (l_1^2 n_1^2 + m_1^2 n_1^2) + \pi_{33} n_1^4 + 2\pi_{44} (m_1^2 n_1^2 + n_1^2 l_1^2) + \pi_{11} (\sin^2 \phi + \cos^2 \phi)^2 \quad \text{(S10)}$$

$$= \pi_{11}$$
\[ \pi_t = \pi_{12} = \sum_{i,j=1}^{6} \alpha_{ij} \Pi_{ij} \alpha_{ij}^{-1} \]
\[ = \pi_{11}(\alpha_{11}^{-1} + \alpha_{12}^{-1} + \alpha_{16}^{-1}) + \pi_{12}(\alpha_{11}^{-1} + \alpha_{12}^{-1} - \alpha_{16}^{-1}) \]
\[ + \pi_{13}(\alpha_{11}^{-1} + \alpha_{12}^{-1} + \alpha_{13}^{-1} + \alpha_{13}^{-1}) + \pi_{13}(\alpha_{11}^{-1} + \alpha_{12}^{-1} + \alpha_{13}^{-1}) \]
\[ = \pi_{11}(l_1^2 l_2^2 + m_1^2 m_2^2 + 2l_1 m_1 l_2 m_2) + \pi_{12}(l_1^2 m_2^2 + m_1^2 l_2^2 - 2l_1 m_1 l_2 m_2) + \pi_{13}(l_1^2 n_2^2 + m_1^2 n_2^2) \]
\[ + n_1^2 n_2^2 + 2n_1 n_2 + 2n_1 m_1 n_2 + n_1 l_1 n_2 l_2 \]
\[ = \pi_{11}(-\cos \theta \sin \phi + \cos \phi \sin \phi)^2 + \pi_{12}(\sin^2 \phi + \cos^2 \phi)^2 \]
\[ = \pi_{12} \]

\[ \pi_s = \pi_{16} = \sum_{i,j=1}^{6} \alpha_{ij} \Pi_{ij} \alpha_{ij}^{-1} \]
\[ = \pi_{11}(\alpha_{11}^{-1} + \alpha_{12}^{-1} + \alpha_{16}^{-1}) + \pi_{12}(\alpha_{11}^{-1} + \alpha_{12}^{-1} - \alpha_{16}^{-1}) \]
\[ + \pi_{13}(\alpha_{11}^{-1} + \alpha_{12}^{-1} + \alpha_{13}^{-1} + \alpha_{13}^{-1}) + \pi_{13}(\alpha_{11}^{-1} + \alpha_{12}^{-1} + \alpha_{13}^{-1}) \]
\[ + \pi_{44}(\alpha_{14}^{-1} + \alpha_{15}^{-1}) \]
\[ = \pi_{11}(l_1^2 l_2^2 + m_1^2 m_2^2 + 2l_1 m_1 (m_1 l_2 + m_1 l_2) + \pi_{12}(l_1^2 m_1 m_2 + m_1^2 l_1 l_2) \]
\[ - 2l_1 m_1 (m_1 l_2 + m_1 l_2) + \pi_{13}(l_1^2 n_1 n_2 + m_1^2 n_1 n_2 + n_1^2 l_1 l_2 + n_1^2 2m_1 m_2) \]
\[ + \pi_{33}(l_1^2 n_1 n_2 + \pi_{44}(2m_1 n_1 (m_1 n_2 + m_2 n_2) + 2n_1 l_1 (m_1 l_2 + m_2 l_2) \]
\[ = 2\pi_{11}(l_1^2 l_2 + m_1^2 m_2 + l_1 m_1 (m_1 l_2 + m_2 l_2) + \pi_{12}(l_1^2 m_1 m_2 + m_1^2 l_1 l_2 - l_1 m_1 (m_1 l_2 + m_2 l_2) \]
\[ + 2\pi_{13}(l_1^2 n_1 n_2 + m_1^2 n_1 n_2 + n_1^2 l_1 l_2 + n_1^2 2m_1 m_2) + 2\pi_{33} n_1^2 n_1 n_2 + 2\pi_{44}(m_1 n_1 (m_1 n_2 + m_2 n_2) \]
\[ + n_1 l_1 (m_1 l_2 + m_2 l_2) \]
\[ = 2\pi_{11}(-\cos^3 \phi \sin \phi + \cos \phi \sin^3 \phi - \sin^3 \phi \cos \phi + \cos^3 \phi \sin \phi) + 2\pi_{12} \times 0 \]
\[ = 0 \]

4 Hole occupation in valence subbands

According to Eq. 3 (in the main article), we have

\[ E_{1}^s = F \]
\[ E_{2,3}^s = \frac{G + \lambda}{2} \pm \sqrt{\left( \frac{G - \lambda}{2} \right)^2 + \Delta^2} \]
\[ = \frac{\Delta_1 - \Delta_2 + 2\lambda + \theta}{2} \pm \sqrt{\left( \frac{\Delta_1 - \Delta_2 + \theta}{2} \right)^2 + \Delta^2} \]

(S10)
In the case $2\Delta \ll \Delta_1 - \Delta_2 + \theta$

\[
E_2^s = \frac{\Delta_1 - \Delta_2 + 2\lambda + \theta}{2} + \frac{\Delta_1 - \Delta_2 + \theta}{2} \sqrt{\left(\frac{\Delta_1 - \Delta_2 + \theta}{2}\right)^2 + \Delta^2}
\]

\[
\approx \Delta_1 - \Delta_2 + \lambda + \theta + \frac{\Delta^2}{\Delta_1 - \Delta_2 + \theta}
\]

\[
E_1^s - E_2^s = 2\Delta_2 - \frac{\Delta^2}{\Delta_1 - \Delta_2 + \theta}
\]

If $2\Delta \gg \Delta_1 - \Delta_2 + \theta$

\[
E_2^s = \frac{\Delta_1 - \Delta_2 + 2\lambda + \theta}{2} + \Delta \sqrt{\left(\frac{\Delta_1 - \Delta_2 + \theta}{2\Delta}\right)^2 + 1}
\]

\[
\approx \frac{\Delta_1 - \Delta_2 + 2\lambda + \theta}{2} + \Delta + \frac{(\Delta_1 - \Delta_2 + \theta)^2}{8\Delta}
\]

\[
E_1^s - E_2^s = 2\Delta_2 - \frac{\Delta^2}{\Delta_1 - \Delta_2 + \theta}
\]

Hole occupation for the highest valence subband

\[
P_1 = 2 \int \left[ e^{-\frac{E_1^s - E_F}{k_BT}} + 1 \right]^{-1} d\kappa
\]

\[
\approx 2 \int e^{-\frac{\Delta_1 + \Delta_2 + \lambda + \theta - E_F}{k_BT}} d\kappa
\]

\[
= 2 \int e^{-\frac{\Delta_1 + \Delta_2 + D_\lambda(\epsilon) + D_\theta(\epsilon) - E_F}{k_BT}} d\kappa
\]

\[
= 2e^{-\frac{\Delta_1 + \Delta_2 + D_\lambda(\epsilon) + D_\theta(\epsilon) - E_F}{k_BT}} \times \int e^{-\frac{A_2 + A_4}{k_BT} k_x^2} d\kappa \int e^{-\frac{A_2 + A_4}{k_BT} k_y^2} d\kappa \int e^{-\frac{A_1 + A_3}{k_BT} k_z^2} d\kappa
\]

where

\[
D_\lambda(\epsilon) = D_1 \epsilon_{zx} + D_2 (\epsilon_{xx} + \epsilon_{yy})
\]

\[
D_\theta(\epsilon) = D_3 \epsilon_{zz} + D_4 (\epsilon_{xx} + \epsilon_{yy})
\]

with either $A_1 + A_3$ and $A_2 + A_4$ are negative.

Subsequently, we derive

\[
P_1 = 2e^{-\frac{\Delta_1 + \Delta_2 + D_\lambda(\epsilon) + D_\theta(\epsilon) - E_F}{k_BT}} \frac{(\pi k_BT)^{-3/2}}{\sqrt{-(A_1 + A_3)(A_2 + A_4)^2}}
\]

Using first order approximation for the second highest valence subband, we have

\[
P_2 = 2e^{-\frac{\Delta_1 + \Delta_2 + D_\lambda(\epsilon) + D_\theta(\epsilon) + \Delta^2 / (\Delta_1 - \Delta_2 + \theta) - E_F}{k_BT}} \frac{(\pi k_BT)^{3/2}}{\sqrt{-(A_1 + A_3)(A_2 + A_4)^2}}
\]
From Eq. S15 and Eq. S16, we have
\[
P_1 \frac{P_2}{P_1} = e^{\frac{2\Delta_2 - \Delta^2}{k_B T}} e^{\frac{E_1^s - E_2^s}{k_B T}}
\]
(S17)

Conducting the same procedure, we also have
\[
P_1 \frac{P_3}{P_1} = e^{\frac{E_1^s - E_3^s}{k_B T}}
\]
(S18)

Deriving from [3], \(\Delta E_{1-2} = 0.15 \text{eV}\) and \(\Delta E_{1-3} = 0.8 \text{eV}\), substitute \(k_B = 1.38 \times 10^{-23}\) and \(T = 298 \text{K}\), we have
\[
P_1 \frac{P_2}{P_1} = e^{0.15} \times \frac{1.62 \times 10^{-19}}{298} \approx 365
\]
(S19)

These results indicate that the number of hole occupied in the \(E_1\) subband is much greater than the other two subbands. Therefore, it can be concluded that the contribution of the hole transfer in \(E_2\) and \(E_3\) subbands to the piezoresistive effect is negligible.

5 Strain induced effect in valence subbands

Taking the second derivative of \(E_1^2\) and \(E_2^2\) [4] at strain-free state, we have

In \(\Gamma A\) direction
\[
\frac{\partial^2 E_1^0}{\partial k_2^2} = 2(A_1 + A_3)
\]
\[
\frac{\partial^2 E_2^0}{\partial k_2^2} = 2(A_1 + A_3 \frac{E_2^0}{E_2^0 - E_3^0})
\]
(S20)

In \(\Gamma M\) and \(\Gamma K\) directions
\[
\frac{\partial^2 E_1^0}{\partial k_2^2} = \frac{\partial^2 E_1^0}{\partial k_6^2} = 2(A_2 + A_4)
\]
\[
\frac{\partial^2 E_2^0}{\partial k_2^2} = \frac{\partial^2 E_2^0}{\partial k_6^2} = 2(A_2 + A_4 \frac{E_2^0}{E_2^0 - E_3^0})
\]
(S21)

It should be noted that \(A_i\) \((i=1,2,3,4)\) and \(E_3^0 < 0\), we have \(\frac{\partial^2 E_1^0}{\partial k_6^2} > \frac{\partial^2 E_1^0}{\partial k_2^2}\) with \(j=(x,y,z)\), or in other words, the holes occupied in \(E_1\) subband has small effective mass and high mobility while the holes in \(E_2\) subband has large effective mass and low mobility.

At strained state, we also have
In $\Gamma A$ direction
\[
\frac{\partial^2 E_1^i}{\partial k_z^2} = 2(A_1 + A_3) \equiv \frac{\partial^2 E_1^0}{\partial k_z^2}
\]
\[
\frac{\partial^2 E_2^i}{\partial k_z^2} = 2A_1 + A_3 \frac{\Delta_1 - \Delta_2 + D_0(\epsilon)}{2 \sqrt{\left(\frac{\Delta_1 - \Delta_2 + D_0(\epsilon)}{2}\right)^2 + \Delta^2}} \tag{S22}
\]

In $\Gamma M$ and $\Gamma K$ directions
\[
\frac{\partial^2 E_1^i}{\partial k_x^2} = \frac{\partial^2 E_1^0}{\partial k_x^2} = 2(A_2 + A_4) \equiv \frac{\partial^2 E_1^0}{\partial k_x^2}
\]
\[
\frac{\partial^2 E_2^i}{\partial k_x^2} = 2A_2 + A_4 \frac{\Delta_1 - \Delta_2 + D_0(\epsilon)}{2 \sqrt{\left(\frac{\Delta_1 - \Delta_2 + D_0(\epsilon)}{2}\right)^2 + \Delta^2}} \tag{S23}
\]

Eq. S22 and Eq. S23 indicate that the variation of subband $E_1$ is independent on the strain direction!

In the case where $2\Delta \ll \Delta_1 - \Delta_2 + D_0(\epsilon)$ with $\theta = A_3k_z^2 + A_4(k_x^2 + k_y^2) + D_3\epsilon_{zz} + D_0(\epsilon)$, we have
\[
\frac{\partial^2 E_2^i}{\partial k_z^2} \approx \frac{\partial^2 E_2^0}{\partial k_z^2}; \quad \frac{\partial^2 E_2^i}{\partial k_x^2} \approx \frac{\partial^2 E_2^0}{\partial k_x^2}; \quad \frac{\partial^2 E_2^i}{\partial k_y^2} \approx \frac{\partial^2 E_2^0}{\partial k_y^2} \tag{S24}
\]

Therefore, the subband $E_2$ is also independent on the strain orientation.

References


Summary

In this chapter, the piezoresistive effect and the isotropic piezoresistance in the (0001) plane in p-type 4H-SiC were investigated with high gauge factors of 32 and -27 for longitudinal and transverse piezoresistor configurations, respectively. The isotropy of the piezoresistance in the basal plane of p-type 4H-SiC was analytically presented and confirmed by experiments. This interesting phenomenon in p-type 4H-SiC is promising for the design and fabrication of mechanical sensors and strain-engineered electronics since high sensitivity and consistent performance can be achieved regardless of the crystallographic orientation.
Chapter 5

Piezoresistive effect in p-type 4H-SiC at high temperature

This chapter presents the temperature dependence of the piezoresistive effect in p-type 4H-SiC. The piezoresistive effect exhibits good linearity and high stability at high temperatures up to 600°C. This is attributed to the robust p-n junction in 4H-SiC which prevents the current from leaking to substrate. This finding demonstrates the capability of p-type 4H-SiC for mechanical sensing in hostile environments.

Statement of contribution to co-authored published papers

This chapter is in a form of a peer-reviewed published paper and the bibliographic details of the paper, including all authors, are:


My contribution to the submitted papers involved:

• Design and fabrication of the samples
• Experimental task
• Data acquisition and analysis
Chapter 5 Piezoresistive effect in p-type 4H-SiC at high temperature

- Writing manuscripts

Appropriate acknowledgements have been stated and included in the papers.

**Reuse permission:** The papers are reused from

*Journal of Materials Chemistry C, 2018, DOI: 10.1039/C8TC03094D.*

Copyright permission from the publisher to reuse the paper in this thesis is attached in the following page.

(Sign) ___________________________  (Date) 30/07/2018

Tuan-Khoa Nguyen (First and corresponding author)

(Sign) ___________________________  (Date) 30/07/2018

Assoc. Prof. Dzung Viet Dao (Principle supervisor)
This article can be cited before page numbers have been issued, to do this please use: T. Nguyen, H. Phan, T. Dinh, A. R. Md Foisal, N. Nguyen and D. Dao, J. Mater. Chem. C, 2018, DOI: 10.1039/C8TC03094D.

This is an Accepted Manuscript, which has been through the Royal Society of Chemistry peer review process and has been accepted for publication.

Accepted Manuscripts are published online shortly after acceptance, before technical editing, formatting and proof reading. Using this free service, authors can make their results available to the community, in citable form, before we publish the edited article. We will replace this Accepted Manuscript with the edited and formatted Advance Article as soon as it is available.

You can find more information about Accepted Manuscripts in the author guidelines.

Please note that technical editing may introduce minor changes to the text and/or graphics, which may alter content. The journal’s standard Terms & Conditions and the ethical guidelines, outlined in our author and reviewer resource centre, still apply. In no event shall the Royal Society of Chemistry be held responsible for any errors or omissions in this Accepted Manuscript or any consequences arising from the use of any information it contains.
High-temperature tolerance of the piezoresistive effect in p-4H-SiC for harsh environment sensing†

Tuan-Khoa Nguyen, Hoang-Phuong Phan, Toan Dinh, Abu Riduan Md Foisal, Nam-Trung Nguyen and Dzung Viet Dao

4H-silicon carbide based sensors are promising candidates for replacing prevalent silicon-based counterparts in harsh environments owing to their superior chemical inertness, high stability and reliability. However, the wafer cost and the difficulty in obtaining an ohmic contact in the metallization process hinders the use of this SiC polytype for practical sensing applications. This article presents the high-temperature tolerance of a p-type 4H-SiC piezoresistor at elevated temperatures up to 600 °C. A good ohmic contact was formed by the metallization process using titanium and aluminium annealed at 1000 °C. The leakage current at high temperatures was measured to be negligible thanks to a robust p–n junction. Owing to the superior physical properties of the bulk 4H-SiC material, a high gauge factor of 23 was obtained at 600 °C. The piezoresistive effect also exhibits good linearity and high stability at high temperatures. The results demonstrate the capability of p-type 4H-SiC for the development of highly sensitive sensors for hostile environments.

The development of hostile environment sensors is hindered by current technologies relying on silicon (Si) materials. For instance, Si experiences plastic deformation at 500 °C and instability at high temperatures or in corrosive environments. In contrast, its superior tolerance to high temperatures, corrosive media, and intensive shock positions silicon carbide (SiC) as a promising material for a broad range of applications in hostile environments. Moreover, the progress of wafer growth and the fabrication process not only cuts down the cost of SiC wafers, but also greatly improves the film quality and reliability of SiC devices. In terms of micromachining, SiC can inherit the strong foundation and maturity of Si technology infrastructures, which enables the batch production of SiC sensing devices for corrosive or harsh environment sensing. The piezoresistive effect has been considered as the main transduction mechanism for mechanical sensors such as strain/pressure/acceleration sensors, thanks to the simplicity of the read-out circuitries and the wide linearity range. When mechanical stress/strain is applied to a piezoresistive based sensing element, the energy band is significantly modified, leading to a large variation in electrical conductivity (or resistivity).

Due to the different arrangement of Si and C atoms in the crystal structure, there are more than 200 SiC polytypes which have been discovered. 3C-SiC, 4H-SiC and 6H-SiC are the most common and commercially available polytypes to date. Over the last four decades, many studies have investigated the piezoresistive effect of these polytypes for mechanical sensing applications. Shor et al. revealed a gauge factor (GF) of ∼31.8 for n-type single crystalline 3C-SiC grown on a Si substrate with a doping concentration of 10¹⁷ cm⁻³. Phan et al. reported the GFs of p-type 3C-SiC to be 25–28 at temperatures varying from 300 K to 573 K using an in situ approach. For n-SiC, the GFs of highly doped (i.e. 2 × 10¹⁹ cm⁻³) n-type and p-type 6H-SiC at room temperature were found to be ∼22 and 27, respectively; these GFs decreased by half at 250 °C. Akiyama et al. measured the GF of n-type highly doped 4H-SiC to be 20.8 at room temperature for transverse piezoresistors. Amongst the three most common SiC polytypes, 4H-SiC possesses the highest energy band gap (i.e. 3.26 eV) with a high potential barrier which can effectively minimize the generation of electron–hole pairs with an increase in temperature. This results in the good stability and reliability of 4H-SiC electronic devices in high-temperature operations. Additionally, the superior physical properties combined with a high break-down voltage makes 4H-SiC the most important SiC polytype for high-power electronics, including Schottky diodes, high power-switching devices, bipolar junction transistors (BJTs) and metal oxide semiconductor field effect transistors (MOSFETS). In terms of sensing devices, to date there are limited studies on 4H-SiC sensors for high temperature applications due to several challenges. Firstly, the development of high quality SiC film growth recently enabled the availability of p-4H-SiC wafers, whereas engineering-grade n-4H-SiC wafers have been available for a decade. Another factor is the difficulty in the formation of an
ohmic contact with p-4H-SiC,15,16 which limits the studies on the piezoresistive effect of p-4H-SiC. Additionally, the deployment of strain configurations which can effectively induce strain to thin-film piezoresistors at high temperatures is challenging. These obstacles result in a lack of understanding of the piezoresistive effect of 4H-SiC at elevated temperatures, which is solely based on theoretical analyses, such as a first principles simulation in SiC nanosheets17 and a deformation potential approximation.18

In this article, we present the first experimental study on the piezoresistive effect of p-type 4H-SiC at high temperatures up to 600 °C. As such, high gauge factors were obtained: 33 at room temperature and 23 at 600 °C. The high sensitivity and good stability at elevated temperatures are attributed to the superior physical properties of 4H-SiC as well as the robust p–n junction which prevents the current from leaking to the substrate. This demonstrates the capability of p-type 4H-SiC for hostile environment sensing where the use of Si or polymer based devices is infeasible.

The p-type 4H-SiC piezoresistive element was fabricated from a bulk 4H-SiC wafer which is commercially available from Cree. The wafer consists of epitaxial highly doped (i.e. carrier density of 10^18 cm^-3) p-type 4H-SiC on the top, n-type 4H-SiC lying in the middle and a low doped n-type substrate. The thickness of both the p-type and n-type layers is 1 μm and the thickness of the substrate is 350 μm. The wafer was cleaned prior to a photolithography process using AZ9245 to form a patterned photoresist mask with a thickness of 4 μm for the subsequent etching of SiC. The patterned mask SiC wafer was anisotropically etched in an STS SR inductive couple plasma (ICP) system for 10 min with 1500 W coil power. A 50 sccm HCl flow at a pressure of 2 mTorr was supplied. After ICP etching, the remaining photoresist mask was removed by acetone and isopropanol, and then thoroughly rinsed in deionized water. An etch depth of 1.3 μm was measured by a Dektak 150 profiler, which ensured that the unmasked p-type regions were completely removed, as shown in Fig. 1(a). To form the metal contact, 100 nm-thick Ti and Al layers were subsequently sputtered in an SNS Sputterer. Another photolithography step was performed to create a patterned mask prior to the wet etching of Al and Ti by an Al etchant (i.e. H_3PO_4) and a mixture of hydrogen fluoride (HF) and hydroperoxide (H_2O_2), respectively. Subsequently, the wafer was thoroughly rinsed and cleaned with deionized water. After the metallisation, the wafer was annealed by a rapid thermal annealing (RTA) process at 1000 °C for 3 min to create the ohmic contact. The ohmic contact with p-type 4H-SiC has been extensively investigated in previous studies,15,16,19 and the ohmic contact formation of Ti/Al to p-type 4H-SiC annealed at 1000 °C occurs in two stages.16 In the early stage, Ti and Al react to form TiAl, TiAl_3 and Ti_3Al, leaving unreacted C atoms at the Ti/SiC interfaces. Subsequently, in the later stage, Ti, SiC and C react to form a ternary Ti_2SiC_3 compound. The use of thin Al and Ti layers ensures that they react with C and form the compound during the two stages. Subsequently, the reaction of SiC and the contact materials increases the doping concentration in the region close to the SiC/metal interface and forms the ohmic contact.

The contact resistance was measured to be approximately 10^-3 Ω cm^2 at room temperature and then increased about 30% at 600 °C. In the final step, the wafer was diced into 30 x 3 mm^2 beams prior to the piezoresistive effect measurement.

The 4H-SiC beams were placed in a temperature-controlled probe system (i.e. Linkam HFS600E-PB2). The temperature of the probe chamber was precisely regulated by a built-in closed-loop control in the temperature controller unit. The upper limit of temperature in this work is restricted by the equipment. The small temperature tolerance (i.e. ± 0.1 °C) minimizes the deviation of the resistance change by the temperature variation. The sensitivity of the 4H-SiC piezoresistor was defined by the fractional change of the resistance versus the applied strain:

\[ GF = (1 + 2\nu) + \frac{\Delta R/R}{\varepsilon} \]  

(1)

where \( \varepsilon \) is the strain induced to the piezoresistor and \( \nu \) is the Poisson ratio of 4H-SiC. For SiC materials, the first term of eqn (1) represents the change due to the geometrical effect under strain which is inferior to the second term of the piezoresistive effect. To characterise the piezoresistive effect of 4H-SiC, uniaxial strain was induced to the piezoresistor using a bending method.9,28,29 The analytical model is a double-layer cantilever with one clamped end, and a static force was applied to the other end. Subsequently, uniaxial strain was effectively transferred to the piezoresistor lying on the top surface of the
cantilever (see ESI† for the analysis of the straining model). The bending configuration is depicted in the Fig. 4 inset in which one end of the 4H-SiC beam was fixed on the hot plate by probes while the other end was deflected by a static force.

For Si based devices the p–n junction cannot withstand temperatures above 200 °C due to the generation of thermally activated electron–hole pairs, leading to instability and unreliability in their operation. In contrast, the large energy band gap in 4H-SiC forms a high potential barrier, enabling stability at high temperatures. We confirmed this property in 4H-SiC by characterizing the rectification behaviour of the 4H-SiC p–n junction at elevated temperatures up to 600 °C using a Keithley 2602. Fig. 2 shows the current–voltage characteristics of the p-type piezoresistor and the current leak through the p–n junction at a temperature range from 23 °C to 600 °C. The measured leakage current was approximately three orders of magnitude smaller than the current flowing in the p-type sensing layer. For example, at an applied voltage of 5 V at 600 °C, the current in the p-type was approximately 460 μA while the leakage current was measured to be below 1 nA. This is attributed to the robust back-to-back p/n diode preventing the current from leaking into the substrate.5,30 The very small current leaking into the substrate demonstrates a significant advantage of 4H-SiC over other low band gap materials for mechanical sensing at elevated temperatures.

The relative resistance change versus the applied strain ranging from 0 to 215 ppm at temperatures ranging from 23 °C to 600 °C is illustrated in Fig. 3. Evidently, the p-type 4H-SiC piezoresistors exhibited good linearity in the chosen strain range. The measured GFs at varying temperatures are shown in Fig. 4. The GF at room temperature was approximately 33, then it gradually decreased to about 23 at 600 °C. This is equivalent to a reduction of sensitivity of approximately 30% in the given temperature range. This result agrees well with the decreasing trend of the GF with an increase in the ambient temperature in other reports for SiC polytypes (Table 1). It should be noted that p-type 4H-SiC is able to retain a relatively high GF at 600 °C, which is ten times higher than the GF of conventional metal strain gauges at room temperature. When the temperature increases, the ionization effect raises the carrier concentration as well as the electrical conductance of p-type 4H-SiC (Fig. 2). As a consequence, the increase in carrier concentration and the carrier scattering will reduce the GF in p-type 4H-SiC. This hypothesis was confirmed by the experimental results, as shown in Fig. 3 and 4. However, the GF did not significantly decrease under the wide range of temperatures.

![Fig. 2](image-url) Current–voltage characteristics of the p-type piezoresistor with a voltage ranging from −5 V to 5 V at various temperatures from 23 °C to 600 °C, showing the linear ohmic characteristics of the Ti/Al contact to the p-type layer. Inset: Schematic sketch of the measurement of the current flowing in the p-type layer and through the p–n junction.

![Fig. 3](image-url) Fractional change of resistance of the p-type 4H-SiC piezoresistor with various applied strains of up to 215 ppm, showing the good linearity of the piezoresistive effect at high temperatures.

![Fig. 4](image-url) Measured gauge factor of p-type 4H-SiC at a temperature ranging from 23 °C to 600 °C; the GF at 600 °C decreases about 30% in comparison with the GF at room temperature. Inset: Schematic sketch of the bending beam experiment in a Linkam chamber (not to scale).
temperature. This result is attributed to the high carrier concentration of the p+ layer used in this work, which stabilized the Fermi–Dirac integral at high temperatures and resulted in a relatively stable GF.

The GFs of different SiC polytypes including 3C-, 6H-, and 4H-SiC at high temperatures are summarised in Table 1. There are a number of factors for the variation in the GF amongst different studies, including the difference in the SiC polytypes, the impurity concentrations, and the quality/growth processes of the SiC films. The dopant type (n or p) typically dictates the sign of the GF (i.e. ‘−’ sign for longitudinal n-type or transverse p-type piezoresistors and ‘+’ sign for transverse n-type or longitudinal p-type piezoresistors). Moreover, the GF normally decreases with an increase in carrier concentration and with an increase in temperature. The positive (or negative) values of the GF indicate that the conductivity of the SiC piezoresistors decreases (or increases) with an increase in stress/strain. In terms of sensing devices, only the magnitude of the GF is significant and not the sign. However, in the case of mobility enhancement, it is possible to significantly increase the electron mobility in p-type/n-type SiC by utilizing transverse/longitudinal strains (with a negative GF). Akiyama et al. reported GFs for n-type 4H-SiC, with a highest value of 20.8 for transverse resistors and −10 for longitudinal resistors at room temperature. In comparison, our measured GFs for p-type 4H-SiC are significantly higher (i.e. 33 at room temperature and 23 at 600 °C). This means that the use of p-type 4H-SiC will yield a higher sensitivity compared to the n-type counterpart. Additionally, the as-fabricated p-type 4H-SiC piezoresistors are able to retain a relatively high GF at elevated temperatures where the use of other common semiconductors (e.g. silicon and polymers) is not possible.

The variation in the electrical conductivity of p-type 4H-SiC is due to the increased ionization effect by temperature. Under strain, there is a hole transfer between the top valence bands including the light hole (LH), heavy hole (HH), and spin-orbit split-off (SOSO) bands. In the energy band of 4H-SiC, the band gaps between the light hole band versus the heavy hole and SOSO bands are 0.15 eV and 0.65 eV, respectively. Additionally, the hole concentration ratio between the valence bands is e^{E_n/k_BT}. By substituting the Boltzmann constant k_B = 1.38 × 10^{-23} J K^{-1} and the temperature range T = 298–873 K, we derive \( p_H/p_{p+} = 7.3 \times 10^3 - 300 \) and \( p_L/p_{pSO} = 5.6 \times 10^3 \), where \( p_L \), \( p_H \), and \( p_{pSO} \) are the hole concentrations of the LH, HH, and SOSO bands, respectively. This means that the number of holes occupying the LH band is much larger than those in the HH and SOSO bands and the majority of the holes are located in the LH band, leading to the dominant effect of the LH band over the HH and SOSO bands in the piezoresitivity of p-type 4H-SiC. Therefore, the change in the electrical conductivity of p-type 4H-SiC can be considered solely dependent on the redistribution of the holes in the LH band under strain and ambient temperature \( T \) as

\[
\frac{\Delta \sigma}{\sigma} \approx \frac{\Delta p_1}{p_1} = \frac{1}{k_B T} \frac{\Delta E_Y}{1 + (m_1^*/m_2^*)^{1/2}}
\]

where \( m_1^* \) and \( m_2^* \) are the effective masses of the hole in the LH and HH bands, respectively; \( E_Y \) is the band splitting energy as a function of the induced stress to the crystal lattice. From eqn (2), it can be realized that the relative resistance change (or conductivity variation) is almost inversely proportional to the increase in ambient temperature, which is in good agreement with the experimental data (Fig. 4). In conclusion, the high gauge factors of p-type 4H-SiC were obtained with good linearity and stability at high temperatures up to 600 °C. At 600 °C the gauge factor was found to be approximately 23 which is at least ten times higher than the conventional metal strain gauges. The measured piezoresitivity of p-type 4H-SiC with temperature is in good agreement with the hole transfer model in p-type SiC. The high gauge factor of p-type 4H-SiC demonstrates its potential for mechanical sensing in hostile environments where the use of Si or polymer counterparts is infeasible.

**Conflicts of interest**

There are no conflicts to declare.

**Acknowledgements**

This work has been partially supported by Australian Research Council grants LP150100153 and LP160101553. This work was also supported by the Queensland node of the Australian National Fabrication Facility, a company established under the
National Collaborative Research Infrastructure Strategy to provide nano and micro-fabrication facilities for Australian researchers.

Notes and references

Electronic Supplementary Information

High-temperature tolerance of piezoresistive effect in p-4H-SiC for harsh environment sensing

Tuan-Khoa Nguyen,∗1 Hoang-Phuong Phan,1 Toan Dinh,1 Abu Riduan Md Foisal,1 Nam-Trung Nguyen,1 and Dzung Viet Dao1,2

1 Queensland Micro- and Nanotechnology Centre, Griffith University, Brisbane, Queensland 4111, Australia
2 School of Engineering and Built Environment, Griffith University, Gold Coast, Queensland 4215, Australia

Straining analysis

To induce a uniaxial strain to the piezoresistor, a bending method method was utilized which is a doubled-layer cantilever as shown in Figure S1 (a). The analytical model is equivalent to a cantilever with one clamped end, while a static force was applied to the other end. Considering

Figure S1: a) Analytical model of the bending beam method consisting of two layers where \( t_2 \ll t_1 \). b) Finite element analysis (FEA) result of the bending beam configuration indicates a uniformly distributed strain in the p-type piezoresistor lying on the top surface of the 4H-SiC beam.

S-1
the mesa structures in the p-type layer lying on the top of a substrate, we can derive the strain function induced on the piezoresistor as [1]

$$\varepsilon(x) = \frac{F}{bD}(l_1 + l_2 - x)t_n$$  \ (S1)

where $F$ is the applied force at the free end, $b$ is the width of the SiC beam, $t_n$ is the length from the neutral plane of the bent beam to the p-type 4H-SiC layer, $l_1$ and $l_2$ are the distance from the piezoresistor to the free end and the length of the piezoresistor, respectively; $D$ is the bending modulus which is defined by

$$D = \frac{E(t_1^4 + t_2^4 + 2t_1t_2(2t_1^2 + 2t_2^2 + 3t_1t_2))/(12(t_1 + t_2))}{12(t_1 + t_2)}$$

where $t_1$ and $t_2$ are the thicknesses of the substrate and the p-type piezoresistor, respectively; the two layers are made of the same material with the Young’s modulus $E$.

The thin sensing layer lies on top of the thick substrate with a ratio of below 0.003. The boundary condition includes one fixed end and the other deflected end. The finite element analysis, shown in Fig. 1(b), indicates the uniformity of strain transferred from the substrate to the functioning layer lying on the top surface.

References

Summary

In this chapter, the temperature dependence of the piezoresistive effect in p-type 4H-SiC was presented. A good linearity and high stability at high temperatures up to 600°C were experimentally demonstrated. This finding demonstrates the capability of p-type 4H-SiC for hostile environment sensing where the use of Si or polymer based devices is infeasible.
Chapter 6

4H-SiC based device development for mechanical sensing

This chapter presents a p-type 4H-SiC van der Pauw strain sensor and a 4H-SiC piezoresistive pressure sensor utilising a laser scribing approach. The van der Pauw sensor exhibits excellent repeatability and linearity with a significantly large output voltage while the pressure sensor is able to operate at a temperature range from cryogenic to elevated temperatures with an excellent linearity and repeatability. The high sensitivity and good reliability at either cryogenic and elevated temperatures are attributed to the profound piezoresistive effect in p-type 4H-SiC and the robust p-n junction which prevents the current from leaking to the substrate. Combining these performances with the excellent mechanical strength, electrical conductivity, thermal stability, and chemical inertness of 4H-SiC, the proposed sensors are promising for strain monitoring in harsh environments. The experimental measurements of 4H-SiC based pressure sensor were performed employing the cryogenic and high temperature chambers provided by the Department of Aeronautics & Astronautics at Stanford University.

Statement of contribution to co-authored published papers
This chapter is in the form of two co-authored published papers. The bibliographic details of the published papers, including all authors, are:

(J.23) **Tuan-Khoa Nguyen**, Hoang-Phuong Phan, Ji-Sheng Han, Toan Dinh, Abu Riduan Md Foisal, Sina Dimitrijev, Yong Zhu, Nam-Trung Nguyen, and Dzung Viet Dao, “Highly sensitive p-type 4H-SiC Van der Pauw strain sensor,” *RSC Advances* vol. 8(6), pp. 3009-3013 (2018).

**My contribution to the published papers involved:**

- Design and fabrication of the samples
- Experimental task
- Data acquisition and analysis
- Writing manuscripts

(Sign) ____________________________  (Date) **30/07/2018**

**Tuan-Khoa Nguyen** (First and corresponding author)

(Sign) ____________________________  (Date) **30/07/2018**

**Assoc. Prof. Dzung Viet Dao** (Principle supervisor)

(Sign, ) ____________________________  (Date) **30/07/2018**

**Dr. Yong Zhu** (Principle supervisor)

&


My contribution to the published papers involved:
Chapter 6 4H-SiC based device development for mechanical sensing

- Design and fabrication of the samples
- Experimental task
- Data acquisition and analysis
- Writing manuscripts

(Sign) ____________________________ (Date) 30/07/2018

Tuan-Khoa Nguyen (First and corresponding author)

(Sign) ____________________________ (Date) 30/07/2018

Assoc. Prof. Dzung Viet Dao (Principle supervisor)

Appropriate acknowledgements have been stated and included in the papers.

Reuse permission: The papers are reused from

RSC Advances vol. 8(6), pp. 3009-3013 (2018) (DOI: 10.1039/C7RA11922D). This Open Access Article is licensed under a Creative Commons Attribution-Non Commercial 3.0 Unported Licence.

&


Copyright permission from the publishers to reuse theses papers in this thesis are attached in the following pages.
6.1 P-type 4H-SiC van der Pauw strain sensor
Highly sensitive p-type 4H-SiC van der Pauw sensor

Tuan-Khoa Nguyen, Hoang-Phuong Phan, Jisheng Han, Toan Dinh, Abu Riduan Md Faisal, Sima Dimitrijev, Yong Zhu, Nam-Trung Nguyen and Dzung Viet Dao

Queensland Micro-Nanotechnology Centre, Griffith University, Brisbane, QLD 4111, Australia. E-mail: khoa.nguyen@griffithuni.edu.au, jhan@griffith.edu.au

School of Engineering, Griffith University, Gold Coast, QLD 4215, Australia

Received 30th October 2017, Accepted 8th January 2018

First published on 15th January 2018
Highly sensitive p-type 4H-SiC van der Pauw sensor

Tuan-Khoa Nguyen, Hoang-Phuong Phan, Jisheng Han, Toan Dinh, Abu Riduan Md Foisal, Sima Dimitrijev, Yong Zhu, Nam-Trung Nguyen and Dzung Viet Dao

This paper presents for the first time a p-type 4H silicon carbide (4H-SiC) van der Pauw strain sensor by utilizing the strain induced effect in four-terminal devices. The sensor was fabricated from a 4H-SiC (0001) wafer, using a 1 μm thick p-type epilayer with a concentration of 10^{18} cm^{-3}. Taking advantage of the four-terminal configuration, the sensor can eliminate the need for resistance-to-voltage conversion which is typically required for two-terminal devices. The van der Pauw sensor also exhibits an excellent repeatability and linearity with a significantly large output voltage in induced strain ranging from 0 to 334 ppm. Various sensors aligned in different orientations were measured and a high sensitivity of 26.3 ppm^{-1} was obtained. Combining these performances with the excellent mechanical strength, electrical conductivity, thermal stability, and chemical inertness of 4H-SiC, the proposed sensor is promising for strain monitoring in harsh environments.

Introduction

Silicon carbide (SiC) is a favorable material for high power and electronics devices, and high temperature applications owing to its wide energy-band gap, high break-down voltage, good electrical conductivity, and thermal stability. Taking advantage of the superior properties and availability of SiC wafers, SiC-based MEMS mechanical sensors have been developed for harsh environments. Specifically, SiC-based pressure sensors, accelerometers and strain sensors have been reported with a good performance even when operating at high temperatures. Possessing a superior large energy-band gap (2.3–3.2 eV), SiC-based devices can operate at higher temperatures by eliminating the thermally induced leakage of the minority carriers. Among more than two hundred SiC polytypes, 4H-SiC is favorable for MEMS devices owing to its excellent properties and commercial availability. 4H-SiC possesses the highest energy-band gap of 3.23 eV in comparison with other SiC polytypes, minimizing the number of electron–hole pairs formed in thermal activation across the bandgap, enabling the high temperature stability of 4H-SiC electronic devices and sensors.*

The strain induced effect on the resistivity (i.e. piezoresistive effect) of two-terminal SiC devices has been reported for mechanical sensing applications with relatively high gauge factors (GF). However, resistance-to-voltage and signal-amplification circuitries are typically required for converting resistance changes to output voltages, creating issues in terms of measurement configurations, as well as exhibiting higher signal noises and delays. Additionally, the difference in the temperature coefficient of the four resistors constructing the Wheatstone bridge circuit, leads to a non-zero offset during operations with varying temperature, hindering the sensitivity of the sensors. Moreover, the thermal coefficient of resistance (TCR) contributes significantly to the measurement of two-terminal devices. This results in a reduction in the signal-to-noise ratio and hinders the reliability of strain sensors. In contrast, the variation in respect to the temperature change is minimized in four-terminal devices since the TCR of piezoresistors would be cancelled out. There are numerous studies on the 3C-SiC based four-terminal strain sensor with a relatively high sensitivity and good reproducibility. However, the strain induced effect and strain sensing in four-terminal 4H-SiC have not been investigated. Yet, the development of SiC devices and electronics is shifting towards 4H-SiC and 6H-SiC, which are widely available. These raise the need for the investigation of the strain induced effect in such devices. Therefore, understanding this phenomenon in 4H-SiC is of great interest for the development of highly sensitive, circuit conversion-free strain sensors which have superior performances even in high temperature operations.

This work presents a 4H-SiC van der Pauw (4HVPS) sensor, with a four-terminal configuration, with excellent performance including a good repeatability, linearity and high sensitivity. The use of a four-terminal structure eliminates the need for the resistance-to-voltage conversion. The orientation dependence of the sensor in the 4H-SiC (0001) wafer was also investigated to obtain the high sensitivity of 26.3 ppm^{-1}.

*Queensland Micro-Nanotechnology Centre, Griffith University, Brisbane, QLD 4111, Australia. E-mail: khoa.nguyentuan@griffithuni.edu.au; j.han@griffith.edu.au

School of Engineering, Griffith University, Gold Coast, QLD 4215, Australia
Fabrication of the van der Pauw sensor

The sensors were fabricated from a 4H-SiC (0001) wafer (sourced Ascatron™) which has 4° off-cut from the basal plane (0001) towards the (1120) orientation. The 4H-SiC wafer has a thickness of 350 μm, consisting of 1 μm p-type epilayer, 1 μm n-type buffer layer, and a low-doped n-type substrate. The p-type layer was formed using aluminum dopants, with a concentration of $10^{18}$ cm$^{-3}$, while doping concentration of the n-type layer was also $10^{18}$ cm$^{-3}$ with nitrogen dopants. Fig. 1(a) summarizes the fabrication process of the p-type 4HVP sensors. The process started with a standard wafer cleaning procedure then spin coating/developing 4.3 μm-thick AZ9245 photoresist by a standard lithography process to create a protective mask for the subsequent etching of the p-type layer (step ②). Inductive coupled plasma (ICP) etching was performed to etch p-type 4H-SiC using a STS™ etcher (step ③). The plasma etching employed HCl (500 sccm) at a low pressure of 2 mTorr and the wafer was continuously cooled down by back-side gas cooling in the etching chamber. The etch rate was measured at approximately 100 nm-per-minute and the final etched depth of 1.3 μm was achieved. This ensures that the 1 μm p-type layer was thoroughly etched in the designated area; then mesa square-shape structures were formed on the p-type layer. Titanium and aluminum metal layers were subsequently deposited to form a contact for the sensor, using a SNS™ sputterer with the thickness of each layer being 100 nm. Next, a second photoresist mask was formed on top of the as-etched p-type 4H-SiC patterns (step ④), followed by a wet etching of Ti/Al to create the designated metal contact for subsequent electrical measurements (step ⑤). An annealing process at high temperature up to 1000 °C was conducted to obtain a good ohmic contact. The detailed annealing process can be found in our previous paper. Finally, the wafer was diced into 30 mm × 3 mm × 0.35 mm beams which were aligned to the (1110) direction.

Results and discussion

Fig. 1(b) shows a good linearity of the current-voltage characteristics of the annealed Ti/Al contact to p-type 4H-SiC in the voltage range from −2 to 2 V using a Keithley 2602B System SourceMeter™. Accordingly, the resistivity of the p-type layer was found to be 0.91 Ω cm. Since strain sensors typically operate in small voltage ranges (e.g. below ±1 V) to avoid the Joule heating effect, the measured I-V characteristics in the given range is sufficient for strain sensing applications. It is also necessary to investigate the current leakage through the p-n junction to the n-type layer and the substrate. As can be seen in Fig. 1(b) inset, when sweeping bias voltages from −2 to 2 V, the leakage current to the n-type layer and the substrate is in the range of tens of pico amperes (pA) which is seven orders of magnitude smaller than the current in the sensor (e.g. hundreds of micro amperes (μA)). This is attributed to the fact that the back-to-back p-n diode prevents the current from leaking into the n-type layer and the substrate. In our previous study, we have formed an ohmic contact to the n-type substrate and investigated the vertical current leakage from the epi-layer (p-type) to the bulk substrate (n-type), showing a small current in the nA order. This result indicated that the n-type substrate did not have a significant contribution on the change in the p-type van der Pawn devices under strain. Consequently, the contribution of the underneath layers other than the p-type layer to the electrical measurements of the devices, can be negligible.

To investigate the orientation dependence of the sensors to the induced strain, the p-type 4HVP sensors were aligned to three different orientations which are 0°, 45°, and 90° to (1100) orientation (Fig. 2(a)). There are two most common techniques to investigate the strain induced effect in semiconductors, including diaphragm deformation and bending beam. For instance, biaxial stress could be generated by applying pressure to diaphragms where resisters were located on the top surface, while bending beam methods can induce uniaxial stresses to resisters lying on the top surface of bent beams. In the present work, the operation of the 4HVP sensor under uniaxial strain/stress was performed using a bending beam method. This technique has been confirmed as a simple but effective way for the characterizations of strain induced effect in semiconductor materials. The configuration of the sensor operation under induced stress/strain is illustrated in Fig. 2(b). The SiC beam with one tightly clamped end was bent downward by applying a static force at the free end. Since the
beam has a cantilever-shape, the bending would yield a uniaxial strain to the top p-type layer, which is parallel to the longer edges of the beam. The strain configuration has been reported elsewhere, which in uniaxial strain is effectively transferred to the mesa top structures by the relationship between applied force $F$ and induced strain $\varepsilon$:

$$\varepsilon = \frac{6F}{E_{\text{SiC}}h^2}$$

where $l$, $b$, $t$ and $E_{\text{SiC}}$ are the length, width, thickness and Young’s modulus of the 4H-SiC beam, respectively. The stress induced can be deduced from the Hooke’s law: $\sigma = \varepsilon E_{\text{SiC}}$. The calculation was in good agreement with a finite element analysis (FEA) model shown in Fig. 3.

Fig. 2(c) shows the configuration of the four-terminal strain sensor where the current was applied at the rectangular-shaped-electrodes, while the output voltage was measured at the dot-like-electrodes. According to the Kanda’s model, the 4HVP sensor is a square-type Wheatstone bridge circuit comprised of four resistors $R_{AC}$, $R_{BC}$, $R_{BD}$ and $R_{AD}$. When a constant current is supplied between terminals A and B, in the stress-free state, all resistors are equal ($R_{AC} = R_{BC} = R_{BD} = R_{AD} = R_0$) and the circuit is in its equilibrium, resulting in a null offset voltage ($V_{\text{offset}} = 0$) between the Hall terminals C and D. However, the resistances of the four resistors can vary differently depending on the orientation and magnitude of stress applied to each resistor since p-type 4H-SiC is a piezoresistive material. Upon the application of uniaxial stress in the (1100) orientation, there are asymmetric variations of the four piezoresistors, depending on the magnitude of stress and its relative direction versus the strain orientation. These different resistance changes yield a significant offset voltage between Hall terminals C and D ($V_{\text{CD}}$). This phenomenon is referred to the pseudo-Hall effect in four-terminal devices where a large offset voltage can be generated without the application of an external magnetic field. Consequently, the four-terminal configuration can be employed as a strain sensor where the initial induced strain can be monitored by measuring the output voltage $V_{\text{out}} = V_{\text{CD}}$. The relationship of the output signal $V_{\text{out}}$ versus the four resistors is given by:

$$\frac{V_{\text{out}}}{V_{\text{in}}} = \frac{R_{AC}R_{BD} - R_{BC}R_{AD}}{(R_{AD} + R_{BD})(R_{AC} + R_{BC})}$$

where $V_{\text{in}}$ is the input voltage at the terminals A and B. The asymmetric variations of the four piezoresistors, in respect to the magnitude and orientation of applied stress, yields offset voltage $V_{\text{out}}$. The orientation dependence of the 4HVP sensor was characterized to obtain a high sensitivity. Fig. 4 shows the result of the offset voltage $V_{\text{out}}$ in three different orientations aligned $0^\circ$, $45^\circ$, and $90^\circ$ to (1100) direction with the supplied current of up to 30 $\mu$A. It can be seen that the output signals of the $0^\circ$ and $90^\circ$ aligned devices were almost zero. This can be explained by the symmetric changes of four piezoresistors in the $0^\circ$ and $90^\circ$ sensors regardless of the magnitude of induced strain. That is the resistances $R_{AC}/R_{BC}$ changed with the same amount under a similar longitudinal strain, while the resistance of $R_{BD}$ and $R_{AD}$ almost changed equally under the same compressive strain. In contrast, a significant output voltage was obtained for the sensor with $\Theta = 45^\circ$. This is attributed to the fact that $R_{AC}$ and $R_{BD}$ were elongated in their longitudinal direction parallel to the flowing current whereas $R_{BC}$ and $R_{AD}$ were stretched in transverse direction perpendicular to the current. Therefore, $R_{AC}/R_{BC}$ and $R_{BD}/R_{AD}$ vary with tensile stress followed the longitudinal GF (increase) and transverse GF (decrease), respectively.

The output voltage linearly varied with the applied strain ranging from 0 to 334 ppm (equivalent to an induced stress from 0 to 168 MPa), indicating that the Joule heating effect and the current leakage to the n-type layer and the substrate are
negligible. Let $\Theta$ be the angle between the diagonal of the square-type circuit and the applied stress orientation (e.g. $(1100)$) (Fig. 2(b)). Since the piezo-coefficients of p-type 4H-SiC have not been elucidated yet, there is no general formula, comprised of the four orientation-dependent piezoresistors, for the relationship between the ratio $V_{\text{out}}/V_{\text{in}}$ and the sensor angle $\Theta$. However, it is still possible to deduce for the case of $\Theta = 45^\circ$ in which a uniaxial stress in $h_1/C_22$ orientation parallel to the diagonal axis of the 4HVP sensor. As such, $R_{AC}$ and $R_{BD}$ were elongated in their longitudinal direction parallel to the flowing current whereas $R_{BC}$ and $R_{AD}$ were stretched in transverse direction perpendicular to the current. Therefore, under the application of a uniaxial tensile strain, $R_{AC}$ and $R_{BD}$ vary by the longitudinal GF while the variation of $R_{BC}$ and $R_{AD}$ change by the transverse GF as follows:

$$\begin{align*}
R_{AC} &= R_{BD} = R_0 (1 + G_{LF} \varepsilon) \\
R_{BC} &= R_{AD} = R_0 (1 + G_{FT} \varepsilon)
\end{align*}$$

(3)

where $G_{LF}$ (positive value) and $G_{FT}$ (negative value) are longitudinal and transverse gauge factors of p-type 4H-SiC, which have been reported in ref. 22. From eqn (2) the sensitivity of the 4HVP sensor, for the case of $\Theta = 45^\circ$, is given by:

$$S_0 = \frac{V_{\text{out}}}{\varepsilon V_{\text{in}}} = \frac{G_{LF} - G_{FT}}{2 + \varepsilon(G_{LF} + G_{FT})}$$

(4)

The experimental sensitivity of the sensor was found to be as high as 26.3 ppm $^{-1}$ which are comparable with the theoretical result of 29.4 ppm $^{-1}$ according to eqn (4). The measured sensitivity of 4HVP sensor is relatively high in comparison with other reported results of other SiC polytypes which were from 6 to 30.\textsuperscript{20} It should be noted that the sensitivity of the 4HVP sensor is independent of the applied current. Fig. 5(a) shows the relationship between the output voltage $V_{\text{out}}$ versus the induced strain up to 334 ppm for the sensor with $\Theta = 45^\circ$. Evidently, the linear response of the sensor’s signal was obtained with different applied currents ranging from 5 to 30 $\mu$A. At a certain strain, the output voltage of the sensor was proportional to the applied current as shown in Fig. 5(b). This characteristic is of interest for strain sensing since the sensor’s calibration for different input currents is no longer required.

![Fig. 4](image1.png) **Fig. 4** The relationship between the ratio of output voltage and input voltage versus applied uniaxial strain. The output voltage of the sensors with $\Theta = 45^\circ$ was significant while the sensors with $\Theta = 0$ or $\Theta = 90^\circ$ resulted a nearly zero output signal ($V_{\text{CD}} = 0$).

![Fig. 5](image2.png) **Fig. 5** (a) Output voltage of 4HVP sensor versus applied strains with different input currents of 5, 10, 20, and 30 $\mu$A. (b) Variation of output voltage versus varying applied current ranging from 5 to 30 $\mu$A at a constant strain of 334 ppm.

![Fig. 6](image3.png) **Fig. 6** 4HVP sensor operation with stepped strains from 0 to 334 ppm (top) and at a constant applied strain of 334 ppm ($I = 20\ \mu$A) (bottom). The Hall terminals C and D of the sensor were connected to an amplifier with a gain $g$. The excellent linearity and repeatability were obtained without any signal drift after numerous loading cycles.
Fig. 6 shows a demonstration of the real-time strain monitoring using the 4HVP sensor. The operations under stepped strain and constant strain were carried out with the input current of 20 μA. The output signal amplitude can be amplified and monitored using a configuration shown in the inset of Fig. 6 with a gain of the circuit to be adjustable. Under stepped strain, the sensor responded linearly with the increase or decrease of strain. The signal also remained stable at each strain level. When the load was completely removed, the sensor’s output returned to its initial value. Additionally, with the application of a certain constant strain, the output was the application of a certain constant strain, the output was extremely stable without any signal drift. The measurement results indicate a good linearity and repeatability of the 4HVP strain sensor. This characteristic is of interest for high performance mechanical strain sensing with the high sensitivity and low signal-to-noise ratio.

Conclusions

A highly sensitive 4H-SiC van der Pauw strain sensor was presented using a four-terminal configuration which simplifies the read-out circuit without the need for the Wheatstone bridge to convert resistance change to voltage. By utilizing a bending beam method to apply uniaxial strain/stress to the sensor, a good repeatability and linearity were obtained for the sensor aligned 45° to the (1100) orientation. A high sensitivity of the strain sensor was found to be 26.3 ppm -1 and was independent of the applied current. The sensor is promising for highly sensitive and low noise mechanical sensing in high temperature operations.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

This work was partially funded by the linkage grants LP150100153 and LP160101553 from the Australian Research Council (ARC). This work was also supported by the Queensland node of the Australian National Fabrication Facility, a company established under the National Collaborative Research Infrastructure Strategy to provide nano and micro-fabrication facilities for Australia’s researchers.

References

Chapter 6 4H-SiC based device development for mechanical sensing

6.2 P-type 4H-SiC pressure sensor at cryogenic and elevated temperatures
Highly sensitive 4H-SiC pressure sensor at cryogenic and elevated temperatures

Author: Tuan-Khoa Nguyen, Hoang-Phuong Phan, Toan Dinh, Karen M. Dowling, Abu Riduan Md Foisal, Debbie G. Senesky, Nam-Trung Nguyen, Dzung Viet Dao

Publication: Materials & Design
Publisher: Elsevier
Date: Available online 10 July 2018

Please note that, as the author of this Elsevier article, you retain the right to include it in a thesis or dissertation, provided it is not published commercially. Permission is not required, but please ensure that you reference the journal as the original source. For more information on this and on your other retained rights, please visit: https://www.elsevier.com/about/our-business/policies/copyright#Author-rights

Copyright © 2018 Copyright Clearance Center, Inc. All Rights Reserved. Privacy statement. Terms and Conditions. Comments? We would like to hear from you. E-mail us at customercare@copyright.com
Highly sensitive 4H-SiC pressure sensor at cryogenic and elevated temperatures

Tuan-Khoa Nguyen, Hoang-Phuong Phan, Toan Dinh, Karen M. Dowling, Abu Riduan Md Foisal, Debbie G. Senesky, Nam-Trung Nguyen, Dzung Viet Dao

Queensland Micro-Nanotechnology Centre, Griffith University, Brisbane, Australia
Department of Electrical Engineering, Stanford University, USA
Department of Aeronautics and Astronautics, Stanford University, USA
School of Engineering and Built Environment, Griffith University, Gold Coast, Australia

HIGHLIGHTS
- A highly sensitive bulk silicon carbide pressure sensor was fabricated using a laser scribing method.
- The sensor’s sensitivity was obtained to be 10.83 mV/V/bar at 198 K and 6.72 mV/V/bar at 473 K.
- The sensor shows a two-fold increment of sensitivity in comparison with other silicon carbide pressure sensors.
- The as-fabricated sensor exhibits excellent sensitivity, linearity and reproducibility from cryogenic to elevated temperatures.

ABSTRACT
The slow etching rate of conventional micro-machining processes is hindering the use of bulk silicon carbide materials in pressure sensing. This paper presents a 4H-SiC piezoresistive pressure sensor utilising a laser scribing approach for fast prototyping a bulk SiC pressure sensor. The sensor is able to operate at a temperature range from cryogenic to elevated temperatures with an excellent linearity and repeatability with a pressure of up to 270 kPa. The good optical transparency of SiC material allows the direct alignment between the pre-fabricated piezoresistors and the scribing process to form a diaphragm from the back side. The sensitivities of the sensor were obtained as 10.83 mV/V/bar at 198 K and 6.72 mV/V/bar at 473 K, which are at least a two-fold increment in comparison with other SiC pressure sensors. The high sensitivity and good reliability at either cryogenic and elevated temperatures are attributed to the profound piezoresistive effect in p-type 4H-SiC and the robust p-n junction which prevents the current from leaking to the substrate. This indicates the potential of utilising the laser scribing approach to fabricate highly sensitive bulk SiC pressure sensors for harsh environment applications.

1. Introduction
Micromachined-pressure sensors are positioned as the most important and ubiquitous micro-electromechanical systems (MEMS) sensing devices. In terms of the sensing mechanism, capacitive and piezoresistive transducers play a dominant role with the simple read-out, good reliability and integration [1–3]. Piezoresistive sensors possessed several advantages in comparison with capacitive counterparts such as the easiness of design configuration and the wider linearity range [4–7]. In the past four decades, Si remains as the most important material used for pressure sensors owing to its...
wide availability and mature fabrication process. However, its intrinsic physical properties, such as the low energy band gap and plastic deformation, have limited its usage in harsh environments. In contrast, the superior mechanical strength, excellent corrosive/shock resistance and high stability at high temperatures position silicon carbide as a promising material for extreme condition sensing. In fact, numerous SiC pressure sensors have been reported including capacitive and piezoresistive sensors [8–10]. For instance, Young et al. characterized a 3C-SiC[100] capacitive pressure sensor with a sensitivity of 7.7 fF/torr at 400 °C [11]. Wu et al. reported a SiC-on-SiO2 piezoresistive pressure sensor with sensitivity of 101.5 V/V/psi at room temperature and 53.4 μV/V/psi at 385 °C [12,13]. Wieczorek et al. reported a 6H-SiC pressure sensor with a sensitivity of 330 μV/V/bar at 23 °C and 200 μV/V/bar at 400 °C [14]. Okojie et al. found a sensitivity recovery at high temperatures of a 4H-SiC pressure sensor with good linearity [15].

The use of bulk SiC materials (e.g. 4H-SiC and 6H-SiC) for pressure sensors can eliminate the thermal expansion mismatch between the sensing layer and supporting diaphragm and extend the working regime of the sensing devices. Moreover, the high energy band gap of 4H-SiC (i.e. 3.23 eV) significantly reduces the number of thermal activated electron-hole pairs, improving the reliability and stability of 4H-SiC based sensors in extreme conditions [16,17]. However, the high resistance to chemical substances of bulk SiC polytypes makes the etching very challenging and expensive. For instance, the only technique to chemically etch SiC is to use molten salt fluxes and hot gases with an electrochemical processing [18]. These corrosive media typically require expensive Pt beakers and masks to withstand those molten solutions. Moreover, the plasma etching of SiC results in a very slow etch rate, making it impractical to form a diaphragm for the pressure sensors from bulk SiC wafers. To overcome the challenge, Akiyama et al. employed a mechanical drilling process to etch bulk SiC material forming a 4H-SiC pressure sensor [19]. This method requires specified wafer holders and alignment tools that significantly increase the preparation time and fabrication cost.

In this paper, we present a laser scribing approach to fabricate a highly sensitive 4H-SiC piezoresistive pressure sensor from a bulk SiC wafer. The good optical transparency of SiC material allows the direct alignment between the pre-fabricated piezoresistors and the laser-scribed diaphragm on the back side. Furthermore, the experimental results show that the sensor exhibits good performance from a cryogenic temperature of 198 K to a high temperature of 473 K. A sensitivity of 8.24 mV/V/bar was achieved at room temperature, slightly increased to 10.83 mV/V/bar at 198 K and decreased to 6.72 mV/V/bar at 473 K. The high sensitivity at cryogenic and elevated temperature is attributed to the profound piezoresistive effect in p-type 4H-SiC and the robust p-n junction which effectively prevent the current from leaking to the substrate. Moreover, the good reproducibility for hundreds of pressurising cycles and excellent stability at cryogenic and high temperatures were also realized.

2. Design and fabrication of 4H-SiC pressure sensor

2.1. Pressure sensor design

In terms of design, circular and rectangular diaphragms are the two common geometries for micromachined pressure sensors. The sensing element is typically placed in the vicinity of the edge of the diaphragm to obtain the maximum sensitivity since the induced stress/strain is maximised at the given area. While the stress/strain is equally distributed along the circumference of a circular diaphragm, in a rectangular diaphragm, it is concentrated in the middle of the edges. From the plate theory, the maximum stress/strain of the square diaphragm is 1.64 times higher than that of a circular diaphragm with equivalent dimensions [20]. Thus, placing the sensing element in the middle of the edges of a rectangular diaphragm would yield the maximum sensitivity for the pressure monitoring. Therefore, in this paper, a square-shaped diaphragm was designed and fabricated with piezoresistors located in the middle of an edge, as illustrated in Fig. 1. An applied differential pressure would deform the diaphragm and induce strain to the piezoresistor lying on the top surface of the diaphragm. Since the sensor is made of 4H-SiC, a piezoresistive material, the magnitude of applied strain can be realized by monitoring the resistance change of the sensor. Moreover, by establishing a straining model, it is possible to detect the input pressure applied to the diaphragm. The stress/strain distribution of a square diaphragm under pressure can be characterized using the Bubnov-Galerkin model for a rectangular thin film based on the maximum deflection of the diaphragm under pressure. The simplified function for the strain distribution along the centre line \((x = (-a/2,a/2), y = 0)\) of a square diaphragm is deduced as [21]

\[
f(x) = \frac{7P}{2304\pi^2D^2} \left( \frac{x^2 - a^2}{4} \right)^2
\]

where \(f(x), P\) and \(a\) are the deflection function, the edge length of the square diaphragm, and the applied pressure, respectively; \(D = E t^3/(12(1-\nu))\) is the flexural rigidity of the diaphragm; \(t\) is the diaphragm thickness; \(\nu\) and \(E\) are the Poisson’s ratio and Young’s modulus of 4H-SiC, respectively. It is worth noting that when the diaphragm deflection is much smaller than the thickness \((f \ll t)\), the relationship between \(P\) and \(f\) is linear.

The strain function with respect to the small deflection of a square diaphragm can be given as

\[
s(x) = \frac{3a^2 f}{4k^2}
\]

where \(z\) denotes the dimension that is perpendicular to the diaphragm plane. The U-shaped piezoresistor consists of two long resistors \(R_l\) and one short resistor \(R_t\). Upon an applied pressure, \(R_l\) varies by longitudinal gauge factor \((G_l)\) while the \(R_t\) change according to transverse gauge factor \((G_t)\), and either \(G_l\) and \(G_t\) were reported in our previous work [22]. Thus, the fractional change of resistance of the combined piezoresistor can be given as

\[
\frac{\Delta R}{R} = \int_0^l \left( \frac{G_l}{R_l} \Delta x + \frac{G_t}{R_t} \Delta z \right)
\]

where \(\Delta(x)\) is the function of the localised strain (see supplementary document for the detailed calculation) and \(\xi_z\) is the strain at the location of \(R_t\), \(\xi_l = 2R_l/R\) and \(\xi_t = R_t/R\) are the ratio of the longitudinal
and transverse resistances. Thus, the sensitivity of the sensor can be given as

$$S = \left| \frac{\Delta R/R}{P} \right| = \left| \frac{\Delta I/I}{P} \right|. \quad (4)$$

The detailed sensitivity analysis can be found in the supplementary document.

2.2. Fabrication

Fig. 2(a) shows the fabrication process of the 4H-SiC pressure sensor. The initial 4H-SiC wafer consists of 1 μm p-type, 1 μm n-type layers and a 350 μm low doped substrate. The sensor was fabricated from p-type 4H-SiC in the s-face (i.e. (0001) face). The U-shaped piezoresistors were patterned on the p-type layer by a photo lithography (step 1) and inductive coupled plasma (ICP) etching (step 2) using a STS™ etcher at an etch depth of 1.25 μm, ensuring that the p-type functioning layer was thoroughly etched and the piezoresistors electrically isolated from the substrate (Fig. 2(b)). The ICP etching rate was approximately 100 nm/min. Next, Ti/Al metallization (step 3) was patterned on top of the p-type layer, following a rapid thermal annealing process (RTA) at 1000 °C to obtain a good Ohmic contact for the sensor's characterization. It is known that p-type 4H-SiC is a wide band gap material with a different work function from that of the metal contact (i.e. Ti/Al), a potential barrier is formed after the metallization process [23]. This leads to the Schottky contact, in which the current-voltage characteristics shows a rectifying behaviour when sweeping a voltage range from negative to positive. The sheet contact resistances before and after the annealing process at were measured to be 2.1 MΩ and 26.7 kΩ, respectively. This means that the thermal annealing has greatly improved the Ohmic behaviour of the contact between p-type 4H-SiC and Ti/Al. The wafer was subsequently diced into 10 x 10 mm² chips. Finally, the square-shaped diaphragms were formed by scribing the back side of each chip (step 4) by a diode-pumped Nd/YVO₄ laser with a peak power of up to 1.5 kW and the average scribing power was 1–3 W (Fig. 2(c), (d)). The details of laser scribing process can be found elsewhere [24]. First, the 4H-SiC sample (with the pre-fabricated piezoresistor) was placed in the chamber to align the laser focus. Subsequently, the scribing process was conducted on the back of the 4H-SiC chip (engraving on the side without the piezoresistor). The material was ablated layer-by-layer using the laser beam with the cross-hatch patterning until the desired depth is achieved. The total time for the laser scribing procedure was just approximately 25 min. The scribing time is far less than that of other etching processes for bulk SiC materials (e.g. inductive plasma etching, deep reactive-ion etching (DRIE)) which could take many hours or even impractical to conduct for the etch depth of hundreds of micrometres. It should be noted that owing to the transparency of the SiC wafer, the alignment of back side scribing to the pre-fabricated piezoresistor in the front side was made straightforward. Fig. 2(c) shows the side-wall and surface roughness of the back side of the diaphragm after the laser scribing process. The final dimensions and thickness of the diaphragms were measured to be 5 x 5 mm² and 70 μm, respectively. After the fabrication process, the chips were attached to ceramic chip carriers which are aimed to work at cryogenic and high temperatures, as shown in Fig. 2(e). The electrical connections were formed by wire bonding from the Ti/Al contact to the contact pads of the chip carrier. The enclosed cavity underneath the 4H-SiC diaphragm was sealed by high-temperature epoxy.

3. Results and discussion

Fig. 2(f) shows the experimental setup for the characterization of the 4H-SiC pressure sensor. The 4H-SiC chip attached to a chip carrier was placed in the chamber of a Linkam™ THMS600. First, the sensor operation at room temperature was characterized. The measurement started with supplying controlled air pressure to the enclosed chamber using a pressure regulator. In order to avoid oxidation at elevated temperature, argon was supplied as the medium in the pressurizing chamber. A constant DC voltage of 1 V was applied to the piezoresistor in the measurement. Subsequently, the output current was monitored by an external read-out device (Agilent™ 1500).

Fig. 3(a) shows the recorded real-time output signal which varied linearly with the increase of the applied pressure from 0 to 268 kPa. Moreover, an increasing resistance with applied pressure can also be observed in Fig. 3(b), indicating that the piezoresistor is in the tensile stress state (the resistance increases with strain). The measured data shows the good linearity of the output signal and the input pressure with high signal-to-noise ratio. It should be noted that when
Fig. 3. a) Real-time measurement of output signal at varying applied pressure ranging from 0 to nearly 270 kPa. b) Linear resistance change upon the application of pressure. c) Output signal at a cyclic applied pressure of 186 kPa, high signal-to-noise ratio and the excellent reproducibility without significant signal drift were obtained. The differential pressure was completely removed, the output signal returned to its initial value without any drift. Another measurement with a cyclic pressure of 186 kPa was also performed, confirming the excellent repeatability of the sensor with cyclic pressure, as shown in Fig. 3(c). The experimental data exhibits the good reproducibility and linearity of the output signal which is crucial for pressure sensing which typically requires long-term stability. It is also necessary to determine the time response of the pressure sensor. From a finite element analysis (FEA) using COMSOL™, the first order resonant frequency of the membrane \( f_1 \) was found to be 108 kHz, corresponding to the response in the time domain of 9.2 \( \mu \text{s} \). This value is equivalent to the time response of other reported SiC pressure sensors.

To demonstrate the capability of the as-fabricated sensor for harsh environments, the operation of the sensor at cryogenic and elevated temperatures were characterized. It is known that many pressure sensors exhibit good performances at room temperature but at high temperatures, there is a significant reduction in the sensitivity and reliability due to the thermal induced leakage current to the substrate. Therefore, the current-voltage characteristics at a temperature range from 273 K to 473 K was measured as shown in Fig. 4. It should be noted that the leakage is four orders of magnitude smaller than the current flowing in the p-type piezoresistors thanks to the robust p-n junction. This barrier layer acts as a back-to-back diode which prevents the electric current leaking to the substrate at the high temperatures [22,25]. Consequently, it can be concluded that only the p-type layer contributed to the measurement of the piezoresistive pressure sensor.

Fig. 4 shows the measured sensitivity of the 4H-SiC pressure sensor at a temperature range from 198 K to 473 K. A sensitivity of 10.83 mV/V/bar was realized at 198 K then gradually decreased to 6.72 mV/V/bar at 473 K. This decrease in sensitivity by temperature can be explained by the strain induced effect of the electrical conductance of p-type semiconductor materials with respect to the temperature variation. The electrical conductivity in p-type 4H-SiC is mainly attributed to the hole transfer in the three top highest valence bands (i.e. the heavy hole (HH), light hole (LH), and spin-orbit split-off (SOSO) bands). Since 4H-SiC is an \( \alpha \)-type SiC in which spin-orbit interaction has weak effect on the shape of the valence bands [26], the electrical conductance can be deduced as [27,28]

\[
\sigma_{4H\text{-SiC}} = \frac{q^2}{\tau \left( \frac{p_1}{m_1} + \frac{p_2}{m_2} \right)}
\]  

(5)

where \( q \), \( \tau \), \( p_i \), and \( m_i \) are the electron charge, the relaxation time, the hole concentration (\( i = 1 \) denotes the HH band and \( i = 2 \) represents the LH band), and the effective mass, respectively. The application of a uniaxial stress lifts the degeneracy of the HH and LH bands, altering the resistivity of the piezoresistor. This phenomenon is attributed to the hole transfer mechanism which is the dominant factor of the piezoresistivity in p-type semiconductors. Subsequently, in the small strain region (\( \Delta E_V \ll 2k_B T \)) the relative variation of the hole concentration in the HH and LH band is given as [27]

\[
\frac{\Delta R}{R} = \frac{\Delta p_i}{p_i} = -\frac{1}{k_B T} \left( \frac{m_i^*}{m_j^*} \right)^{3/2} \Delta E_V
\]  

(6)

Fig. 4. The measured sensitivity at various temperature ranging from 198 K to 473 K. It is worth noting that the decreasing of sensitivity with increasing temperature is in good agreement with the hole transfer mechanism under strain with the varying temperature. Inset: Linear current-voltage characteristics of the sensor measured at various temperatures.
where \( m^* \) are the density-of-states effective masses, \( p \) are the hole concentration, \( \Delta E_v \) is the band splitting energy and \( k_B \) is the Boltzmann constant. Assuming that the component \( \Delta E_v / (1 + (m^*/m_j)^{3/2}) \) is independent on temperature change, the relative resistance change or the sensitivity is inversely proportional to the increase of the ambient temperature. Therefore, the measured sensitivity of the 4H-SiC in the given temperature range is in good agreement with the aforementioned variation with temperature of the hole transfer mechanism in p-type 4H-SiC.

4. Conclusion

In summary, we present the fabrication and characterization of a highly sensitive 4H-SiC pressure sensor using a laser scribing approach. The sensor was aimed to work in a wide range of temperature from 198 K to 473 K with high sensitivities of 10.83 mV/V/bar at 198 K and 6.72 mV/V/bar at 473 K. These results exhibited a two-fold sensitivity increase in comparison with other reported SiC pressure sensors with the excellent linearity and repeatability. The variation of the sensor’s piezoresistance with temperature can be explained by the hole transfer mechanism between the light hole and heavy hole bands of p-type 4H-SiC. The high sensitivity and good reliability at either cryogenic and elevated temperatures were achieved thanks to the profound piezoresistive effect of p-type 4H-SiC material and the robust p-n junction which prevents the current from leaking to the substrate at either cryogenic and high temperatures. The good Ohmic contact was formed using a rapid thermal annealing at 1000°C, which is then confirmed by the linear current-voltage characteristic at the temperature range. This temperature-tolerance Ohmic characteristic is favourable for the piezoresistive sensing in terms of sensitivity and reliability. The as-presented laser scribing approach shows the feasibility of fast prototyping bulk SiC pressure sensors for harsh environment sensing.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

Acknowledgments

This work has been partially supported by Australian Research Council (ARC), Australia grants LP15010153 and LP160101533. This work was performed in part at the Queensland node of the Australian National Fabrication Facility (ANFF), Australia, a company established under the National Collaborative Research Infrastructure Strategy to provide nano- and micro-fabrication facilities for Australian researchers. T.-K. Nguyen acknowledges Postgraduate Research Scholarship (GUIPRS) and International Postgraduate Research Scholarship (GUIPRS) from Griffith University.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.matdes.2018.07.014.

References


[8] T.-K. Nguyen, H.-P. Phan, H. Kambale, R. Vadivelu, T. Dinh, A. Iacopi, G. Walker, L. Hold, N.-T. Nguyen, D.V. Dau, Superior robust ultrathin single-crystal silicon cantilever sensors with the excellent linearity and repeatability. The variation sensitivity increase in comparison with other reported SiC pressure sensors and the robust p-n junction which prevents the current from leaking to the substrate at either cryogenic and high temperatures. The good Ohmic contact was formed using a rapid thermal annealing at 1000°C, which is then confirmed by the linear current-voltage characteristic at the temperature range. This temperature-tolerance Ohmic characteristic is favourable for the piezoresistive sensing in terms of sensitivity and reliability. The as-presented laser scribing approach shows the feasibility of fast prototyping bulk SiC pressure sensors for harsh environment sensing.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

Acknowledgments

This work has been partially supported by Australian Research Council (ARC), Australia grants LP15010153 and LP160101533. This work was performed in part at the Queensland node of the Australian National Fabrication Facility (ANFF), Australia, a company established under the National Collaborative Research Infrastructure Strategy to provide nano- and micro-fabrication facilities for Australian researchers. T.-K. Nguyen acknowledges Postgraduate Research Scholarship (GUIPRS) and International Postgraduate Research Scholarship (GUIPRS) from Griffith University.
Highly sensitive 4H-SiC pressure sensor at cryogenic and elevated temperatures

Tuan-Khoa Nguyen, Hoang Phuong Phan, Toan Dinh, Abu Riduan Md Foisal, and Nam-Trung Nguyen
Queensland Micro-Nanotechnology Centre,
Griffith University, Brisbane, Australia
Karen M. Dowling
Department of Electrical Engineering, Stanford University, USA
Debbie G. Senesky
Department of Aeronautics and Astronautics, Stanford University, USA
Dzung Viet Dao
Queensland Micro-Nanotechnology Centre,
Griffith University, Brisbane, Australia and
School of Engineering, Griffith University, Gold Coast, Australia

Strain distribution in a square diaphragm under a uniformly distributed load

The governing equation of a square diaphragm under pressure $P$ is given from the Von Karman theory of the superposition of plates [1]:

$$
D\nabla^4 f = tL(f,\phi) + P \\
2\nabla^4 \phi = EL(f,f)
$$

(1)

where $\nabla^2$ is the Laplacean operator, $f$ is the deflection function of the SiC membrane, $D = Et^3/(12(1-\nu))$ is the flexural rigidity of the diaphragm, $t$ and $E$ are the diaphragm
FIG. 1. a) Normalized strain distribution along centre line \( y=0, x=(-a/2,a/2) \). b) Finite element analysis of strain distribution on the diaphragm at an applied pressure of 1bar (\( \sim 100\text{kPa} \)) using COMSOL\textsuperscript{TM}.

thickness and Young’s modulus, respectively; the Airy’s stress function \( \phi \) is given by

\[
\nabla^4 \phi(x,y) = E \left[ \left( \frac{\partial^2 w}{\partial x \partial y} \right)^2 - \frac{\partial^2 w \partial^2 w}{\partial x^2 \partial y^2} \right]
\]

and the Lagrange function \( L \) is defined as

\[
L(f, \phi) = \frac{\partial^2 \phi}{\partial y^2} \frac{\partial^2 f}{\partial x^2} - 2 \frac{\partial^2 \phi}{\partial x \partial y} \frac{\partial^2 f}{\partial x \partial y} + \frac{\partial^2 \phi}{\partial x^2} \frac{\partial^2 f}{\partial y^2}
\]

Using the Bubnov-Galerkin’s first order approximation, the general equation for the deflection of the square diaphragm is

\[
f(x,y) = \frac{7P}{144a^4D}(x^2 - \frac{a^2}{4})^2(y^2 - \frac{a^2}{4})^2
\]

Considering the piezoresistor lying on the center line of the square \( y=0 \), we have

\[
f(x,0) = \frac{7P}{2304a^4D}(x^2 - \frac{a^2}{4})^2
\]

The strain equation is

\[
\varepsilon(x) = z \frac{\partial^2 f}{\partial x^2} = \frac{7zP}{2304a^4D}(12x^2 - a^2)
\]
The fractional change of the resistance upon pressure can be calculated as

\[
\frac{\Delta R}{R} = 2 \int_{-a/2}^{-a/2+l_1} \bar{r}_l G_l \varepsilon(x) dx + \bar{r}_l G_t \varepsilon_t = \alpha G_t \int_{-a/2}^{-a/2+l_1} (12x^2 - a^2) dx + \bar{r}_l G_t \varepsilon_t
\]

where \( l_1 \) is the length of the U-shaped piezoresistor, \( \alpha = (14zP\bar{r}_l)/(2304a^4D) \), and \( \varepsilon_t \) is the strain at the location of the transverse resistor (i.e. \( \varepsilon_t = \varepsilon(-a/2 + l_1) \)). For example, at an applied pressure of 1 bar (\( \sim 100 \text{kPa} \)), the resistance change at room temperature is calculated as \( \Delta R/R = 8.92 \times 10^{-3} \).

Summary

In this chapter, the device integration aspect was demonstrated by a p-type 4H-SiC van der Pauw strain sensor and a 4H-SiC piezoresistive pressure sensor utilising a laser scribing approach. Possessing excellent repeatability and linearity combined with the superior mechanical strength, electrical conductivity, thermal stability, and chemical inertness of 4H-SiC, the proposed sensors are promising for strain/pressure monitoring in harsh environments.
Chapter 7

Conclusion and future work

7.1 Conclusion

This thesis investigated the piezoresistive effect in p-type 4H-SiC micro structures and developed 4H-SiC based sensing devices. The following conclusions are remarked:

(i) The magnitude of the piezoresistive effect in p-type 4H-SiC was characterized in which the longitudinal GF has a positive value of about 31 while the transverse GF has a negative value of approximately -27. The significantly high GFs found in p-type 4H-SiC demonstrate the potential for mechanical sensing applications. Additionally, it is confirmed that the mobility of p-type 4H-SiC based devices can be improved by employing strain engineering in the transverse direction.

(ii) The isotropic piezoresistance in the (0001) plane of p-type 4H-SiC was discovered by the calculations of the hole energy shift in the top valence bands and the coordinate transformation. The interesting phenomenon was also verified by the experimental results using a bending beam method where a uniaxial strain was applied to the piezoresistor. The longitudinal and transverse PZR coefficients were found to be $6.43 \times 10^{-11}$ Pa$^{-1}$ and $-5.12 \times 10^{-11}$ Pa$^{-1}$, respectively, which are independent of the strain orientation in the (0001) plane. This isotropic piezoresistance is attributed to the isotropic hole energy shift under uniaxial stress/strain in the basal plane. The relatively high and isotropic piezoresistivity of p-type 4H-SiC is promising for...
mechanical sensing and strain engineering since a high sensitivity can be achieved regardless of the sensor orientation.

(iii) At high temperatures, large gauge factors of p-type 4H-SiC were obtained with an excellent linearity and stability. At 600°C, the GF was found to be approximately 23 which is at least ten times higher than the conventional metal strain gauges. The hole redistribution model was also proposed which agrees with the variation under temperature change of the piezoresistivity of 4H-SiC. The large gauge factor of p-type 4H-SiC demonstrates the potential for mechanical sensing in extreme environments where the use of Si and polymer counterparts is infeasible.

(iv) A highly sensitive 4H-SiC van der Pauw strain sensor was developed using a four-terminal configuration without the need for the Wheatstone bridge circuitry to convert the resistance change to voltage. By utilizing a bending beam to apply uniaxial strain/stress, a good repeatability and linearity were obtained for the sensor aligned 45° to the \(<1\overline{1}00>\) orientation. A high sensitivity of the strain sensor was found to be 26.3 ppm\(^{-1}\) and was independent of the applied current. The sensor is promising for highly sensitive and low noise mechanical sensing in high temperature operations.

(v) The fabrication and characterisation of a highly sensitive 4H-SiC pressure sensor using laser scribing was carried out. The sensor was able to work in a wide range of temperature from 198K to 473K with high sensitivities of 10.83 mV/V/bar at 198K and 6.72 mV/V/bar at 473K. These results exhibited a two-fold sensitivity increase in comparison with other reported SiC pressure sensors with the excellent linearity and repeatability. The variation of the sensor’s piezoresistivity with temperature can be explained by the hole transfer mechanism between the light hole and heavy hole bands of p-type 4H-SiC. The high sensitivity and good reliability at either cryogenic and elevated temperatures were achieved owning to the profound piezoresistive effect of p-type 4H-SiC material and the robust p-n junction which prevents the current from leaking to the substrate at either cryogenic and high temperatures. Good Ohmic contact was achieved after rapid thermal annealing process at 1000°C. This temperature-tolerance Ohmic characteristic is favourable for the piezoresistive sensing in terms of sensitivity and reliability. The laser scribing approach allows fast prototype of bulk SiC pressure sensors which are suitable for harsh environments.
7.2 Future work and outlook

The fundamental piezoresistive effect at room and high temperatures and the piezoresistive coefficients in (0001) of 4H-SiC have been discovered. Moreover, the device integration aspects have also been demonstrated through this thesis. To further explore the capability of p-type 4H-SiC material for mechanical sensing in harsh environments, the following future work can be carried out:

(i) Investigation of other piezoresistive coefficients in 4H-SiC

In this thesis, the piezoresistance coefficients in the (0001) plane was presented. However, more efforts are required to fully discover piezoresistive coefficients in all crystalllographic orientations of 4H-SiC. This is imperative for the development of highly sensitive 4H-SiC based sensors and electronics for harsh environment applications. Additionally, although 4H-SiC exhibits the high and isotropic piezoresistance in the basal (0001) plane, it is still unclear if the isotropic effect also occurs in other planes.

(ii) Characterization of temperature dependence of pseudo-Hall effect in 4H-SiC

In terms of the theoretical analysis, the variation in respect to the temperature change is minimized in four-terminal devices since the temperature coefficient of resistance of the piezoresistors will be cancelled out. This results in the self-temperature compensation which eliminates the requirement for external circuitries and is considered as an significant advantage of using four-terminal devices in mechanical sensing over two-terminal configurations. Therefore, the operation of 4H-SiC four-terminal devices in high temperatures is needed to be characterized to demonstrate the advantages in high temperatures.

(iii) Characterization of piezo-Hall effect in 4H-SiC at room and high temperatures

The piezo-Hall effect is an important mechanism for magnetic field sensors. There are a large number of Si based piezo-Hall devices in literature. However, its intrinsic physical properties, such as the low energy band gap and plastic deformation, have limited its usage in harsh environments. On the other hand, the superior mechanical strength and high stability at high temperatures make silicon carbide a promising material for extreme condition sensing. Therefore, it is necessary to investigate the
piezo-Hall effect in 4H-SiC in order to develop 4H-SiC magnetic field sensors in harsh environments.

(iv) Characterization of the piezoresistive effect in p-type 4H-SiC nano wires

Further investigations on the piezoresistive effect in p-type 4H-SiC will be developed towards miniaturization with nanostructures. It is possible to fabricate 4H-SiC NWs by a top-down approach utilising FIB method. In such small scale, the piezoresistive effect will be expected to result in many interesting phenomena.

In the recent studies shown in this thesis, the main focus regarding harsh environment is the extreme temperature applications. In the future work, utilising the good chemical inertness of 4H-SiC, the harsh environments can be extended to chemical aggressive media, high shock, and intensive vibration etc.