

OPTICAL STUDIES OF ION IRRADIATED SILICON BASED PHOTONIC STRUCTURES

CHANDRA PRAKASH VERMA



**DEPARTMENT OF PHYSICS
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OPTICAL STUDIES OF ION IRRADIATED SILICON BASED PHOTONIC STRUCTURES

by

CHANDRA PRAKASH VERMA

Department of Physics

Submitted in fulfillment of the requirements for the degree of

Doctor of Philosophy

to the



**DEPARTMENT OF PHYSICS
INDIAN INSTITUTE OF TECHNOLOGY DELHI
FEBRUARY 2023**

To my Parents
&
Supervisor

CERTIFICATE

This is to certify that the thesis entitled "**Optical Studies of Ion Irradiated Silicon Based Photonic Structures**" is being submitted by **Mr. Chandra Prakash Verma** to the Indian Institute of Technology Delhi, New Delhi, for the award of the degree of **Doctor of Philosophy** in Physics is a record of bonafide research work carried out by him under my supervision and guidance. He has fulfilled the requirements for submission of the thesis, which has reached the requisite standard to the best of my knowledge.

The material contained in the thesis has not been submitted in part or full to any other University or Institute for the award of any degree or diploma.

Prof. G. Vijaya Prakash

Professor
Department of Physics
Indian Institute of Technology Delhi
New Delhi-110016, India

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(Chandra Prakash Verma)

ABSTRACT

Photonic structures are tailor-engineered materials that comprise light wavelength ordered periodicity of the modulated refractive index with great potential in optoelectronic devices technology. Recently, porous silicon (PSi)-based photonic structures provide scientific and technological distinctive possibilities due to the unique property of PSi to combine with foreign materials to form functional systems. Further, the optical, electrical, and multi-dynamic PSi properties demonstrate their advantages and advances in the development of next-generation photonic, electronics, and optoelectronic devices. In this increasing study age, PSi has shown its worth in various technical applications, including semiconductor optics, materials science, photovoltaics, sensors, biomedical, and biotechnology.

This thesis elaborates on the fundamental features of semiconductors, Si, and Si-based nanoporous photonic architectures such as distributed Bragg reflectors (DBRs) and microcavities and their practical implications. The broad goal is to investigate the irradiation/implantation effects on the optical properties and bring out the merit of PSi-based structures (DBRs, MCs, etc.). The present study is primarily based on a comprehensive investigation of PSi-based optical microcavities after exposure to different ions (Ar^+ , N, O, H^+ , and He^+) beam irradiation/implantation of various energies (10 keV–1 MeV) and with a range of ion fluences (1×10^{13} – 1×10^{16} ions/cm²). The microcavities are realized by an active $\lambda/2$ spacer layer sandwiched between two DBRs fabricated by galvanostatic electrochemical etching of heavily doped p^+ -type Si. Understanding the physical and chemical mechanisms of ion irradiation/implantation composite formation and their effects on PSi properties is the key objective of the thesis. Therefore, the current work focuses on explaining the inexpensive electrochemical methodology and subsequent modifications in structural, surface morphology, and optical properties of new PSi systems after the various ion-matter interactions.

The dominant resonant cavity peak of the microcavities shows a notable redshift of ~10–60 nm towards the higher wavelength region, and significant color contrast is evident in the reflection images after irradiations. These experimental results compare well with the ion propagation and are in good agreement with transfer matrix method (TMM) simulations. A substantial modification of PSi microcavity surface states is visualized through the Raman and X-ray photoelectron spectroscopies (XPS). The tunability of the optical characteristics arises due to surface modification in the Si–Si and Si–O structural bonds and thereby change in the refractive index of individual PSi layers of the microcavity induced by ions. These experimental results, along with analytical simulations and cavity modelling, conclusively support the realization of cavity tunability and substantial modification in the optical field intensity and photon confinement within the microcavity. The results suggest that ion irradiation could be an effective and practical method to produce highly stable, broadly optically tunable, and designer-suitable photonic structures. These comprehensive studies of these unique ion-matter frameworks bring up new possibilities to explore fundamental features as well as advanced and novel optoelectronic applications.

फोटोनिक संरचनाएं अनुकूलित सामग्री हैं जिनमें ऑप्टोइलेक्ट्रॉनिक उपकरण प्रौद्योगिकी में बड़ी क्षमता के साथ संशोधित अपवर्तक सूचकांक की प्रकाश तरंग दैर्ध्य क्रमित आवधिकता शामिल है। हाल ही में, छिद्रपूर्ण सिलिकॉन (पीएसआई) आधारित फोटोनिक संरचनाएं कार्यात्मक प्रणालियों को बनाने के लिए बाह्य सामग्रियों के साथ गठबंधन करने के लिए पीएसआई की अनूठी संपत्ति के कारण वैज्ञानिक और तकनीकी विशिष्ट संभावनाएं प्रदान करती हैं। इसके अलावा, ऑप्टिकल, इलेक्ट्रिकल और बहु गतिशील पीएसआई गुण अगली पीढ़ी के फोटोनिक, इलेक्ट्रॉनिक्स और ऑप्टोइलेक्ट्रॉनिक उपकरणों के विकास में अपने फायदे और प्रगति प्रदर्शित करते हैं। अध्ययन के इस बढ़ते युग में, पीएसआई ने अर्धचालक प्रकाशिकी, सामग्री विज्ञान, फोटोवोल्टिक्स, सेंसर, बायोमेडिकल और जैव प्रौद्योगिकी सहित विभिन्न तकनीकी अनुप्रयोगों में अपना मूल्य दिखाया है। यह शोध प्रबंध अर्धचालक, सिलिकॉन, और सिलिकॉन-आधारित नैनोपोरस फोटोनिक आर्किटेक्चर जैसे वितरित ब्रैग रिफ्लेक्टर (डीबीआर) और माइक्रोकैविटी और उनके व्यावहारिक प्रभावों की मूलभूत विशेषताओं पर विस्तृत है। व्यापक लक्ष्य, ऑप्टिकल गुणों पर विकिरण/आरोपण प्रभावों की जांच करना और पीएसआई-आधारित संरचनाओं (डीबीआर, एमसी, आदि) की योग्यता को सामने लाना है। वर्तमान अध्ययन मुख्य रूप से, विभिन्न आयनों (एआर, एन, ओ, एचई, और एच), विभिन्न ऊर्जाओं (10 केईवी-1 एमईवी) और एक श्रेणी के आयन फ्लुएंस के साथ (1×10^{13} - 1×10^{16} आयन/सेमी²) के बीम विकिरण/आरोपण के संपर्क में आने के बाद पीएसआई-आधारित ऑप्टिकल माइक्रोकैविटी की एक विस्तृत जांच पर आधारित है। माइक्रोकैविटी को एक सक्रिय $\lambda/2$ स्पेसर परत द्वारा महसूस किया जाता है जो भारी मिश्रित पी⁺ टाइप सिलिकॉन के गैल्वानोस्टैटिक इलेक्ट्रोकेमिकल नक्काशी द्वारा निर्मित दो डीबीआर के बीच स्थित होता है। आयन विकिरण/आरोपण समग्र गठन के भौतिक और रासायनिक तंत्र और पीएसआई गुणों पर उनके प्रभाव को समझना शोध प्रबंध का मुख्य उद्देश्य है। इसलिए, वर्तमान कार्य सस्ती इलेक्ट्रोकेमिकल पद्धति और विभिन्न आयन-पदार्थ इंटरैक्शन के बाद नये पीएसआई प्रणाली के

संरचनात्मक, सतह आकृति विज्ञान और ऑप्टिकल गुणों में बाद के संशोधनों को समझाने पर केंद्रित है। माइक्रोकैविटीज का प्रमुख अनुनाद गुहा शिखर उच्च तरंग दैर्घ्य क्षेत्र की ओर ~ 10 - 60 नैनोमीटर का उल्लेखनीय रेड-शिफ्ट दिखाता है, और विकिरण के बाद प्रतिबिंब छवियों में महत्वपूर्ण रंग विरोधाभास स्पष्ट है। ये प्रयोगात्मक परिणाम आयन प्रसार के साथ अच्छी तरह से तुलना करते हैं और स्थानांतरण मैट्रिक्स विधि (टीएमएम) सिमुलेशन के साथ अच्छे समझौते में हैं। रमन और एक्स-रे फोटोइलेक्ट्रॉन स्पेक्ट्रोस्कोपी (एक्सपीएस) के माध्यम से पीएसआई माइक्रोकैविटी सतह क्षेत्रों का पर्याप्त संशोधन देखा जाता है। ऑप्टिकल विशेषताओं की ट्यूनेबिलिटी सिलिकॉन-सिलिकॉन और सिलिकॉन-आक्सीजन संरचनात्मक बंधों में सतह संशोधन के कारण उत्पन्न होती है और इस प्रकार आयनों द्वारा प्रेरित माइक्रोकैविटी के व्यक्तिगत पीएसआई परतों के अपवर्तक सूचकांक में परिवर्तन होता है। विश्लेषणात्मक सिमुलेशन और कैविटी मॉडलिंग के साथ ये प्रयोगात्मक परिणाम, निर्णायक रूप से कैविटी ट्यूनेबिलिटी की प्राप्ति और माइक्रोकैविटी के भीतर ऑप्टिकल क्षेत्र तीव्रता और फोटॉन कनफाइनमेंट में पर्याप्त संशोधन का समर्थन करते हैं। परिणाम बताते हैं कि आयन विकिरण अत्यधिक स्थिर, व्यापक तौर पर ऑप्टिकल रूप से असमर्थ और डिजाइनर-उपयुक्त फोटोनिक संरचनाओं का उत्पादन करने के लिए एक प्रभावी और व्यावहारिक तरीका हो सकता है। इन अद्वितीय आयन-पदार्थ ढांचे के ये व्यापक अध्ययन मूलभूत विशेषताओं के साथ-साथ उन्नत और नवीन ऑप्टोइलेक्ट्रॉनिक अनुप्रयोगों का पता लगाने के लिए नई संभावनाएं लाते हैं।

Table of Contents

Content	Page No.
Certificate	i
Acknowledgements	ii
Abstract	v
List of Figures	xiv
List of Tables	xxiv
Abbreviations and Symbols	xxvi
CHAPTER 1:	1-18
Introduction	
1.1. Introduction	1
1.2. Basics of Semiconductors	2
1.2.1. Optical Phenomena in Semiconductors	4
1.3. Photonics and Photonic Structures	6
1.4. Porous Silicon (PSi): A Multifunctional Material	8
1.5. Porous Silicon-based Photonic Structures	10
1.6. Porous Silicon-based Optical Microcavity	10
1.7. Introduction to Ion-Matter Interaction and Ion Irradiation/implantation Techniques	12
1.8. Motivations	16
1.9. Objective/Aims of the Thesis	17
CHAPTER 2:	19-51
Experimental Techniques	
2.1. Introduction	19
2.2. Fabrication Methodologies	20
2.2.1. Electrochemical Etching/Deposition	21
2.2.1.1. Working Electrode (WE)	22
2.2.1.2. Reference Electrode (RE)	22
2.2.1.3. Counter Electrode (CE)	23
2.3. Characterization Techniques	24

2.3.1. Structural Characterizations	24
2.3.1.1. Scanning Electron Microscopy (SEM)	25
2.3.1.2. Field Emission Scanning Electron Microscopy (FE-SEM)	26
2.3.1.3. Raman Spectroscopy	28
2.3.1.4. X-ray Photoelectron Spectroscopy (XPS)	30
2.3.1.5. Fourier-Transform Infrared Spectroscopy (FTIR)	33
2.3.2. Optical Characterizations	35
2.3.2.1. Reflection Spectroscopy	35
2.3.2.2. Angle-resolved Reflection Spectroscopy	37
2.3.2.3. Photoluminescence (PL) Spectroscopy	38
2.3.2.4. Spectrofluorometer for Photoluminescence	39
2.3.2.5. UV-Visible Reflectance, Absorption, and Transmission Spectrophotometry	40
2.3.2.6. Reflectance Measurements using Confocal Microscopy	41
2.4. Ion Accelerators	44
2.4.1. Table-Top Ion Accelerators	44
2.4.1.1. 30 kV Tabletop Ion accelerator	45
2.4.1.2. 60 kV Tabletop Ion accelerator	47
2.4.2. Low Energy Ion Beam Facility (LEIBF)	48
CHAPTER 3:	52-86
Fabrication and Characterization of Photonic Structures	
3.1. Introduction	52
3.2. Experimental Details	53
3.3. Germination of Porous Silicon	56
3.3.1. Lehmann and Gösele Mechanism of Chemical Dissolution	56
3.3.2. Porous Silicon Formation	58
3.4. Structural and Optical Characterization of Porous Silicon	59
3.4.1. Photoluminescence (PL) Spectra and Images of Porous Silicon	60
3.4.2. Scanning Electron Microscopy (SEM) Images of Porous Silicon	63
3.5. Reflection Spectra Analysis of Porous Silicon	63
3.6. Single-layered Porous Silicon Fabrication and Optimization of Fabrication Etching Parameters	64

3.7.	Transfer Matrix Model (TMM)	68
3.8.	Reflecting Spectra Simulation of Single-layered Porous Silicon	70
3.9.	Design and Fabrication of Multi-layered Porous Silicon	73
3.9.1.	Distributed Bragg Reflectors (DBRs)	73
3.9.1.1.	Fabrication of Distributed Bragg reflectors (DBRs)	74
3.9.2.	Optical Microcavities (MCs)	77
3.9.2.1.	Fabrication of Optical Microcavities	78
3.10.	TRIM/SRIM Simulations	85
3.11.	Conclusions	86
CHAPTER 4:		87-111
Cavity Resonance Tunability of Porous Silicon Microcavities by Argon (Ar⁺) Ion Irradiation		
4.1.	Brief Introduction	87
4.2.	Methodologies and Experimental Details	88
4.2.1.	PSi Microcavities Fabrication	88
4.2.2.	Low Energy Ar ⁺ Ion Irradiation of PSi Microcavities	89
4.3.	Optical Characterization	90
4.3.1.	Reflection Spectral Properties	90
4.3.2.	Field Emission Scanning Electron Microscope (FE-SEM) Studies	92
4.3.3.	Angle-Tuning Reflection Spectral Measurements	93
4.3.4.	Spatial Reflection Spectral Mapping Studies	95
4.3.5.	Raman Spectroscopic Studies	97
4.3.6.	FTIR Spectroscopic Studies	100
4.4.	TMM Simulations and Experimental Correlation Studies	102
4.5.	Transfer of Ions in Matter (TRIM) Depth Profile Simulations	107
4.6.	Discussion	109
4.7.	Conclusion	111
CHAPTER 5:		112-136
Tunable Characteristics of Porous Silicon Optical Microcavities by Energetic Nitrogen (N) Ion Beam Interactions		

5.1.	Introduction	112
5.2.	Methodologies	113
5.2.1.	Multi-layered PSi Fabrication	113
5.2.2.	N Ion Beam Irradiation on PSi Microcavities	114
5.3.	Optical Characterization	115
5.3.1.	Reflection Spectral Studies	115
5.3.2.	Studies of Spatial-Spectral Reflection Scan Mapping	117
5.3.3.	Raman Spectroscopic Studies	120
5.4.	Structural Characterization	123
5.4.1.	High-resolution FE-SEM Studies	123
5.4.2.	X-ray Photoelectron Spectroscopic (XPS) Studies	124
5.4.3.	FTIR Spectroscopic Studies	126
5.5.	TMM Simulations and Correlation with Experimental Results	128
5.6.	Study of TRIM/SRIM Simulated Penetration Depths	132
5.7.	Discussion	134
5.8.	Conclusion	135

CHAPTER 6: 137-155

Tuning the Optical Properties of Porous Silicon-based Microcavities by Energetic Oxygen Ion Beam for Optoelectronic Applications

6.1.	Introduction	137
6.2.	Structure of PSi Microcavities	139
6.3.	Study of Oxygen Ion beam Irradiation on PSi Microcavities	140
6.4.	Optical Properties of Irradiated PSi Microcavities	140
6.4.1.	Reflection Spectral Studies of High energy (1 MeV) O Ion Irradiation on PSi Microcavities	141
6.4.2.	Reflection Spectral Studies of Low energy (20 keV) O Ion Irradiation on PSi Microcavities	143
6.5.	Study of TMM Simulations and Correlation with the Experimental Results	145
6.6.	Study of TRIM/SRIM Simulated Ion Propagation in PSi Microcavities	150
6.7.	Discussion	154
6.8.	Conclusion	155

CHAPTER 7:**156-175****Photonic Cavity Mode Tuning in Porous Silicon-based Microcavities by He⁺ and H⁺ Ion Irradiation**

7.1.	Brief Introduction	156
7.2.	Low-energy Ion Beam Irradiation Process	158
7.3.	Optical Characterization	158
7.3.1.	Study of Reflection Spectra of Low Energy He ⁺ Ion Irradiated PSi Microcavities	158
7.3.2.	Study of Reflection Spectra of Low Energy H ⁺ Ion Irradiated PSi Microcavities	161
7.3.3.	Study of Reflection Spectra of Unirradiated and Irradiated (He ⁺ and H ⁺) PSi Microcavities	163
7.4.	Study of TRIM/SRIM simulated Ion Propagation in PSi Microcavities	168
7.5.	Discussion about Ion Irradiation/Implantation Effects on PSi Microcavities	172
7.6.	Conclusion	175

CHAPTER 8:**176-181****Conclusions and Future Scope of the Work**

8.1.	Broad Conclusions	176
8.2.	Future Scope of the Studies	180

References **182-193****A List of Publications Resulted from the Thesis Work** **194-195****Papers Published in JCR Journals** **194****International and National Conferences Presentations and Workshop Precipitations** **195****Author's Bibliography** **196-197**

List of Figures

Chapter 1: Introduction

Figure 1.1: (a) Diagram showing the energy-momentum ($E-k$) level for (a) direct bandgap and (b) indirect bandgap, shown schematically.

Figure 1.2: (a) The dispersion relationship of frequency (ω) with wave vector (k) inside a photonic crystal. The schematic illustration of photonic crystals (b) DBR and (c) Microcavity. The yellow curve represents the electric field (E_{field}) propagation within the microcavity which is optimum at the central layer.

Figure 1.3: An illustrative picture of ion-matter interaction processes occurring at different energies (1 keV to 100 MeV) and various ion-fluences (1×10^{13} to 1×10^{17} ions/cm²).

Figure 1.4: A schematic representation to reveal the process of ion beam irradiation at PSi layers

Chapter 2: Experimental Techniques

Figure 2.1: (a) Schematic diagram of internal circuit assembly of potentiostat/galvanostat.

Figure 2.2: (a) Camera picture and (b) schematic design of scanning electron microscope (SEM) Hitachi TM300 SEM.

Figure 2.3: (a) and (b) shows the photograph of the FE-SEM instrument of model name JSM-7800FPrime [179].

Figure 2.4: In Raman Spectroscopy, an energy level diagram depicting the various energy transitions is shown.

Figure 2.5: (a) Renishaw inVia Raman Microscope Photograph (b) schematic of Renishaw inVia Raman Microscope [180].

Figure 2.6: X-ray photoelectron spectroscopy schematic diagram.

Figure 2.7: The X-ray photoelectron spectroscopy (XPS) laboratory at the Maxwell Centre, University of Cambridge, United Kingdom [181].

Figure 2.8: Photograph of FTIR NICOLET - IS-50 instrument, (a) Attenuated Total Reflectance (ATR) mode FTIR and (b) Attenuated Total Absorption (ATA) mode FTIR [179].

Figure 2.9: Schematic diagram of FTIR spectroscopy.

Figure 2.10: Schematic diagram of homemade reflection setup.

Figure 2.11: Schematic of reflection setup.

Figure 2.12: (a) Picture of Ocean Optics RSS-VA variable-angle reflection system and (b) cross-sectional view of the same RSS-VA setup for working description [182].

Figure 2.13: Schematic setup of Photoluminescence measurement.

Figure 2.14: Photographs of (a) spectrofluorometer (Shimadzu RFPC 5301PC) and its (b) sample container construction.

Figure 2.15: (a) The UV-Vis spectrophotometer (Shimadzu UV3600) in action. (b) The reflectance and transmittance measurements are shown schematically.

Figure 2.16: Schematic representation of UV-Vis spectrometer.

Figure 2.17: (a) Real picture of modified Olympus BX-51 microscope with (b) the schematic representation of the same modified optical microscope

Figure 2.18: The photograph of a 30 kV Tabletop Accelerator [185].

Figure 2.19: The photograph of a 60 kV Tabletop Accelerator [185].

Figure 2.20: Illustrations of the Low Energy Ion Beam Facility (LEIBF) ion implanter facility, which includes (a) an ion source (ECR) at the high voltage stages, (b) an accelerating column for ion acceleration, (c) a dipole magnet for switching ion beams to the beamline, and (d) a target chamber at the end of beamline setup [185].

Chapter 3: Fabrication and Characterization of Photonic Structures

Figure 3.1: Schematic diagram of the electrochemical etching (or anodization) process.

Figure 3.2: Sequences of Chemical Dissolution mechanism involved in the electrochemical etching process.

Figure 3.3: The photoluminescence (PL) spectra of the (a) *n*-type porous silicon and (a) *p*-type PSi.

Figure 3.4: (a-d) The red and (e-g) green ($J=50 \text{ mA/cm}^2$ and $t=1800 \text{ sec}$) photoluminescence (PL) images of the *n*-type PSi samples were prepared at different parameters i.e., $J=25 \text{ mA/cm}^2$ and $t=1500 \text{ sec}$ for (a-d) and $J=50 \text{ mA/cm}^2$ and $t=1800 \text{ sec}$ for (e-g) respectively.

Figure 3.5: The bright field (BF) microscope images of the *n*-type PSi prepared at various etching parameters i.e., $J=50 \text{ mA/cm}^2$ and $t=3000 \text{ sec}$ for (a-c) and $J=25 \text{ mA/cm}^2$ and $t=1500 \text{ sec}$ for (d-f) respectively.

Figure 3.6: (a-b) shows the dark field images of the *n*-type PSi, and (c-d) displays the bright

field microscope images of the *p-type* PSi, i.e., $J=50 \text{ mA/cm}^2$ and $t=1800 \text{ sec}$ for (a-b) and $J=25 \text{ mA/cm}^2$ and $t=1500 \text{ sec}$ for (c-d) respectively.

Figure 3.7: SEM images of *n-type* PSi prepared at $J=50 \text{ mA/cm}^2$ and $t=1800 \text{ sec}$ is shown in (a-b), while *p-type* PSi produced at $J=25 \text{ mA/cm}^2$ and $t=1500 \text{ sec}$ is shown in (c) respectively.

Figure 3.8: (a) Schematic diagram of the PSi film having an effective refractive index (n_{eff}) which is related to the correlation of the porosity of c-Si (n_{Si}) with the air (n_{air}). (b) shows the interference procedure of incident light after reflection at interfaces of air-PSi-Si.

Figure 3.9: The reflection spectra for samples prepared at a range of anodization current densities (J) for the same time of 30 sec on an area of 0.65 cm^2 of the *p+-type* Si wafer.

Figure 3.10: The reflection plots for ten samples prepared at the two different anodization current densities (a) $J=77 \text{ mA/cm}^2$ and (b) $J=15 \text{ mA/cm}^2$ for various time durations (1.2 sec to 10 sec) on an area of 0.65 cm^2 of the *p+-type* Si wafer. The typical color pictures of these PSi samples are also shown alongside spectra.

Figure 3.11: Curve fitting plots of thickness and time for samples prepared at different optimum anodization current densities ($J=15 \text{ mA/cm}^2$ and 77 mA/cm^2) for various time durations on an area of 0.65 cm^2 of the *p+-type* Si wafer.

Figure 3.12: shows a plane wave strikes at a periodic arrangement of layers with various refractive indexes. E and H represent the electric and magnetic fields between two neighboring layers at the interfaces.

Figure 3.13: The experimental reflection plots of prepared samples at anodization current density ($J=77 \text{ mA/cm}^2$) for various time duration (a) $t=1.2 \text{ s}$, (b) 3 s, (c) $t=5 \text{ s}$, and (d) 10 s fitted with their best fittings.

Figure 3.14: The experimental reflection plots of prepared samples at anodization current density ($J=15 \text{ mA/cm}^2$) for various time duration (a) $t=1.2 \text{ s}$, (b) 3 s, (c) $t=5 \text{ s}$, and (d) 10 s fitted with their best fittings.

Figure 3.15: Schematic representation of 10 layered DBR structures.

Figure 3.16: The reflection plots for ten layered DBR prepared from electrochemical etching in which the wavelength is centered at (a) $\lambda=885 \text{ nm}$ (b) and $\lambda=740 \text{ nm}$.

Figure 3.17: The experimental reflection plots of 10 layered DBRs are simulated with the best TMM fittings of center wavelength at (a) $\lambda=885 \text{ nm}$ (b) and $\lambda=740 \text{ nm}$. Transverse Electric (TE) field intensity distribution of DBR in which band gap is centered at the wavelengths (c) 885 nm and (d) 740 nm.

Figure 3.18: The typical color pictures of the DBRs at the various fabrication parameters are mentioned in Table 3.2.

Figure 3.19: Schematic representation of microcavity structure that contains a centre layer between two DBRs.

Figure 3.20: (a) The experimental reflection plots of 17 layered microcavities have cavity wavelengths at $\lambda=725$ nm (MC1) and $\lambda=615$ nm (MC2). The typical real images of the same microcavities are shown alongside displays of the respective reflection color of the cavity mode.

Figure 3.21: (a) The reflectance spectra of 17 layered microcavities have cavity wavelength at $\lambda=663$ nm. (b) The reflection spectra of the same microcavity are finest fitted with simulated reflection spectra. Same as Fig. 3.21 (a), the experimental reflectance spectra for the different cavity wavelengths at (c) $\lambda=624$ nm and (e) $\lambda=708$ nm and their respective reflection spectra of these same microcavities are finest fitted with simulated reflection spectra in (d) and (f) respectively.

Figure 3.22: (a) The experimental reflection plots of 17 layered microcavities have different cavity wavelengths at (a) $\lambda=517$ nm, (b) $\lambda=605$ nm, (c) $\lambda=672$ nm, (d) $\lambda=713$ nm, and (e) $\lambda=740$ nm prepared at various parameters.

Figure 3.22: The PSi optical microcavity samples demonstrated charming colors that were prepared with different optimized etching parameters are listed in Table 3.3.

Chapter 4: Cavity Resonance Tunability of Porous Silicon Microcavities by Argon (Ar^+) Ion Irradiation

Figure 4.1: Schematic diagram of the PSi-based microcavity, consisted of two DBRs divided by a high porous spacer layer (n_s) having $\lambda/2$ optical thickness, where n_L , d_L , and n_H , d_H are refractive index and thickness of high and low porous layers, respectively.

Figure 4.2: Experimental reflection spectra of both PSi microcavities samples (MC2 and MC3) before and after Ar^+ ion irradiation. Dotted lines are the reflection spectra of the respective unirradiated microcavities. Conventional reflection images of both (MC2 and MC3) samples show the respective color change in the irradiated region before and after Ar^+ ion irradiation.

Figure 4.3: Experimental reflection spectra of two PSi microcavities samples before and after Ar^+ ions irradiation with higher ion fluences ($5\text{E}15$ and $1\text{E}16$ ions/ cm^2). Note that there is considerable redshift in their cavity peaks. The dotted lines are the reflection spectra of the respective unirradiated microcavities. Conventional reflection images of both irradiated microcavities ($5\text{E}15$ and $1\text{E}16$ ions/ cm^2) show respective color changes from yellow to dark red before and after Ar^+ ion irradiation.

Figure 4.4: Shows the cross-sectional high resolution FESEM image of the (a) fabricated unirradiated (b) Ar^+ irradiated ($1\text{E}14$ ions/ cm^2) and (c) Ar^+ irradiated ($1\text{E}15$ ions/ cm^2) PSi microcavities respectively with the schematic representation of layers.

Figure 4.5: Experimental angle-resolved reflection spectra of the (a) unirradiated microcavity (MC1) (b) Ar⁺ irradiated microcavity of fluence 1E14 ions/cm² (MC2) and (c) Ar⁺ irradiated microcavity of fluence 1E15 ions/cm² (MC3) at distinct angles.

Figure 4.6: Experimental angle-resolved reflection spectra plot of the (a) resonant cavity peaks (P_{cav}) of both Ar⁺ irradiated microcavities (MC2 and MC3) and unirradiated microcavity (MC1) (b) same as Figure 4.6 (a), but in cavity resonant peaks in energy scale. The dotted lines represent the fitting using equation 4.1.

Figure 4.7: (a) Reflection intensity map of MC3 at $\lambda=576$ nm where the dark red region represents the Ar⁺ irradiated portion and (b) the conventional image of the same microcavity. (c) Line scan of reflection spectral map. A shift of the photonic cavity mode (region 560-580 nm) for the unirradiated and Ar⁺ irradiated region is marked with blue color square lines.

Figure 4.8: (a) The scanning reflection spectral map images of Ar⁺ irradiated microcavity (1E15 ions/cm²) in two different directions (say X and Y directions) with respect to the irradiated region of the sample showing the red-shift of photonic cavity mode in the Ar⁺ irradiated region of MC3. Note that dark blue arrows indicate the narrow yellow line, which represents the photonic cavity mode of MC3.

Figure 4.9: Reflection spectral line scan profile images of MC3, which represents (a) highly Ar⁺ irradiated region of the MC3 at $\lambda=584$ nm and (b) unirradiated area of the same sample at $\lambda=570$ nm, which confirms the actual conventional image.

Figure 4.10: Raman spectra of (a) pristine silicon (b) unirradiated microcavity (MC1) (c) Ar⁺ irradiated microcavity (MC2) of fluence 1E14 ions/cm² and (d) Ar⁺ irradiated microcavity (MC3) having fluence 1E15 ions/cm² with excitation 532 nm laser.

Figure 4.11: Raman spectra of pristine silicon (Si_p), unirradiated microcavity (MC1) and Ar⁺ irradiated microcavities (MC2 and MC3) at different spectral ranges as (a) from 200 cm⁻¹ to 450 cm⁻¹, and (b) from 450 cm⁻¹ to 650 cm⁻¹ with 785 nm laser.

Figure 4.12: FTIR absorbance spectra of pristine silicon (Si_p), unirradiated microcavity (MC1) and Ar⁺ irradiated microcavities (MC2 and MC3) at various spectral ranges as (a) from 450 cm⁻¹ to 800 cm⁻¹, (b) from 800 cm⁻¹ to 1200 cm⁻¹ and (c) from 2600 cm⁻¹ to 3100 cm⁻¹.

Figure 4.13: (a) Experimental and TMM simulated reflection spectra of MC1 at angle 15° almost overlap each other and offer fitting parameters. (b) TMM simulated optical field (TE) intensity localized in the unirradiated microcavity (MC1) at 15° (c) spatial map of optical intensity (reflectivity) vs. wavelength at angle 15° (d) spatial map of optical intensity (reflectivity) vs. angle at $\lambda=562$ nm (e) experimental angle-dependent reflection images of MC1.

Figure 4.14: (a) Shows the experimental and TMM simulated reflection spectra of MC2 at angle 15° (b) TMM simulations of optical (reflectivity) field (TE) intensity localized in the Ar⁺ irradiated microcavity (1E14 ions/cm²) (MC2) at angle 15° for $\lambda= 570$ nm having energy 2.17 eV (c) spatial map of optical (reflectivity) intensity vs. wavelength at angle 15° and (d) spatial map of optical (reflectivity)

intensity vs. angle at $\lambda=570$ nm and (e) experimental angle-dependent reflection images of MC2. The open white circles represent the experimental resonance energy cavity peak positions of reflection spectra dips, and the solid line is from the corresponding simulated resonance cavity mode of the same sample.

Figure 4.15: (a) Shows the experimental and TMM simulated reflection spectra of MC3 at angle 15° (b) TMM simulations of optical (reflectivity) field (TE) intensity localized in the Ar^+ irradiated microcavity ($1\text{E}15$ ions/ cm^2) (MC3) at angle 15° for $\lambda=576$ nm having energy 2.15 eV (c) spatial map of optical (reflectivity) intensity vs. wavelength at angle 15° and (d) spatial map of optical (reflectivity) intensity vs. angle at $\lambda=576$ nm (e) experimental angle-dependent reflection images of MC3. The open white circles represent the experimental resonance energy cavity peak positions of reflection spectra dips, and the solid line is from the corresponding simulated resonance cavity mode of the same sample.

Figure 4.16: Shows the TRIM simulated penetration depth profile ranges of Ar^+ ions (a) in pristine Si at 15 keV energy and proceeds up to the first two layers of PSi DBR having eight layers at energies (b) 5 keV (c) 10 keV and (d) 15 keV respectively with a schematic representation of DBR with eight layers.

Figure 4.17: The cross-sectional high-resolution FESEM image of the (a) fabricated unirradiated (MC1) PSi microcavity with the schematic representation, (b) and (c) shows the TRIM simulations considering 10000 Ar^+ ions of 10 keV, in the top first two PSi layers.

Chapter 5: Tunable Characteristics of Porous Silicon Optical Microcavities by Energetic Nitrogen (N) Ion Beams Interactions

Figure 5.1: The 3-D schematic representation of the PSi-based microcavity with a spacer layer (n_s , d_s) of high porosity, $\lambda/2$ optical thickness between two DBRs (where n_H , d_H , and n_L , d_L are refractive index and thickness of low and high porous layers, respectively. $\lambda=690$ nm).

Figure 5.2: Experimental reflectance spectra of both PSi microcavities (MC2 and MC3) before and after N ion beam interactions. A dotted line shows the reflectance spectra of respective unirradiated microcavity (MC1). The color changes before and after N ion beam interaction is shown in the conventional reflection images of both (MC2 and MC3) samples.

Figure 5.3: (a) Line scan of reflection spectral horizontal map of MC2 (half the portion is ion-implanted with 200 keV, fluence $1\text{E}15$ ions/ cm^2) to differentiate the irradiated and unirradiated regions. The photonic cavity mode shows a redshift (region 690-760 nm) from unimplanted to N ion-implanted regions marked with a blue arrow. This is also confirmed by the respective (b) redshift of the reflectance spectrum of MC2 (highlighted with colored squares lines). The conventional image of the MC2, where the dark black region represents the N ion implanted portion.

Figure 5.4: Reflection spectral mapping images represent the implanted (I) and unimplanted (II) regions of the same microcavity at corresponding wavelengths (a) $\lambda_c=750$ nm (irradiated) and (b) $\lambda_c=700$ nm (unirradiated) respectively.

Figure 5.5: (a) Reflection intensity map of MC2 where red region (I) and orange region (II) represents the N irradiated and unirradiated portion, respectively. (b) The conventional image of the same microcavity confirms the above spatial map image by indicating the same regions. Line scan reflection spectral map of N irradiated microcavity (200 keV, $1E15$ ions/cm²) in two different directions, say (c) right (X) and (d) left (Y), with respect to the irradiated part of the sample. A redshift of the photonic cavity mode (region 600-750 nm) for unirradiated and N irradiated region are marked with blue color square lines. Note that arrows are indicating towards the narrow yellow line, which represents the photonic cavity mode of MC2.

Figure 5.6: Raman spectra of (a) pristine silicon (Si_p); (b) unirradiated (MC1), (c) MC2 and (d) MC3 microcavities. The excitation wavelength used is 532 nm.

Figure 5.7: The high resolution FESEM cross-sectional micrograph of the (a) fabricated unirradiated (MC1) (b) N implanted (200 keV, fluence- $1E15$ ions/cm²) (MC2) and (c) N irradiated (1 MeV, fluence- $1E15$ ions/cm²) (MC3) PSi microcavities respectively along with the schematic representation of PSi layers.

Figure 5.8: (a) Si-2p core-level XPS spectra of MC1, MC2, and MC3. The deconvoluted Gaussian peaks marked as 1,2,3,4, and 5 are attributed to Si 2p_{1/2}, Si 2p_{3/2}, Si₃N₄, SiON_x, and SiO₂. XPS spectra of (b) N-1s and (c) O-1s core levels.

Figure 5.9: FTIR absorbance spectra of pristine Si (Si_p), unirradiated (MC1), MC2 and MC3 microcavities at various spectral ranges as (a) from 800 cm⁻¹ to 1200 cm⁻¹, and (b) from 2200 cm⁻¹ to 3600 cm⁻¹.

Figure 5.10: (a) Experimental reflection spectra of unirradiated microcavity (MC1) simulated with TMM spectra, cavity peaks nearly coincide and evolving fitting parameters. (b) TMM simulated optical field (TE) intensity contained in the MC1 at 4°. The spatial maps of optical (reflectivity) intensity (c) vs. angle at $\lambda_c=690$ nm (d) and vs. wavelength at incident angle 4° are exhibited. (e) The TMM simulated reflection spectral map implies an angle-dependent cavity mode variation of MC1 (highlighted in the dotted ellipse).

Figure 5.11: (a) Shows the experimental and TMM simulated reflection spectra of MC2 at angle 4° (b) TMM simulations of optical (reflectivity) field (TE) intensity localized in the N implanted microcavity (200 keV, $1E15$ ions/cm²) (MC2) at incident angle 4° for $\lambda=750$ nm having energy 1.65 eV (c) spatial map of optical (reflectivity) intensity vs. angle at $\lambda_c=750$ nm and (d) spatial map of optical (reflectivity) intensity vs. wavelength at incident angle 4° and (e) experimental angle-dependent reflection images of MC2. The solid line is from the corresponding simulated resonance cavity mode of the same sample.

Figure 5.12: (a) Shows the experimental and TMM simulated reflection spectra of MC3 at angle 4° (b) TMM simulations of optical (reflectivity) field (TE) intensity

localized in the N irradiated microcavity (1 MeV, $1\text{E}15\text{ ions/cm}^2$) (MC3) at incident angle 4° for $\lambda=722\text{ nm}$ having energy 1.72 eV (c) spatial map of optical (reflectivity) intensity vs. angle at $\lambda_c=722\text{ nm}$ (d) spatial map of optical (reflectivity) intensity vs. wavelength at incident angle 4° and (e) experimental angle-dependent reflection images of MC3. The solid line is from the corresponding simulated resonance cavity mode of the same sample.

Figure 5.13: (a) The TRIM simulated ion-range distribution of N ions in pristine Si and PSi layers of microcavity structure at energies (a) 200 keV and (b) 1 MeV layers, respectively, with the schematic representation of optical microcavity of 17 layers having a spacer layer embedded between two DBRs. Note that lower energy N ions (200 keV) penetrated all PSi layers of microcavity while higher energy ions (1 MeV) settled down in the Si substrate beyond the PSi layers. Figures (c) and (d) show TRIM simulated ion-penetration depth profile of N ions, same as Figures 5.13 (a) and (b) but in X-Y axes scale, respectively.

Chapter 6: Tuning the Optical Properties of Porous Silicon-based Microcavities by Energetic Oxygen Ion Beams for Optoelectronic Applications

Figure 6.1: 3-D schematic diagram of PSi-based optical microcavity consisted of two DBRs divided by a high porous spacer layer (n_s, d_s), where n_L, d_L , and n_H, d_H are refractive index and depth of high and low porous layers, respectively.

Figure 6.2: The experimental reflectance spectra of both unirradiated microcavities (UC1, UC2) and next to 1 MeV O ion irradiated at fluences (a) $1\text{E}15$ (MCM15) and (b) $1\text{E}16$ (MCM16) ions/cm². Post-ion interaction, the conventional reflection images of both samples show substantial color change from unirradiated to irradiated regions.

Figure 6.3: The experimental reflectance spectra of 20 keV energetic O ion irradiated microcavities with ion-fluences (A) $1\text{E}13$ (MCK13), (B) $1\text{E}15$ (MCK15) and (C) $1\text{E}16$ (MCK16) ions/cm² along with their respective (dotted lines) reflectance spectra of unirradiated microcavities (UC3, UC4, UC5). The colored elliptical orbits indicate their concerned cavity-peak shift. The conventional reflection pictures of each sample (a-c) show substantial color change from (i) unirradiated to (ii) irradiated portions of these images.

Figure 6.4. (a) Experimental and TMM simulated reflection spectra of MCM15 almost coincide with each other and proposed fitting parameters. (b) TMM simulated optical (reflectivity) field (TE) intensity localized in MCM15 at $\lambda_c=674\text{ nm}$, (c) spatial maps of optical intensity (reflectivity) vs. angle at $\lambda_c=674\text{ nm}$ (d) and vs. wavelength, and (e) angle-dependent reflection layout of MCM15.

Figure 6.5: (a) Shows the experimental and TMM simulated reflection spectra of unirradiated (UC1) microcavity (b) TMM simulated optical (reflectivity) field (TE) intensity localized in UC1 at $\lambda_c=654\text{ nm}$ (c) spatial maps of optical (reflectivity) intensity vs. angle at $\lambda_c=654\text{ nm}$ and (d) intensity vs. wavelength (e) experimental angle-dependent reflection images of UC1. The solid line forms the corresponding simulated resonance cavity mode of the same sample.

Figure 6.6: (a) Shows the experimental and TMM simulated reflection spectra of unirradiated (UC2) microcavity (b) TMM simulated optical (reflectivity) field (TE) intensity localized in UC2 at $\lambda_c = 645$ nm (c) spatial maps of optical (reflectivity) intensity vs. angle at $\lambda_c = 645$ nm and (d) intensity vs. wavelength (e) experimental angle-dependent reflection images of UC2. The solid line forms the corresponding simulated resonance cavity mode of the same sample.

Figure 6.7: (a) Shows the experimental and TMM simulated reflection spectra of 1 MeV O irradiated (MCM16) microcavity of fluence $1E16$ ions/cm² (b) TMM simulated optical (reflectivity) field (TE) intensity localized in MCM16 at $\lambda_c = 665$ nm (c) spatial maps of optical (reflectivity) intensity vs. angle at $\lambda_c = 665$ nm and (d) intensity vs. wavelength (e) experimental angle-dependent reflection images of MCM16. The solid line forms the corresponding simulated resonance cavity mode of the same sample.

Figure 6.8: (a) Experimental and TMM simulated reflection spectra of 20 keV O irradiated at a fluence of $1E13$ ions/cm² (MCK13) almost coincide with each other and fitting resolved optical parameters. (b) TMM simulated optical (reflectivity) field (TE) intensity localized in MCK13 at $\lambda_c = 545$ nm, (c) spatial maps of optical intensity (reflectivity) vs. angle at $\lambda_c = 545$ nm (d) and vs. wavelength, and (e) angle-dependent reflection layout of MCK13.

Figure 6.9: (a) Shows the experimental and TMM simulated reflection spectra of 20 keV O irradiated (MCK15) microcavity of fluence $1E15$ ions/cm² (b) TMM simulated optical (reflectivity) field (TE) intensity localized in MCK15 at $\lambda_c = 540$ nm (c) spatial maps of optical (reflectivity) intensity vs. angle at $\lambda_c = 540$ nm and (d) intensity vs. wavelength (e) angle-dependent layout of MCK15. The solid line forms the corresponding simulated resonance cavity mode of the same sample.

Figure 6.10: The TRIM simulated pictures considering 10000 O ions of 1 MeV, (a) and (b) show the projected range and ion propagations in PSi layers of microcavity and compared with bare Si.

Figure 6.11: The TRIM simulated photographs of 10000 O ions having 20 keV, (a) and (b) show projected depth and ion configurations in PSi layers of microcavity and assimilated with bare Si.

Chapter 7: Photonic Cavity Mode Tuning in Porous Silicon-Based Microcavities by He⁺ and H⁺ Ion Irradiation

Figure 7.1: (a) The experimental reflectance spectra of 35keV He⁺ ion irradiated microcavities at ion-doses 1×10^{15} (MCHe1), 5×10^{15} (MCHe2), and 1×10^{16} (MCHe3) ions/cm². The dotted lines represent their unirradiated microcavities (UC1, UC2, UC3) reflectance spectra. Note that after the post-ion treatment, there is a shift in cavity peaks. (b) The typical reflection images of samples indicate substantial color change after ion interaction (indicated by arrows).

Figure 7.2: (a) The experimental reflectance spectra of H⁺ irradiated microcavities of 35 keV

energy with ion-doses 1×10^{15} (MCH1), 5×10^{15} (MCH2), and 1×10^{16} (MCH3) ions/cm² inclusive of their dotted lines reflectance spectra of unirradiated microcavities (UC4, UC5, UC6). The colored dotted ellipses pointed out their concerned cavity peaks. (b) The typical reflection pictures of each sample feature considerable color change due to ion interaction.

Figure 7.3: Show the experimental reflection spectra of unirradiated PSi microcavities (a) (UC1-UC3) and (b) (UC4-UC6). These spectra are compared with corresponding TMM simulated reflection spectra. Details are listed in Tables 7.1 and 7.2.

Figure 7.4: Show the experimental reflection spectra of He⁺ ion irradiated microcavities (a) (MCHe1-MCHe3) and (b) H⁺ ion irradiated microcavities (MCH1-MCH3) are compared with their corresponding TMM simulated spectra. Details are listed in Tables 7.1 and 7.2.

Figure 7.5: (a) TMM simulated optical (reflectivity) field (TE) intensity localized in unirradiated microcavity (UC4) at $\lambda_c = 556$ nm (b) spatial maps of optical (reflectivity) intensity vs. angle at $\lambda_c = 556$ nm and (c) intensity vs. wavelength. Similar simulated maps (d-f) and (g-i) are shown for the other two unirradiated microcavities (UC5 and UC6) with their cavity peak wavelengths at $\lambda_c = 548$ nm and $\lambda_c = 550$ nm, respectively.

Figure 7.6: (a) TMM simulated optical (reflectivity) field (TE) intensity localized in of 35keV H⁺ irradiated at fluence of 1×10^{15} ions/cm² (MCH1) at $\lambda_c = 573$ nm (b) spatial maps of optical (reflectivity) intensity vs. angle at $\lambda_c = 573$ nm and (c) intensity vs. wavelength. Similar simulated maps (d-f) and (g-i) are shown for the other two 35 keV H⁺ irradiated microcavities of ion fluences 5×10^{15} and 1×10^{16} ions/cm² (MCH2 and MCH3) with their cavity peak wavelengths at $\lambda_c = 574$ nm, and $\lambda_c = 575$ nm respectively.

Figure 7.7: : (a) TMM simulated optical (reflectivity) field (TE) intensity localized in unirradiated microcavity (UC1) at $\lambda_c = 546$ nm (b) spatial maps of optical (reflectivity) intensity vs. angle at $\lambda_c = 546$ nm and (c) intensity vs. wavelength. Similar simulated maps (d-f) and (g-i) are shown for the other two unirradiated microcavities (UC2 and UC3) with their cavity peak wavelengths at $\lambda_c = 564$ nm and $\lambda_c = 562$ nm, respectively.

Figure 7.8: (a) TMM simulated optical (reflectivity) field (TE) intensity localized in of 35keV He⁺ irradiated at fluence of 1×10^{15} ions/cm² (MCHe1) at $\lambda_c = 574$ nm (b) spatial maps of optical (reflectivity) intensity vs. angle at $\lambda_c = 574$ nm and (c) intensity vs. wavelength. Similar simulated maps (d-f) and (g-i) are shown for the other two 35 keV He⁺ irradiated microcavities of ion fluences 5×10^{15} and 1×10^{16} ions/cm² (MCH2 and MCH3) with their cavity peak wavelengths at $\lambda_c = 597$ nm, and $\lambda_c = 610$ nm respectively.

Figure 7.9: The TRIM simulated pictures of ion propagations and distributions inside PSi layers of the microcavity. (a) for He⁺ ions of 35 keV, and (b) for H⁺ ions of the same energy. Figures (c) and (d) exhibit the same as Figure 7.9 (a) and (b) but on another scale (X-Y axes), respectively. Here, 10000 ions are considered.

List of Tables

Chapter 2: Experimental Techniques

Table 2.1: List of elements with an available energy range for exposure in 30kV Tabletop accelerator [185].

Table 2.2: List of elements with an available energy range for exposure of light mass elements in 60 kV Tabletop accelerator [185].

Table 2.3: The following are some examples of commonly available positive ion beams, along with their charge states, energies, and beam currents [185]:

Chapter 3: Fabrication and Characterization of Photonic Structures

Table 3.1: The Table represents the porosity, thickness, and refractive index for all samples, which are etched with various anodization current densities and different etching times of the same etching area. The reflection spectra and their corresponding fittings are shown in Figures 3.13 and 3.14.

Table 3.2: The Table shows the fabrication specifications for different prepared DBRs with a range of beautiful colors, as seen in Figure 3.18 above.

Table 3.3: As shown in Figure 3.23 above, the Table displays the fabrication specifications for various prepared microcavities in a variety of lovely hues.

Chapter 4: Cavity Resonance Tunability of Porous Silicon Microcavities by Argon (Ar^+) Ion Irradiation

Table 4.1: Microcavity resonant cavity fitting parameters derived from equation (4.1) are listed here.

Table 4.2: The fitting parameters of unirradiated and Ar^+ ion irradiated PSi microcavities at 15° determined from TMM simulations.

Table 4.3: The ion beam parameters considering 5, 10, and 15 keV Ar^+ irradiations as determined from SRIM simulations.

Chapter 5: Tunable Characteristics of Porous Silicon Optical Microcavities by Energetic Nitrogen (N) Ion Beams Interactions

Table 5.1: The effective change in the resulting optical parameters of the PSi microcavities after their exposure to N ions of different energies.

Table 5.2: The various Si-phases and structure identification in the observed Raman bands of pristine Si, MC1, MC2, and MC3 are determined from the best curve-fitting.

Table 5.3: The resulting parameters of unirradiated and N irradiated PSi microcavities at incident angle 4° determined from TMM simulations fitting.

Table 5.4: The ion beam parameters considering different energies of N treatments as determined from SRIM simulations.

Chapter 6: Tuning the Optical Properties of Porous Silicon-based Microcavities by Energetic Oxygen Ion Beams for Optoelectronic Applications

Table 6.1: The experimentally obtained optical parameters from TMM simulations fittings demonstrate pronounced alteration due to 1 MeV O ions treatment.

Table 6.2: The experimental optical parameters obtained for microcavities after 20 keV O ion beam interactions from the TMM simulations display changes due to irradiation.

Table 6.3: The energy-dependent parameters considering 1 MeV and 20 keV O ion beams treatments as determined from SRIM simulations.

Chapter 7: Photonic Cavity Mode Tuning in Porous Silicon-Based Microcavities by He⁺ and H⁺ Ion Irradiation

Table 7.1: The experimental optical parameters obtained for microcavities after 35 keV He⁺ ion beam interactions from the TMM simulations.

Table 7.2: The experimental optical parameters obtained for microcavities after 35 keV H⁺ ion beam interactions from the TMM simulations.

Table 7.3: The energy-dependent parameters of He⁺ and H⁺ ion beams with 35 keV energy treatments as determined from SRIM simulations.

Table 7.4: The list of previous experimental optical results obtained from the TMM simulations that compared the pristine microcavities after Ar, N, and O ion beam interactions of these microcavities (Ref. 18-20).

Abbreviations and Symbols

PSi	Porous Silicon
eV/keV/MeV	Electron volt/ Kilo electron volt/Mega electron volt
nm/ μ m/cm	Nanometers/micrometers/centimeters
a.u./arb.units	Arbitrary units
n	Refractive index
t/d	Thickness/optical thickness
k	Extinction coefficient
n_{eff}	Effective refractive index
E	Energy
EM	Electromagnetic
TE	Transverse electric
DBRs	Distributed Bragg Reflectors
MCs	Microcavities
UCs	Unirradiated microcavities
TMM	Transfer matrix model
TRIM	Transfer of ions in matter
SRIM	Stopping and ranges of ions in matter
EMA	Effective medium approximation
DI	Deionized water
1-D	One-Dimensional
2-D	Two-Dimensional
3-D	Three-Dimensional
SEM	Scanning Electron Microscope
FE-SEM	Field Emission Scanning Electron Microscope
FTIR	Fourier Transform Infra-Red Spectroscopy
XPS	X-ray Photoelectron Spectroscopy
EDX	Energy Dispersive X-ray Spectroscopy

DPXPS	Depth profiling X-ray photoemission spectroscopy
ARXPS	Angle-resolved X-ray photoemission spectroscopy
UPS	Ultraviolet photoemission spectroscopy
RSS-VA	Variable-angle Reflection Sampling System
SMA	Sub Miniature version A
NIR	Near Infrared region
PL	Photoluminescence
FWHM	Full width at half maxima
Q-factor	Quality factor
P_{cav}	Cavity peaks
E_{ph}	Cavity photon energy at angle θ
E_{c}	Cavity photon energy at normal incidence (or $\theta=0^\circ$)
$S_{\text{e}}/S_{\text{n}}$	Electronic/Nuclear energy loss
BF/ DF	Bright Field/ Dark Field
SED	Secondary Electron Detector
BSED	Back Scattered Electron Detector
ND	Neutral density
CW	Continuous Wave
Si _p	Pristine silicon
c-Si	Crystalline Silicon
n-Si	Nanocrystalline Silicon
a-Si	Amorphous Silicon
TO/LO	Transverse optical/Longitudinal optical
TA/LA	Transverse acoustical/Longitudinal acoustical
UV, Vis	Ultraviolet, Visible
IR	Infrared
LEIBF	Low Energy Ion Beam Facility
PIBF	Positive Ion Beam Facility
NIIBF	Negative Ion Implanter Beam Facility

PIG	Penning Ion Generator
ATR	Attenuated Total Reflectance
ATA	Attenuated Total Absorption
NPs	Nanoparticles
NAP	Near ambient pressure
ECR	Electron Cyclotron Resonance
UHV	Ultra-high vacuum
BE	Binding energy
WE	Working Electrode
CE	Counter Electrode
RE	Reference Electrode
SCE	Saturated calomel electrode
AgCl	Silver chloride electrode
CRT	Cathode ray tube
CCD	Charge coupled device
RIE	Reactive ion etching
VLS	Vapor-liquid-solid
LED	Light emitting diode
MIVOC	Metal Ions from Volatile Organic Compounds
GBSH	Gentle Beam Super High
Ref.	References