OPTICAL STUDIES OF ION IRRADIATED SILICON BASED PHOTONIC STRUCTURES

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DEPARTMENT OF PHYSICS

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by

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Department of Physics

Submitted in fulfillment of the requirements for the degree of

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to the

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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.

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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/cm^2). 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.

सार

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 ऑप्टोइलेक्ट्रोनिक 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/सेमी^{2}) 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 नक़्क़ाशी 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 दैर्ध्य 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 ऑप्टोइलेक्ट्रोनिक अनुप्रयोगों का पता लगाने के लिए नई संभावनाएं लाते हैं।

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Figure 5.7: The high resolution FESEM cross-sectional micrograph of the (a) fabricated unirradiated (MC1) (b) N implanted (200 keV, fluence-1E15 ions/cm^2) (MC2) and (c) N irradiated (1 MeV, fluence-1E15 ions/cm^2) (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 $2p_{3/2}$, Si_3N_4, $SiON_x$, and SiO_2. 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 4º. The spatial maps of optical (reflectivity) intensity (c) vs. angle at λ_c= 690 nm (d) and vs. wavelength at incident angle 4º 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 4º (b) TMM simulations of optical (reflectivity) field (TE) intensity localized in the N implanted microcavity (200 keV, 1E15 ions/cm^2) (MC2) at incident angle 4º for λ= 750 nm having energy 1.65 eV (c) spatial map of optical (reflectivity) intensity vs. angle at λ_c= 750 nm and (d) spatial map of optical (reflectivity) intensity vs. wavelength at incident angle 4º 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 4º (b) TMM simulations of optical (reflectivity) field (TE) intensity

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 λ_c= 645 nm (c) spatial maps of optical (reflectivity) intensity vs. angle at λ_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/cm^2 (b) TMM simulated optical (reflectivity) field (TE) intensity localized in MCM16 at λ_c= 665 nm (c) spatial maps of optical (reflectivity) intensity vs. angle at λ_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/cm^2 (MCk13) almost coincide with each other and fitting resolved optical parameters. (b) TMM simulated optical (reflectivity) field (TE) intensity localized in MCk13 at λ_c=545 nm, (c) spatial maps of optical intensity (reflectivity) vs. angle at λ_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/cm^2 (b) TMM simulated optical (reflectivity) field (TE) intensity localized in MCk15 at λ_c=540 nm (c) spatial maps of optical (reflectivity) intensity vs. angle at λ_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/cm^2. 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-doses1×10^{15} (MCH1), 5×10^{15} (MCH2), and 1×10^{16} (MCH3) ions/cm^2 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 λ_c= 556 nm (b) spatial maps of optical (reflectivity) intensity vs. angle at λ_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 λ_c= 548 nm and λ_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/cm^2 (MCH1) at λ_c= 573 nm (b) spatial maps of optical (reflectivity) intensity vs. angle at λ_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/cm^2 ions/cm^2 (MCH2 and MCH3) with their cavity peak wavelengths at λ_c=574 nm, and λ_c= 575 nm respectively.

Figure 7.7: : (a) TMM simulated optical (reflectivity) field (TE) intensity localized in unirradiated microcavity (UC1) at λ_c= 546 nm (b) spatial maps of optical (reflectivity) intensity vs. angle at λ_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 λ_c= 564 nm and λ_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/cm^2 (MCHe1) at λ_c= 574 nm (b) spatial maps of optical (reflectivity) intensity vs. angle at λ_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/cm^2 ions/cm^2 (MCH2 and MCH3) with their cavity peak wavelengths at λ_c=597 nm, and λ_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.

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Abbreviations and Symbols

PSi	Porous Silicon
eV/keV/MeV	Electron volt/ Kilo electron volt/Mega electron volt
nm/μm/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 θ=0º)
S_e/S_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