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ABSTRACT

THE PHOTOLITHOGRAPHIC PATTERNING OF POROUS SILICON USING SILICON NITRIDE AND SILICON CARBIDE FILMS AS MASKS

by Hong Wang

In this study, a simple photolithographic pattern process for porous silicon is described. The process utilizes silicon nitride or silicon carbide coated on the top of silicon wafer as a masks. Then a test pattern was projected on the sample by illumination during anodic etching in HF:ethanol solution which produced microcracks in regions illuminated during anodization. Creation of these microcracks resulted in formation of porous silicon in the underlying regions. The cracking is related to the stress in the thin film. The films with tensile stress exhibit cracking while compressive stress samples do not. Compared to silicon carbide films, silicon nitride seems to be a more suitable etch mask for patterning porous silicon. The patterning of 100 micron features seems possible with this technology.

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> by Hong Wang

A Thesis Submitted to the Faculty of New Jersey Institute of Technology in partial Fulfillment of the Requirements for the Degree of Master of Science in Engineering Science

Interdisciplinary Program in Materials Science and Engineerin

January 1995

APPROVAL PAGE

THE PHOTOLITHOGRAPHIC PATTERNING OF POROUS SILICON USING SILICON NITRIDE AND SILICON CARBIDE FILMS AS MASKS

Hong Wang

Dr. John J. Federici, Thesis Advisor Associate Professor of Physics,

Dr. Roland A. Levy, Thesis Advisor Professor of Physics, Director of Materials Science and Engineering program, NJIT

Dr. David Kristol Professor of Chemistry, Director of Biomedical Engineering Program, NJIT

Date

Date/

1

BIOGRAPHICAL SKETCH

Author: Hong Wang

Degree: Master of Science in Engineering Science

Date: January 1995

Undergraduate and Graduate Education:

- Master of Science in Engineering Science, New Jersey Institute of Technology, Newark, New Jersey, 1995
- Master of Science in Materials Science & Engineering, Jiao Tong University, Shanghai, P. R. China, 1986
- Bachelor of Science in Materials Science & Engineering Beijing Aeronautics Institute, Beijing, P. R. China, 1983

Major: Materials Science and Engineering

Professional Background

- Research Associate, Shanghai Second Medical University, Shanghai, P. R. China, March 1986 - December 1992
- Adjunct Faculty, Shanghai Materials Institute, Shanghai, P. R. China, June 1986 - June 1989

This thesis is dedicated to my husband and daughter, and my parents

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CHAPTER 1

INTRODUCTION

The electronic revolution, which has made a very profound impact on every aspect of modern life, has been made possible by very large scale integration (VLSI) which offers a high level of function integration with a simultaneous reduction of cost compared to large scale integration (LSI). Due to its easy processability and superior structural properties, silicon is at the heart of the revolution and has been called the ideal semiconductor material for microelectronics [1]. The integration of optical components onto a Si-base chip would have a significant impact on numerous technologies including optoelectronic integrated circuit, optical memories and logic, and advanced display system [2].

Unfortunately because of its 1.1 eV indirect band gap, silicon is also characterized by a very poor optical radiative efficiency and only produces light outside visible range. To a limited extent, optoelectronic integration has been achieved within the confined of the technology of III-V materials and their hybridization with Si chips [2]. But until now even the most promising III-V light-emitting compounds, such as GaAs and AlGaAs, are not readily deposited directly onto silicon substrates due to lattice and thermal expansion mismatches. There are numerous problems with these III-V compounds: The stability and long term reliability of the material, and their poor integratility into silicon based circuits [3]. The incompatibility problems of these materials generally degrade the performance of any III-V compound light-emitting device grown onto silicon. An alternative approach involves mounting GaAs-based devices onto silicon wafers; this appears to be costly, timeconsuming, and labor intensive. The idea that one might take advantage of a mature silicon technology to combine communications and display technology with microelectronics technology has generated considerable activity directed at developing a silicon-compatible optoelectronic material.

1.1. Fundamentals of Opto-electronic Interaction in Semiconductor

In order to develop a silicon-based optoelectronic materials, It is important to understand the principle of opto-electronic processes of semiconductor. The term opto-electronics covers basic physical phenomena and device behavior which arise from the interaction between electromagnetic radiation and electrons or holes in a solid. Generally these phenomena result either from the absorption of radiation, with consequent electronic effects or, conversely, from the generation of radiation by electron flow within a semiconductor.

The absorption process of greatest importance in the study of semiconductors involves transitions from states of the valence-band electrons and arises from the optical excitation of electrons across a forbidden gap E_G into the conduction band. Figure 1.1 shows a hypothetical absorption spectrum as a function of photon energy for a typical semiconductor [4]. As can be seen, a number of processes can contribute to absorption. At high energies, photons are absorbed by the transitions of electrons from filled valence band states to empty conduction band states. For energies just below the lowest forbidden energy gap, radiation is absorbed due to the formation of excitons and electron transitions between band and impurity states. The transitions of free carriers within energy bands produce an absorption continuum which increases with decreasing photon energy. Also, the crystalline lattice itself can absorb radiation, with the energy being given off in optical phonons. Finally, at low energies, or long wavelengths, electronic transitions can be observed between impurities and their associated bands. In practice the resulting absorption spectrum is a continuum of intense absorption at short wavelengths, bounded by a more or less steep absorption edge (at $hv = E_G$) beyond which the material is relatively transparent. For most semiconductors this edge occurs in the infra-red part of the spectrum and the absorption can result from 'direct' or 'indirect' optical transitions, depending on the energy band structure of the semiconductor.



Figure 1.1 Hypothetical absorption spectrum for a semiconductor as a function of photon energy [4]

1.2 Transition Process and Energy Band Structure

1.2.1 Band Structure

The band structure of a materials is intimately dependent on several factors, including crystal structure, lattice constant, chemical species, bonding and bonding lengths, electronegativity, stiffness, and elasticity [5]. Many of these factors are lumped into the crystal potential, which is then used in the crystal Hamiltonian to solve for the crystal wave equation. Conventionally, the band structure of a semiconductor is represented by the dispersion relation $E_n(k)$, where E is the energy of an electron (or hole) at the band edge with a wave vector k in the first Brillouin zone (BZ).

Crystal symmetry requires that E(k) have extreme at the zone center and the zone boundary. However, these are not the only points at which extreme can occur. In the elements and compounds of covalent group IV (for example, C, SiC, Si, and Ge), additional extreme occur in the lowest of conduction bands far away from the zone center.

It is known that the valence band structure for most semiconductors is similar, especially at k = 0, the zone center. The energy gap is the difference between the valence band at k = 0 and the lowest point in the conduction band.

1.2.2 Direct Transitions

In a direct semiconductor, the lowest point of the conduction occurs at k= 0, the band structure is shown in figure 1.2. As a consequence of an optically induced transition, an electron is transferred between the two bands without a change in momentum hv since the momentum of the photon involved is negligible. In a quantum-mechanical calculation of the transition probability, this condition for allowed transitions is apparent as energy conservation rule

$$E_f - E_i = hv$$

where initial and final electron energy states are given by E_i and E_f , respectively, and hv is the photon energy.



Figure 1.2 Direct optically induced transition of electron. The incoming photon possessed an energy equal to the band gap

In the process electrons with a given wave vector in the valence band can make only 'vertical' transitions to states in a higher band having the same wave vector. Non-vertical transitions are nominally forbidden. In other words radioactive transitions can occur quite easily as an electron excited into the conduction band minimum at k = 0 with both the initial and final states having the same momentum vector k = 0 and spontaneously decay into the valence band state also at k = 0, yielding a photon of energy equal to the band. (In a semiconductor, this refereed to as an electron-hole radioactive recombination). A good example of such a case is GaAs.

1.2.3 Indirect Transitions

While in the case of indirect semiconductor (such as Si), the case is different. That is, an indirect transition requires the absorption or emission of a phonon to conserve the wavevector. Since the simultaneous absorption of a photon and a phonon is a higher-order process, three-body event (electron, hole, and phonon) is significantly less likely to occur than a direct electron hole recombination, one would expect indirect transition probabilities to be much less than those for direct transition. This simple explanation predicts the inability of indirect-gap semiconductor to emit light efficiently. As shown in figure 1.3, an indirect transition can be described by a direct transition from state 0 in the valence band to a short-lived virtual state I_c in the conduction band with simultaneous absorption or emission of a phonon to scatter the electron from I_c to conduction band state 1. In such a treatment conservation of energy can be relaxed in the transitions to the virtual states because of the short times the electron remains in these states. However, energy must be conserved in the complete indirect process.



Figure 1.3 Indirect optically induced transitions of electrons

1.3 Introduction to the Excitonic Luminescence

All luminescent processes must first involve the excitation of carriers from lower to higher energy states. Once in these higher states, they may decay to lower states by giving up heat to the lattice or emitting photons of a given energy. One more introductory point concerning excitonic luminescence is in order. Excitons are electron-hole pairs that are bound to each other and may, in addition, be either free or bound to impurities. When excitons decay radiatively, light is emitted. This excitation process typically moves electrons from the valence band to the conduction band and may be achieved through several methods.

1.3.1 Engineering Luminescent Transitions in Silicon

A number of ways are available for the engineering of luminescent transitions in an indirect material. Luminescence is a result of significant overlap in the electron and hole wave functions. Whenever there is such overlap, luminescence is possible. However, the strength of the luminescence, that is, the emission rate and quantum efficiency depends on the extent of this overlap and the transition probability. Until now engineering solutions seeking to increased this overlap can be split into four classes[2].

1) In impurity-mediated luminescence, an impurity that has an energy level in the gap of the semiconductor is used as an intermediary state through which the electron can recombine with the hole. The impurity level must be dispersed in k-space to be efficient.

2) Band structure engineering makes use of alloys. Two or more group IV elements are alloyed to shift the energy bands a little so that a direct transition is allowed.

3) Quantum confinement can also increase the probability of a direct transition, which means an ultra-short-period superlattice with periodicity comparable with the lattice constant induces greater overlap of the conduction band states at the zone edge with the valence band states at the zone center, increasing the possibility of a direct transition and other strict quantum confinement in one or zero dimensions, such as quantum wires, dots or porous silicon.

4) Strictly hybrid approaches, in which a direct bandgap material is grown on or joined to Si. A typical case is to integrate a III-V material on Si.

Porous silicon, as a special case of the quantum confinement, has attracted much attention because of its efficient room-temperature visible-light photo-luminescence (PL) and also electroluminescence (EL) [6, 7].

1.4 Luminescent Porous Silicon

1.4.1 The History of Luminescent Porous Silicon

Porous silicon is not a new material. It was first reported over 30 years ago during the electropolishing of silicon in aqueous hydrofluoric acid (HF) [8], and was known to exhibit visible photoluminescence (PL) at 4.2 K [9]. But in 1990, it was discovered that electrochemical and chemical etching of Si in HF solution for extended period of time results in a material to exhibit relatively intense visible PL even at room temperature [6].

A general method for making porous silicon is anodic etching of single-crystal polished (100) wafers in a 50:50 (by volume) solution of aqueous 49% HF and ethanol. The anodization process generates an array of small holes near the Si surface. The pore size (~10Ű-500Ű) depends on the composition of the electrolyte, the type and resistivity of the initial Si substrate, the current density during anodization, and the post-anodization treatment [2]. A typical PL spectra from porous silicon is shown in figure 1.4. Since porous silicon exhibits strong visible and is inexpensive to produce, it is believed to be a promising building block for a new generation of optoelectronic integrated circuits fully compatible with silicon-based microelectronics.



Figure 1.4 Room-temperature photoluminescence after anodical etching in HF solution for the times indicated [6]

1.4.2 Mechanism of Visible Luminescence from Porous Silicon

To date, several mechanisms have been proposed to account for the PL from porous Si. The above-bandgap emission was initially modeled as two dimensional confinement in quantum wire-like structures of highly porous Si [6]. Alternatively, it has also been suggested that luminescence originates in the amorphous regions in porous Si [10, 11]. Other hypotheses for the luminescence of porous Si include surface passivation [12, 13] and the formation of hydride complexes [14] or siloxene [15].

1.4.2.1 Quantum Confinement

The first model to describe the photoluminescence from porous silicon attributed it

to quantum confinement. In this model, Canham [6] suggests that charge confinement within the thin silicon "wires" result in an increase in the band gap of the silicon which "pushed" it into the visible region, and more importantly, increases the probability of a direct band-gap recombination. Presumably, the quantum aspects relax the k-selection rule either through a Heisenburg uncertainty effect which increases the effective electron (hole) concentration at direct transitions in the energy-versus-momentum diagrams or through a fundamental restructuring of the silicon energy bands. Lehman and Gosele [16] have provided optical absorption evidence for an increased band-gap energy to about 1.5 eV in p-type silicon in support of the proposed mechanism.

It is known that dramatic quantum size effects should occur as the features size shrink below the Bohr radius (~5 nm) of the free excition in bulk Si. Transmission electron microscopy, Raman spectroscopy, and X-ray diffraction measurements have confirmed that the principal feature of porous silicon layer (PSL) is extremely fine structure (~3 nm). Under the right conditions, a large pore structure is created, in which as much as 85 to 90% of the Si is etched off leaving filaments as thin as a few nanometers across.

The principal evidence supporting the quantum confinement model is the observed general blue shift of the PL as the porosity increases (i.e., with longer anodization times). The blue shift has been attributed to the decreasing dimensions of the quantum-sized features which results in an increase in the energy bandgap.

1.4.2.2 Amorphous Si-H_x Model

This model suggests that luminescent PS is primarily amorphous in character and is supported by some experimental evidence. Micro-Raman spectroscopy indicates that the upper regions of PSL, from where the PL is believed to originate, are primarily amorphous-like in character and are similar in nature to amorphous Si:H (a-Si:H) with extremely high impurity levels. The a-Si:H films, grown by homogeneous CVD, was demonstrated a decade ago to emit visible PL at room temperature and are characterized by extremely high concentrations of H and relatively low dangling band densities [17]. But this mechanism was disputed by initial luminescent PS work, which reported that high porosity PSL retained complete crystallinity.

1.4.2.3 Surface Passivation

Although quantum confinement can explain why porous silicon emits light in the visible range, experimental evidence exists suggesting that the simple quantum confinement model is either inadequate or incorrect. A simple example is the apparent red shift of the PL peak when a freshly anodized PSL is removed from solution. Hydrogen thermaldesorption studies revealed the importance of hydrogen passivation for light emission from as-anodized wafers. It has also been demonstrated that surface passivation with good quality oxide grown by electro-oxidation or rapid thermal oxidation produces efficient luminescence. The phenomenon suggests possible contribution from surface passivation. The role of surface passivation in luminescent PS is very complex. The relatively low dangling bond density on the hydrogen or oxygen passivated porous Si is thought to enhance radiative recombination. The internal surface of as-anodized PSL has been reported to be passivated with H and extremely high concentrations of C, N, O, and F were observed by secondary ion mass spectroscopy even in freshly anodized luminescent PS sample. An impurity, in high enough concentrations at the surface, might serve as a perturbing potential that might cause a red shift.

1.4.2.4 Formation of Hydride Complexes or Siloxene

Another interpretation of the luminescence from PSL is based not on quantum confinement but rather on the formation of complexes. Of particular emphasis of this interpretation are Si-O-H compounds derived from siloxene (Si₆O₃H₆). Fluorescence from these compounds was observed over half a century ago. The three basic structural models of siloxene proposed in the literature are sheet-like structures, layers of hexagonal Si rings connected by bridging O with H termination of the remaining Si dangling bonds and linear Si chains connected by bridging O with H termination of the remaining Si dangling bonds [18]. Pure siloxene is greenish white in color and luminescent in the green, which is coincidentally the in situ luminescence color of porous Si. With the large reactive surface area, gradual displacement of H with other impurities such as O during air exposure could effect the red shift that is observed when PSL are removed from solution.

As synthesized siloxene is polycrystalline. The two dimensional corrugated crystalline siloxene layers change into a three dimensional amorphous compound in the presence of moisture through oxidation. As the structure changes, shifts in the absorption edge can be observed. These conclusions are supported by changes in Fourier transform infrared (FTIR) spectra.

A fundamental inconsistency of data supporting a siloxene-based mechanism for luminescence from PSL is the fact that siloxene annealed at 400°C in air still luminescent, whereas most luminescent PSL annealed either in air or in vacuum at 400°C do not luminesce (or luminesce very weakly). This fact is especially important since surface passivation studies indicate that the FTIR signatures change and luminescence disappears when PSL are annealed in vacuum at 400°C.

1.4.3 The Application of Luminescent Porous Silicon

Although the microscopic details of luminescent porous silicon are complex and as yet incompletely understood, some simple devices including light-emitting diodes [19, 20] and photodetectors [21] have been successfully fabricated in light-emitting porous silicon.

Figure 1.5 shows the current-voltage (I-V) characteristics of the metal-PS-silicon (MPS) photodiode in the dark and under irradiation of room light. A rectifying behavior can be seen from the I-V curves. The forward bias case is similar to a Schottky-like junction. The reverse bias current depends very strongly on the presence of room light. Curve (a) indicates the leakage current (I_d) in the dark and curve (b) is the current (I_b) obtained with the room light on. The current difference (I_p = I_b - I_d) is generated by incident photons.

Recently, it has been reported that films of luminescent porous Si can be fabricated to display well-resolved Fabry-Perot fringes in their luminescence spectra [22]. Also It has

been shown that polycontrast images can be replicated and that the variation in light intensity can be used to change the etch rates at the Si electrode [23]. This procedure produces two phenomena: first, the etched image appears with false colors under



Figure 1.5 Current-voltage characteristic of a MPS photodiode without light (a) and (b) with room light [21]

incandescent or fluorescent lights because of thin-film optical interference effects and seconds, the image photoluminesces with different emission energies at different spots. The variation in PL energy across the image depends on the photocurrent that existed at each particular location on the wafer during the etch. In addition, photoetching a grid or multi-line pattern generates a three-dimensional diffraction grating, which suggests that holographic information can also be stored in this process. These results may be applicable to optical and holographic display, waveguide, or data storage technologies.

On the other hand, it has shown that patterns of luminescent porous Si can be etched into Si substrates by projecting a high-contrast image onto the electrode during anodization [24]. Figure 1.6 presents a fluorescence microscope image of the luminescent pattern etched in an n-type silicon sample. The regions that were not illuminated during etch display no evidence of porous Si formation, while those that had been illuminated show the characteristic luminescent porous Si surface.



Figure 1.6 Fluorescence micrographs of an n-Si wafer showing the lithographic pattern of luminescent porous Si [24]

The another research work about patterning porous silicon was reported by Steckl and et al in 1993 [25]. In their works, patterned SiC/Si heterostructures have been treated by anodic etching. The SiC/Si structure was initially formed by the rapid thermal chemical vapor deposition (RTCVD) of a thin crystalline β -SiC layer on a Si substrate. Next, the SiC film is patterned by reactive ion etching (RIE) to produce defined exposed Si regions and complementary Si regions protected by the SiC film. For n-type Si substrate, short etching times (<3 min.) result in selective-area UV-induced visible photoluminescence being observed only from the SiC regions. In contrast, for p-type Si, the exposed Si regions readily emit after even short anodization times, while the SiC-masked region do not emit visible light even after 5 min. anodization. This selective-area photoemission is shown in figure 1.7 and attributed to rapid lateral etching of the n-type surface under the SiC layer due to high surface stress caused by the lattice mismatch between SiC and Si.



Figure 1.7 Images of patterned and anodically-etched SiC/Si samples [25] (a) SEM microphotograph, n-type Si (b) UV-induced emission, n-type Si (c) UV-induced emission, p-type Si

1.5 Objectives of This Thesis Work

As part the eventual goal of converting electrical to optical signal directly and efficiently on a Si computer chip, which would result in faster interconnections and possible optical logic elements, it is essential to test the compatibility of light-emitting porous silicon with the present Si microelectronics technology. A key step in the fabrication of integrated optical/electronic circuits utilizing porous silicon is the incorporation of the material into a circuit design directly on the single crystal Si substrate.

Microcircuit fabrication requires that precisely controlled quantities of impurities be introduced into tiny regions of the silicon substrate, and subsequently these regions must be interconnected to create components and VLSI circuits. The patterns that define such regions are created by lithographic processes. That is, layers of photoresist materials are first spin-coated onto the wafer substrate. Next, the resist layer is selectively exposed to a form of radiation, such as ultraviolet light, electrons, or x-rays. An exposure tool and mask, or data tape in electron beam lithography, are used to define the desired selective exposure. For positive resist, the patterns in the resist are formed when the wafer undergoes the subsequent "development" step. The areas of resist remaining after development protect the substrate regions which they cover. Locations from which resist has been removed can be subjected to a variety of additive or subtractive processed that transfer the pattern onto the substrate surface.

Previous efforts to pattern porous silicon have utilized selective etching of ionimplanted or thin film mask patterned prior to anodic etching to form selected porous silicon region [25, 26]. In this paper, a simple photolithography process to pattern porous silicon was studied where silicon wafer is first coated with a thin film of SiC or SiN as a mask, then a test pattern is projected onto the coated wafer using photoanodic etching. It is known that SiN and SiC are impervious to most chemicals which make them were widely used in numerous semiconductor applications for insulation, isolation, passivation and etch masking. In this thesis works, wafers were coated with SiN or SiC films and were then illuminated during the etching process. The resulting porous silicon was selectively formed: luminescence from porous silicon is only observed from regions of the wafer which were illuminated during the etching process. It is expected that this simple, inexpensive and reproducible process for patterning porous silicon would stimulate more experimental activity in this rapidly growing field.

CHAPTER 2

EXPERIMENTAL PROCEDURE

2.1 Sample Preparation

2.1.1 Materials

The samples used in this work were single-crystal polished (100) wafers of boron-doped (p-type) Si of 10-15 Ω cm resistivity. The exact measurement of resistivity was done by a model SPP 5000 Four-Points Probe (Veecp Inc.) in NJIT clean room.

2.1.2 Al Deposition

To form a good ohmic contact to a pure Si sample, a thin layer of Al was evaporated on the back of the wafers (unpolished side). The evaporating equipment consists of Varian vacuum system which includes evaporation chamber, roughing (mechanical) pump, high vacuum (diffusion) pump and vacuum gauges (CHA Industries), a type 3PN1010 Variable Autotransformer (Staco Energy Products Co.) and a model IL800 thickness gauges (Intellemetrics).

Before evaporation all the wafer must be immersed in the dilute HF solution (HF:H₂O = 1:40) about 1 minute to remove oxide and dirt, so that a direct contact between Al and Si wafer can be achieved. The thin Al films were deposited by evaporation with resistance-heated sources. That is, a wire of low vapor pressure metal (e.g. W), is used to support small strips of the Al to be evaporated. The W-wire is then resistively-heated. The Al first melts and wets the heated filament, and evaporation ensues. Evaporation is carried out

under high-vacuum conditions. The roughing pump (mechanical pump) was first used to evacuate the chamber to a medium vacuum range($\sim 10^{-2}$ Pa), then the high-vacuum pump (diffusion pump)is used to reduce the pressure to 10^{-5} Pa. Once the pressure of the chamber was about $1*10^{-5}$ Pa, turning on the autotransformer slowly to heat the Al wire. When the voltage was increased to 40~60 V, the Al started melting. After Al evaporation, the voltage was reduced to zero. After about 10 minutes of cooling the sample is removed out and kept in a clean box. Generally, the thickness of Al film was kept around 70 Ű.

2.1.3 Chemical Films Deposition

The amorphous silicon carbon or silicon nitride films were deposited on the top (polished side) of silicon wafers. The amorphous silicon carbon films were synthesized by low pressure chemical vapor deposition (LPCVD) at temperatures below 850°C using ditertiarybutylsilane (DTBS) as a single precursor [27]. The amorphous silicon nitride films were synthesized either by LPCVD using DTBS and ammonia (NH₃) as precursor [28] or by plasma enhanced chemical vapor deposition (PECVD) using diethylsilane (DES) as precursor [29].

The LPCVD reaction chamber consists of a fused silica tube. It was mounted horizontally within a three zone Lindberg furnace and connected to a vacuum station comprised of a booster pump model EH 250 and a mechanical pump model E2M40. The furnace temperature was kept constant across all three zones and confirmed using a calibrated type K thermocouple. An MKS mass flow controller model 8240 and an MKS200 sccm vapor phase flow controller model 1150B-162M were used to measure and control the flow rate of ammonia and DTBS respectively.

PECVD was carried out in Applied Material AMP 3300 IIA Plasma system in which a glow discharge progresses in a low pressure gas using a high-impedance power supply. The reaction chamber is an aluminum cylinder with aluminum plates on the top (upper electrode) and the bottom (susceptor). There are three resistance heater rings which divided the susceptor into three zones: inner, center and outer zone. The silicon wafers were placed separately in the outer zone of the susceptor.

2.1.4 The Stress Measurement of Films

Film stress was determined with a home built system that measured change in the radius of curvature of a wafer resulting from deposition an a single side. Such depositions were achieved by placing two wafers back to back. Figure 2.1 shows an optical imaging system for setup. The distance between two points generated by light from two fixed and parallel He-Ne lasers was determined after reflection from the surface of a wafer before and after deposition. An angled mirror was used to project the reflection of the two points onto a wall where their separation could be more accurately measured.

2.1.5 The Thickness Measurement of Films

The film thickness was measured using a Nanometrics NanoSpec/AFT nanospectrometer by inputting refractive index value. The refractive index value were determined by a Rudolph Ellipsometer, which contains a He-Ne laser (6238 A° wavelength).



Figre 2.1 Optical system for stress measurement setup

2.2 Etching Procedure

2.2.1 Electrochemical Etching

The porous silicon was prepared by the electrochemical etching process. The etching bath was a 50:50 (by volume) solution of aqueous 49% hydrofluoric (HF) and 98% ethanol (C_2H_5OH). The apparatus shown in figure 2.2 are consisted of a model 225 Current Source (Keithley Instruments) and EW 24 Galvanometer to measure and monitor the current amount during etching process. The partially submerged Si sample was connected to the positive terminal of current source while a platinum wire was used for the cathode. The etching was carried out in optical-quality plastic cuvettes. During one hour anodization process, the current density was kept approximately 10-15 mA/cm². After etching process, the samples were rinsed in deionized water and dried in air.



Figure 2.2 Schematic of electrochemical etching process

2.2.2 Photoelectrochemical Etching Process

Photoelectrochemical etching was carried out in the apparatus shown in figure 2.3. During anodization the wafer was illuminated from the front using a mercury arc lamp and two reducing lens. Then the light was filtered through a 546 nm (green color) bandpass filter. To demonstrate the lithographic fabrication without the use of photoresists, using photoanodic etching, a pattern was projected onto the sample during photoelectrochemical etching process. The pattern used in the work was shown in figure 2.4. It was fabricated as the light only can pass through the NJIT letters. The light measured at the sample was about 8 mW.



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Figure2.3 Schematic of photoelectrochemical etching process



Figure 2.4 NJIT Pattern

2.3 Photoluminescence Measurements

The photoluminescence spectrum from porous silicon was gathered at room temperature using a home built-system which measured the luminescence from sample excited using an argon-ion laser (wavelength = 488 nm, power = 10 mW) and spectrally resolved using a scanning grating monochrometer. Preliminary measurements of PL is shown in figure 2.5. The resultant PL is detected using a photo multiplier tube and a model SR510 lock-in amplifier (Stanford Research Systems). All data was collected by a computer automatically in the range of 500-1000 nm. To make all data consistent, the system is calibrated before every measurement, using two light laser wavelengths (488 and 514 nm).

2.4 Optical Microscopy Examination

After etching process, all the samples were examined by optical microscopy to check the presence of microcracks on the samples and take photographs. All the optical examination were done on model Zepopan-Binolux Microscope (Hacker Instruments) in clean room.



Figure 2.5 Schematic illustration of PL measurement

CHAPTER 3

RESULTS AND DISCUSSION

3.1 Thickness Measurement of Films

The SiC films used in this study were synthesized by LPCVD, while the SiN films were synthesized either by LPCVD or by PECVD. The results of film thickness were listed in Table 3.1.

Table 3.1 The thickness results of chemical fill	ms
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Sample No.	Synthesis Method	Thickness (A°)
SiN 1	LPCVD	3051
SiN 3	LPCVD	2675
SiN 6	LPCVD	1665
SiN 8	LPCVD	2367
SiN 11	LPCVD	4306
SiN Y1	PECVD	~2500
SiN Y2	PECVD	~3000
SiC12	LPCVD	~100
SiC 13	LPCVD	~100
SiC 16	LPCVD	~100

3.2 Stress Measurement of Film

It is known that nearly all films are found to be in a state of internal stress, regardless of the means by which they have been produced. The total stress usually is written as [30]:

$$\sigma = \sigma_{th} + \sigma_{int}$$

where σ_{th} stands for the thermal stress and σ_{int} is the intrinsic stress. Thermal stress results from the difference in the coefficients of thermal expansion between the film and the substrate. The intrinsic stress results from the structure of the growing films. The thermal stress is due to the constraint imposed by the film-substrate bonding and is given by:

$$\sigma_{\rm th} = (\propto_{\rm f}, \propto_{\rm s}) (\Delta T) E_{\rm f}$$

where: α_f and α_s are the average coefficients of thermal expansion for the film and the substrate, respectively; ΔT is the temperature of film growth minus the temperature of measurement; and E_f is the Young's modulus of the film. The σ_{th} can be of either sign (positive is tensile, negative is compressive), based on the relative values of α_f and α_s . The intrinsic stress in a film depends on thickness, deposition rate. deposition temperature, ambient pressure, method of film preparation, and type of substrate used and other parameters [30].

Measurement of stress is a relatively straight-forward procedure based upon beam bending. A tensile stress in the film bends the substrate so that it becomes concave, while a compressive stress makes the substrate convex [30]. The stress is measured by homemade system, which used two He-Ne laser beams shown in figure 2.1. In this study the stress was determined by the formula given below [29]:

$$=\frac{Et_s^2}{6(1-\tau)Rt}$$

where E = Young's modulus for the substrate,

 τ = Poisson ratio for the substrate,

 $t_s =$ thickness of the substrate,

t = thickness of the film,

R = net radius of curvature.

R, the radius of curvature is calculated from following formula:

$$R = 1 / (1/R_2 - 1/R_1)$$

where R_1 is the radius of curvature of the wafer before deposition, R_2 is the radius of curvature of the wafer after deposition. For Si (100), E / (1- τ) = 1.8 * 10¹¹ Pa. Wafer with a thickness of about 525 μ m were used for stress measurement. Thus for the system used in this experiment, a simple formula is produced [29]:

$$= 12.4 \times \frac{d(mm)}{t_f(m)} (Pa)$$

Where d = the distance change on the screen before and after deposition, and $t_f =$ film thickness.

All deposited films by LPCVD were found to have a tensile stress, while as-deposited films by PECVD were found to have a compressive stress. Compressively stressed films would like to expand parallel to the substrate surface, and in the extreme, films in compressive stress will buckle up on the substrate. On the other hand, films in tensile stress would like to contract parallel to the substrate, and may crack if their elastic limits are exceeded [30]. Table 3.2 lists all experiment stress results.

 Table 3.2 Stress measurement result

Sample No.	Stress type	Stress (Pa)
SiN 1	tensile	1.19 * 109
SiN 3	tensile	1.84 * 10 ⁸
SiN 6	tensile	8.10 * 10 ⁸
SiN 8	tensile	4.15 * 10 ⁸
SiN 11	tensile	6.71 * 10 ⁸
SiN Y1	compressive	-3.90 * 10 ⁸
SiN Y2	compressive	-1.70 * 10 ⁸
SiC12	tensile	5.50 * 10 ⁸
SiC 13	tensile	5.50 *10 ⁸
SiC 16	tensile	7.50 * 10 ⁸

3.3 Photoelectrochemical Etching Process

In this study, it has been demonstrated that the silicon wafer coated with a thin film of either SiC or SiN do not exhibit photoluminescence characteristic of porous silicon, when the coated wafers are either etched in HF in the presence of illumination without current or etched in HF solution in the absence of current with illumination. However, when silicon with either SiC or SiN deposited by LPCVD coatings are etched by photoelectrochemical etching process, (e.g. illuminated during the anodized etching process), photoluminescence characteristic of porous silicon is observed from areas exposed to light during the etching process. While silicon coated with SiN synthesized by PECVD failed to exhibit this effect.

It is known that films consisting of SiC and SiN are chemically inert and impervious to HF solutions. What is the reason to make the anodized SiN/Si and SiC/Si structure synthesized by LPCVD exhibit visible luminescence only in the regions exposed to light during the etching process? To explain this phenomenon, microscopic examination on various of samples were carried out.

Microscopic examination of SiN/Si or SiC/Si samples exhibiting luminescence showed the presence of microcracks in the illuminated areas of both samples, while areas which were not illuminated showed no evidence of microcracking. Also the SiN/Si samples synthesized by PECVD showed no cracking either. The photographs of microcracks of both the SiC and SiN films were taken and showed in figure 3.1. Based on this observation, it is reasonably to postulate that these microcracks are related to tensile stress in the films and provided path for HF solution to permeate the coatings and etch the underlying silicon to form porous silicon in these defined areas.

From table 3.2, it is clear that there exists stress in the SiC and SiN films no matter how the films was deposited. The SiC and SiN films synthesized by LPCVD presented tensile stress. Conversely, SiN films deposited by PECVD showed compressive stress. It is believed that the combination of illumination and anodized etching exceed the elastic limits of films with tensile stress because of tensile stress intending to pull the film apart which results in microcrack formation, while the films with compressive stress tend to hold the films together. This assumption is consistent with the observation obtained by microscopic







SiN3

Figure 3.1 (a) The presence of microcracks in both the SiC and SiN films







SiN8









SiC12

Figure 3.1 (c) The presence of microcracks in both the SiC and SiN films









Figure 3.1 (d) The presence of microcracks in both the SiC and SiN films

examination of microcracks of films. Figure 3.2 showed the stress in the films versus the crack area fraction of the films. The fraction of crack area was estimated by measuring the crack regions (figure 3.1 dark area) compared to the total area. The measured results of crack area fraction of coated SiN films samples are shown in Table 3.3. As shown in figure 3.2, it is obvious that the films with tensile stress (positive stress) presented crack while the films with compressive stress (compressive stress) did not.

Sample No#	Crack area fraction of films
SiN 1	0.33
SiN 3	0.22
SiN 6	0.29
SiN8	0.22
SiN11	0.11
SiN Y1	0
SiN Y2	0

 Table 3.3 The Crack area fraction of coated SiN films samples

Meanwhile the hypothesis of the microcracks in the films is related to the stress and permits the HF solution to etch the underlying Si surface and form porous silicon is also supported by the photoluminescence intensity of SiN films coated on silicon wafers versus stress of films shown in figure 3.3. The photoluminescence measurements indicated that the luminescence was emanated from the cracks in the case of the SiN coatings (darker areas of figure 3.1). Similarly, the measurement on SiC films showed that the photoluminescence originates not from within the cracks but from near the periphery of the SiC/silicon interface, which is similar to the results reported by Steckl in SiC/Si heterstructures and was explained by the onset of a preferential anodization due to stressinduced charge carrier generation at the interface [25].

Since most of the luminescence from the SiN films appear to originate from the cracks, one would expect that the photoluminescence intensity should be proportional to the crack areas of films. This assumption also was verified by the plots of the photoluminescence intensity versus crack area fraction of the films shown in figure 3.4. From figure 3.4, it is apparent that the photoluminescence intensity increase linearly with the increasing of crack area fraction.

3.4 Patterning of Porous Silicon

A photolithographic fabrication of porous silicon without the use of photoresists was conducted in the work. A simple shadow mask with the letters NJIT was illuminated with the mercury lamp with the resulting image focused with a pair of lenses onto the coated silicon wafer. When the coated samples were illuminated during the anodical etching process, the NJIT pattern was successfully transferred to the silicon samples. The selective-area photoemission is shown in figure 3.5. It clearly indicates that only NJIT was emitting. Uniform emission is obtained in these regions, with a 100 microns features. The size of the pattern on the silicon wafer was approximately 1.5 mm by 5 mm. Better pattern



Figure 3.2 The stress in the films verse the crack area fraction of the films



Figure 3.3 The photoluminescent intensity of SiN films verse the stress of SiN films



Figure 3.4 The photoluminescent intensity of SiN films verse the crack area fraction of the films

transfer was obtained for SiN coated wafers compared to SiC coated wafers. For SiC coated samples, the pattern is clearly visible on the sample, but the process was not always reproducible. For SiN coated wafers, the pattern is reproducible transferred to the porous silicon. Features of ~100 microns are deemed possible because of the exposed crack size.



Figure 3.5 Image of patterned in anodically-etched SiN/Si sample

To improve patterning resolution, two areas of work are underway: (1) by controlling the fracturing of the SiC and SiN films to achieve better patterning resolution and (2) removal of the fractured fragments on the top of the wafer to expose more silicon area to improve the luminescence intensity of the patterned porous silicon. Several lift-off procedures are being tested in our laboratories.

CHAPTER 4

CONCLUSIONS

1. In this study it has been demonstrated that the silicon wafers coated with a thin film of either SiC or SiN do not present photoluminescence characteristic of porous silicon when the coated wafers are anodized etched in HF solution or etched with illumination but without current in HF solution.

2. A new technique of patterning porous silicon was described in the paper. After the SiC or SiN thin films, synthesized by LPCVD, were coated on the top of silicon wafer as a mask, a test pattern was projected successfully onto the coated wafer by illuminating the sample during the anodic etch. The main advantages of this technique are its simplicity and minimal processing steps.

3. A hypothesis proposed to explain the selective-area photoemission of coated wafer was that localized light-induced fracturing of the thin film permits the HF:ethanol solution to etch the underlying silicon wafer. The cracking of the thin film was related to the stress in the thin film. The film with tensile stress exhibited cracking while the film with compressive stress do not. It was suggested that SiN would be a more suitable etch mask for patterning porous silicon because SiC film was not always reliable.

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