ENGINEERING STRAIN-INDUCED SELF-ROLLING
SEMICONDUCTOR TUBES THROUGH GEOMETRY AND
PATTERNING

BY
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THESIS
Submitted in partial fulfillment of the requirements
for the degree of Master of Science in Electrical and Computer Engineering
in the Graduate College of the
University of Illinois at Urbana-Champaign, 2010

Urbana, Illinois

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ABSTRACT

This thesis investigates the effect of the geometry of the strained bilayer mesa on the physical shape of semiconductor nanotubes, which is essential for precise positioning and uniform large area assembly. Experimental and simulation results of the rolling behavior of tubes are discussed for various geometries. The study attempts to understand the energy minimization process of strained heterofilms at the nanoscale level and to successfully interpret these phenomena for design and control of semiconductor nanotubes. Generally, III-V compound semiconductor tubes tend to roll up along the length of the mesa due to the lowest energy associated with the state in contrast to short side or mixed rolling behavior. However, the configuration of tubes can be modified by engineering the mesa geometry through additional patterning. Possible applications of new structures and devices obtained through semiconductor nanotubes are considered.
To Harish, my parents, and my sister
ACKNOWLEDGMENTS

I would like to thank Professor Xiuling Li for her invaluable guidance and continuous encouragement throughout the term of the project. I would also like to specially mention Ik Su Chun’s contribution during the initial stages of the project. He has been my mentor and has put in a lot of time and effort to train me in cleanroom fabrication processes and concepts used for this thesis project.
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CHAPTER I: INTRODUCTION

Over the last decade, tremendous effort for the heterogeneous integration of III-V and CMOS (complementary metal-oxide-semiconductor) functionality has been made. One of the biggest challenges that nanotechnology faces is fabrication and reproducibility of precisely dimensioned nano- and micro-objects and their reproducibility. Research shows that deviation of more than 10% of an element’s size in semiconductor devices is intolerable for accurate device operation [1, 2]. Hence, precise positions and dimensions are essential. Lately, nano-objects such as nanotubes, nanoscrolls, nanohelices and various other structures have acquired great significance in many fields due to their ease in fabrication and unique physical and optical characteristics. This thesis discusses the characteristics of semiconductor nanotubes and the essential parameters required for achieving a precise, ordered, and uniform large area assembly of these tubes.

1.1 Compound Semiconductors

Compound semiconductors have been an integral part of solid state electronics for more than three decades. This unique class of compounds is formed by integrating elements from group II to group VI of the periodic table. Most often, compounds obtained from groups III-V and II-VI have been widely studied and are known to exhibit distinctive optical and electrical properties. While silicon-based systems are manufactured at production scale in the microelectronics industry, III-V compound semiconductors are being used for development of novel electronic and optoelectronic device structures in industry because of the advantages they offer such as direct bandgap, better bandgap tunability, versatile heterojunctions, increased carrier mobility and controllability of doping levels and types [3, 4]. The composition of the constituent materials determines the physical characteristics of these semiconductors, which are being used in many
optoelectronic and photonic devices, such as high electron mobility and heterostructure bipolar transistors [5], diode lasers [6], light emitting diodes [7, 8], and photo detectors [9] etc.

1.2 Properties of Compound Semiconductors

Compound semiconductors are binary and ternary alloys composed of elements from different groups of the periodic table. Because of the combination of different elements, the physical properties of these materials have various bandgaps, crystal lattices, electron and hole mobility, and thermal conductivity. The difference in the electronegativity of these elements results in both covalent and ionic bonds between the neighboring atoms. The atomic arrangement is usually either a zinc-blende cubic crystal or an hexagonal wurtzite structure depending on the nature of the bond formed. Larger bandgaps are possible when the bonds are more ionic. In direct bandgap materials, electron transition from valence to the conduction band is possible without any phonon generation, as opposed to indirect bandgap materials. The lattice constant of these structures depends on the constituent atoms and increases with diameter of the atoms. Thus, by tailoring the bandgap of these materials, various optoelectronic and photonic devices can be produced. The electrical and optical behavior is influenced by the role of dopants, defects, and impurities in the semiconductor materials. The electrical properties of compound semiconductors depend mainly on the carrier concentration and mobility, which are influenced by scattering mechanisms inside the crystal lattice. Because of the direct transition bandgap and small effective mass, electron mobility in compound semiconductors is greater than in silicon. These electrical and optical properties are strongly temperature dependent and hence can be tuned appropriately.
1.3 Compound Semiconductor Nanostructures

Three-dimensional nanostructures are important for the realization of quantum effects at the device level. The physics at small dimensions not only is interesting but can be exploited for the purpose of discovering novel device structures. Semiconductor nanostructures have been an active research proposition for many years now and are becoming significant in such fields as solar cells, CMOS engineering, and biological and sensing applications. Structures like nanowires [10], nanotubes [11, 12], nanorods, and nanohelices [13] incorporated with active materials such as quantum wells and quantum dots are used to control the available energy states within the semiconductor device. The fabrication of nanostructures includes a wide variety of approaches starting from bottom-up self-assembly techniques like MOCVD (metal organic chemical vapor deposition), MBE (molecular beam epitaxy), template-assisted growth to top-down lithographic etch techniques. The bottom-up approach allows for formation of nanostructures as small as 1 nm because of the atomic-level precision which is otherwise physically impossible to fabricate using top-down lithographic processes. An emerging class of 3-D nanostructures and nano-objects can be fabricated by strain-driven engineering: a combinatorial approach that uses top-down and bottom-up fabrication methods to form self-rolling structures. Of these, semiconductor nanotubes are studied in detail and analyzed in this thesis.

1.4 Classification of Nanotubes

The term nanotube refers to any hollow cylindrical tube-like structure having dimensions of the order of a few tens of nanometers (10^{-9} m). Nanotubes are classified based on the basis of their structural properties and the materials they are formed from; for example there are carbon
nanotubes, metal oxide nanotubes, and semiconductor nanotubes. To date, carbon nanotubes (CNTs) have been of tremendous interest for many applications of nano- and microelectronics, optics, bioengineering, sensors, and solar cells [14, 15]. The III – V semiconductor nanotubes, on the other hand, are proven to exhibit novel optical properties that are promising for optoelectronic applications such as photonic crystals, waveguides, and lasers [16].

Semiconductor nanotubes are made from elements in groups IIIA, IVA, and VA of the periodic table. Some of the common elements are In, Ga, As, Sb, N and P. Although the name nanotube is common to semiconductor nanotubes and carbon nanotubes, their synthesis and structural properties differ significantly.

1.4.1 Carbon nanotubes

Carbon nanotubes (CNTs) are essentially an arrangement of graphitic carbon sheets of monolayer thicknesses wrapped into cylindrical and cyclical tubular structures. These versatile structures have been reported to have extraordinary mechanical strength and excellent electronic and thermal properties. Carbon nanotubes can either be semiconductors or metals depending on the chirality and diameter; thus they offer infinite possibilities for integration into nanoelectronic devices or active components in transistors. The synthesis of CNTs involves a bottom-up approach [17] through methods of arc discharge, laser ablation of graphite targets, or chemical vapor deposition of carbon.

1.4.2 Inorganic nanotubes

Inorganic nanotubes, sometimes referred to as metal oxide nanotubes, consist of elements from groups II and VI of the periodic table. The importance of these inorganic semiconductor nanotubes is their incorporation of ferroelectric, ferromagnetic, luminescence, and piezoelectric
properties into nanostructures for practical applications. Some popular examples of this class of nanotubes are TiO$_2$, CdSe, and ZnO. Fabrication usually is template assisted in that the nanotubes are grown by thermal evaporation or electrodeposition over nanorods or nanowires [18-20]. These nanotubes, because of their large surface area, nontoxicity and high reactivity, are used in environmental applications, dye sensitized photovoltaic cells, and lithium ion batteries.

1.4.3 Semiconductor nanotubes

Semiconductor nanotubes made from InGaAs/GaAs were first fabricated by Prinz et al. in 2000 [12, 13]. Subsequently, this method was applied to form GeSi/Si [21] micro- and nanotubes that could add to the silicon technology for ICs. Strain-induced formation of other nano-objects [22] has been demonstrated on many epitaxial structures, such as InGaAs/GaAs, InSbAs/InAs, InP/InAs, Si/GeSi, Si/GeSi/Si$_3$N$_4$/Cr, InAs, and Au/Ti films, polymers. On the other hand, semiconductor nanotubes made from other materials such as InP, GaP, GaN can be fabricated using chemical deposition or electrodeposition on nanowires [23-25].

1.5 Compound Semiconductor Nanotubes

Semiconductor InGaAs/GaAs nanotubes are fabricated using self-rolling, strained bilayer films. Heterostructure thin films are grown on a substrate, which is then patterned according to the dimensions of the nanotube required. A buried-in, sacrificial layer underneath acts as a strain-inducing agent that provides entry to the etchant to release the strained bilayer. When these bilayer films are debonded from the substrate, they roll up into micro- or nanotubes. It has been observed that these bilayer films perfectly adhere to form the walls of the tube.

This method combines top-down and bottom-up approaches in fabrication. While the lattice matched bilayer films are grown in a bottom-up fashion using MOCVD (metal organic chemical
vapor deposition) or MBE (molecular beam epitaxy), etching the sacrificial layer to release the bilayer film entails the top-down approach. Such a method has been demonstrated to produce highly ordered arrays of nanotubes of precise length and diameter control [11] with reproducibility. The tube diameter can be controlled according to the lattice mismatch (strain) between the bilayer films and the thickness of the bilayer films. It is important to remember that the thickness of the bilayer films should be less than the critical thickness value for pseudomorphous metastable growth. The top-down technique used to define the mesa pattern ultimately governs the nanotube parameters, such as the number of rotations, location, and orientation of the tube with respect to the substrate. Nanotubes are loosely held on the substrate because of surface tension van der Waals forces. Hence, the tubes can be dispersed in solution and transfer printed to other substrates [26]. Thus, semiconductor nanotubes of precise dimensions can be formed by processes compatible with IC and planar technology.

The formation of the tube is the result of minimizing the total strain energy, which is realized by strain relaxation of the bilayer system. Such a strain-induced formation mechanism has also been applied to construct other 3-D architectures useful for micro-machining and MEMS devices. Due to the possibility of using different materials, controlling the shape of objects and embedding different active materials inside the strained layer system, these 3-D objects are promising candidates for integrated optoelectronic devices, cavity quantum electrodynamics, and biosensing applications.

1.5.1 Physics behind the formation mechanism

A clear understanding of the rolling mechanism is important to obtaining nano- or microtubes of precise dimensions. The bilayer film in nanotubes usually consists of two films, which are
somewhat lattice mismatched (typically less than 7% for III—V material systems) and pseudomorphically grown using MBE or MOCVD techniques. Shown in Figure 1.1 is a schematic representation of the formation process.

In the schematic, the InGaAs layer is shown to have a larger lattice constant compared to AlGaAs and GaAs layers and thus undergoes compressive strain when it is pseudomorphically grown. Throughout this thesis, the bilayer refers to the composite stack grown from above the sacrificial layer. During selective etching of the underlying sacrificial layer, the bilayer film is detached from the substrate. This detachment tends to relax one of the layers with the larger lattice constant in the bilayer film allowing it to expand to its original lattice constant; but such an expansion is restrained by the other layer. Since these forces are generated in adjacent layers and are opposite in direction, a moment of force is created there by bending the bilayer film. The bending is such that the layer with the larger lattice constant forms the outer wall and the layer with smaller lattice constant assembles as the inner wall [27]. It is apparent that an inverted tube can be formed by engineering an opposite type of strain in the bilayer stack. Shown in Figure 1.1 (b) is a schematic of the formation process of a strain-induced self-rolling semiconductor nanotube in three stages. As the sacrificial layer is etched, the bilayer film rolls up to form a tubular structure.

1.6 Properties of III-V Nanotubes

At room temperature, III-V nanotubes are stable and are semiconducting with bandgaps that vary with the composition, thickness, and curvature of the active structure. The III-V nanotubes can be transfer printed onto other substrates and are compatible with large area assembly of ordered arrays [11]. To ensure uniformity in diameter through an individual tube and between tubes, it is
essential to know the factors affecting the resultant tube dimensions such as orientation, geometry of the stripe pattern, and strain in the bilayer. Because of the combination of top-down and bottom-up approach, nanotubes can be fabricated with high aspect ratios while maintaining epitaxially smooth sidewalls with ultra-thin profiles.

Radial flexibility is a significant parameter for tubular structures. InGaAs/GaAs nanotubes roll up along the <100> crystal direction irrespective of the stripe pattern orientation because rolling is influenced by the anisotropy of Young’s modulus in $A_3B_5$ cubic $[28, 29]$ crystals. The Young’s modulus of GaAs in the <100> direction is far less than that in the <111> direction.

Compound semiconductor nanotubes are said to optically superior due to the confinement of light in both radial and axial directions. High index contrast is essential for obtaining strong optical mode confinement. Total internal reflection of light leads to wave guiding in the tube wall and formation of ring modes by constructive interference after a round trip. Due to the high lateral refractive index contrast of semiconductor nanotubes, the field is tightly confined in the lateral direction so that small bending radii can be achieved. A long cavity length folded in a small area increases the quality factor and relaxes the resonance matching condition while maintaining a compact footprint of the device. Three-dimensional nanostructures have been demonstrated to provide an appropriate platform for confining light on the scale of its wavelength in semiconductor microcavities, and are being used to understand the physics of quantum emitters in optical cavities at the nano-scale level. Nanotubes, embedded with light emitting structures such as quantum wells, and quantum dots, have been proven to produce enhanced optical properties. Photoluminescence studies indicate greater than 10-fold enhancement in the intensity compared to the planar analogue due to quantum confinement effects. Some of the devices realized include photonic crystal microcavities, optical microtube
resonators [30, 31], and microtube lasers [32]. Quantum well micro- and nanotubes [33] have been demonstrated to increase the emission intensity before and after formation of the tubular structure due to the radiative recombination of the electron and heavy-hole ground sub-band states inside the strained QW and better optical confinement. Because of the smooth interfaces between layers and the high contrast in refractive index of the semiconductor and air, light is guided efficiently in the tube wall around the tube axis; this makes it possible to design optical microtube ring resonators and lasers. The non-radiative carrier recombination and optical scattering losses are greatly reduced because of the smooth surface of these tubes, which further lowers the threshold and enhances output power. The lasing behavior and optical properties can be further enhanced by modulating the geometry of the edge of the nanotube [34].

1.7 Applications of III-V Nanotubes

- **Optoelectronics:** Quantum dots embedded in III-V semiconductor nanotubes have been used to produce nanoscale lasers [32, 35]. Nanotubes can be designed to form nanocavities that can be included in photonic crystal cavities, or waveguides [31, 36-38].
- **Biomedical engineering:** Active research is being done to investigate the use of nanotubes as a vessel for drug transport or delivery. The need for microfabricated needles with smaller diameters is tremendous, and III-V semiconducting nanotubes answer this need. To deliver DNA and other molecules into living cells for the purpose of diagnosis or treatment, nanotubes are multifunctional and act as microinjecting needles [39, 40]. They also serve as dissection tools that provide deep penetration and reduced tissue damage. Inorganic nanotubes are also used in cell guiding and growth applications [41].
Microfluidics: Nanotubes filled with colloidal nanocrystals [42] or fluorescent dye molecules form nanopipelines, nanochannels or nanoneedles used in biosensors, biolabeling and fluorescence experiments.

Devices: Nanotubes can be doped either n or p type, and hence can be used for implementing non-planar low-dimensional electron systems [43, 44]. Their use as active devices (p-n junctions, diodes) has not been demonstrated yet remains an active research proposition.

NEMS: Nanotubes are being used as NEMS (nanoelectromechanical systems) elements because of their reduced feature sizes, submicron bending radius, mechanical degree of freedom in fabrication, and scalable structures suitable for practical applications such as magnetic conductors, electrostatic actuators, and hot tube sensors [45].
1.8 Figures

Figure 1.1 Schematic showing (a) the pseudomorphic growth process and (b) the formation of a strain-induced self rolling semiconductor In$_x$Ga$_{1-x}$As/GaAs nanotube during selective etching of the AlGaAs sacrificial layer. The formation process involves a bottom-up epitaxial growth with defined thickness and misfit strain, and a top-down aspect with the release of the strained bilayer to form precisely positioned nanotubes.
2.1 Process Design and Mask Patterns

There are two primary aspects that need to be considered for the formation of nanotubes through a strain-induced rolling mechanism. The first step is to build up a strain in a plane by pseudomorphic growth of bilayers on a substrate. The second step is selective etching of the sacrificial layer to release the strain in the bilayer. Before the growth procedure, the strain and composition of each layer in the nanotube stack is calculated from theory. Since most layers are ternary or quaternary alloys, the corresponding lattice constant varies linearly with composition and is calculated based on Vegard’s law. The strain between two adjacent layers is a function of their lattice constants and is represented by the following equation:

\[ \varepsilon = \frac{a_e - a_s}{a_s} \]

where \( \varepsilon \) represents the misfit strain between the substrate with lattice constant \( a_s \) and the epitaxially grown layer \( a_e \). The final tube diameter is a function of the thickness of the strained bilayer stack above the sacrificial layer and the built-in strain. The following equation is used to estimate the diameter of the resultant tubes:

\[ D = \frac{d[3(1 + m)^2 + (1 + m.n)[m^2 + (mn)^{-1}]]}{3\varepsilon(1 + m)^2} \]

where \( D \) is the final diameter of the tube, \( m \) is the thickness ratio of the layers, \( n \) is the Young’s modulus ratio of the two layers, and \( \varepsilon \) is the misfit strain between them. Experimental values are normally 15% – 20% smaller for the materials system studied in this thesis [11, 46], probably due to surface tension, which is not considered in the above equation.
Shown in Figure 2.1 is an example of a typical planar stack structure grown on a GaAs (100) substrate with a buffer layer on top. The structure usually consists of an optically active material such as a quantum well or a quantum dot layer sandwiched between barrier layers (AlGaAs here) grown on a strained layer (InGaAs) with a sacrificial layer of AlGaAs (different composition than the barrier layer) underneath.

It is important to note that while growing the stack structure epitaxially, misfit locations are generated if the layer thickness is greater than the critical thickness at the interface between the adjacent layers. To minimize such defects, calculations are performed to determine the boundary of pseudomorphic and relaxed growth regions for different misfits on the basis of the composition of the individual layers. While this is helpful, the critical thickness can also vary according to experimental parameters, such as growth temperature, rate of growth, and preparation of substrate surface.

Prior to growth, mask patterns consisting of squares and rectangles with dimensions ranging between 3 to 105 µm were designed in LASI software. The rectangular strips were oriented 45° to the wafer cleaving edge <110> during fabrication. It has been demonstrated earlier than nanotubes tend to roll up in the <100> direction irrespective of the strip pattern. Thus, the 45° angle conforms to the <100> direction for a (100) GaAs substrate. Shown in Figure 2.2 is a schematic of the finished mask design for a pattern size of 19 × 50 µm.

2.2 Metal Organic Chemical Vapor Deposition (MOCVD)

For epitaxial growth of strain-engineered structures, a high degree of uniformity with precise control of thickness, doping, and composition is required. Metal organic chemical vapor deposition (MOCVD) is a growth technique based on vapor phase epitaxy (VPE) that deposits a
thin film on a crystalline substrate such that the atoms in the growing layer mimic the arrangement of the substrate. Usually employed for compound semiconductors, MOCVD uses metal organic compounds and offers the advantages of abrupt interfaces, in situ doping, and faster growth rate compared to other VPE growth techniques such as molecular beam epitaxy (MBE). Metal organic precursor sources containing the target element such as tri-methyl gallium (TMGa) for Ga or tri-methyl indium (TMIn) for indium, are flown into a quartz tube reactor along with hydrides, such as arsine (AsH₃) or phosphine, and dopants such as diethyl zinc (DEZn) or disilane (Si₂H₆), which are typical p and n type dopants, respectively. Under suitable growth temperatures, the chemical reaction undergone by the precursors and the hydride sources deposits the target material on the substrate, and the by-products are released through an exhaust.

The samples required for this study were grown by Kevin Bassett and Ik Su Chun in an AIXTRON low pressure MOCVD reactor or a Thomas Swan atmospheric MOCVD reactor. To grow the nanotube structure, a semi-insulating (100) on axis GaAs substrate is placed on the susceptor inside the reactor chamber. The precursor and hydride gases are flown, and deposition occurs on the surface of the substrate. Starting with a growth of a GaAs buffer layer, an AlGaAs sacrificial layer is grown next, followed by the growth of strained InGaAs bilayer. The growth is finished with a final cap layer of GaAs to prevent oxidation of the sacrificial layer during etching. To obtain the required thickness, the growth of each layer is controlled by a particular temperature, precursor gas volume, and time.

2.3 Fabrication

Fabrication of semiconducting InGaAs/GaAs nanotubes can be done by two methods. The first method involves deposition and patterning of SiO₂ and subsequent transfer of the pattern on the
epitaxial structure using dry etching techniques. This method is employed for smaller feature sizes where undercutting is an issue. The second method, which was used to fabricate nanotubes for this study, employs traditional photolithography and isotropic wet chemical etching.

The sample obtained after growth was thoroughly degreased using acetone, methanol, and isopropyl alcohol in succession to remove organic and metallic impurities. Lithography and wet chemical etching were performed to define the rectangular strip pattern on the bilayer stack all the way down to the sacrificial layer. After degreasing, the sample was placed on a hot plate for 3 min at 125 °C to drive out any moisture, following which photoresist (AZ 5214E) was spun on for 30 s at the rate of 4500 rpm. Positive lithography was done for 60 s and the sample was developed for 50 s in AZ-327 developer. The sample was inspected for any underdevelopments.

The mesa strips defined by lithography act as etch masks for shallow etching. The concentration of the etchant and etch rates are discussed in the following section. After shallow etching, the photoresist was removed with acetone before selective etching of the sacrificial layer using an HF solution. The bilayer is detached from the substrate and results in a strain relaxation process that leads to spontaneous rolling and formation of a tubular structure. The resultant sample was then rinsed thoroughly in de-ionized (DI) water and soaked in methanol prior to drying the sample on a hot plate. Methanol was used to address the problem of hydrophobicity in nanotubes so that the tubular structure does not collapse on evaporation.

Patterning self-rolled-up nanotubes with metal-assisted chemical etching involved additional steps – such as patterning gold spots prior to sacrificial layer etching and etching the self-rolled-up tubes with an oxidizing agent – to aid catalysis of metal on the bilayer. The preliminary stage of defining the bilayer mesa was done as described above, following which a 1.5 nm layer of
gold was evaporated on the mesa. Lift-off was performed to obtain patterned gold spots on the rectangular mesa. Subsequently, the samples were etched in HF to remove the sacrificial layer and roll up the tubes. The last stage involved etching the sample for 15 s in a dilute solution of potassium permanganate and hydrofluoric acid. The samples were then rinsed thoroughly in deionized water and methanol before drying. This resulted in tubes with patterned holes.

2.4 Etching Solution and Etch Rates

Etch selectivity is the main criterion that decides the choice of the etchant. For most GaAs-based structures, the underlying mechanism in etching involves oxidation of Ga and As on the surface, and removal of these oxides using aqueous solution of acids or bases. More frequently, hydrogen peroxide is used with most acids or bases to assist surface oxide formation in contrast to other oxidizing agents such as nitric acid, or potassium dichromate. Some of the most popular etchants for GaAs-based alloys are aqueous solutions of H$_2$SO$_4$, NH$_4$OH, HCl combined with H$_2$O$_2$ and Br$_2$/methanol. The chemical reactions that occur on the surface are adsorption of reagents, oxide formation, and removal of oxides. The dissolution of the oxide in the etchant determines the etch rate and profile. The concentration of the etchant alters the selectivity and etch rate for different GaAs-based alloys.

Based on previous studies [47, 48], a solution of H$_2$SO$_4$, H$_2$O$_2$, and DI water (in the ratio 1:8:80) was prepared and samples were etched for 23 s to a depth of about 150 nm to define the shallow mesa pattern all the way down to the sacrificial layer. During etching, the sidewalls are exposed and thus the sacrificial layer for lateral etching. The solution etch rate was determined to be 0.5 µm/min and had very low selectivity between the InGaAs, GaAs, and AlGaAs compounds. The sacrificial layer was then laterally etched using HF:DI (1:2), which showed good selectivity
towards Al$_x$Ga$_{1-x}$As for $x > 0.6$. The sample was etched for 5 min in HF solution to completely remove the sacrificial layer. Etching was conducted at room temperature for this study. It is critical to note that altering the temperature can increase or decrease the etch rate of the solution.

Metal assisted chemical etching was used to pattern the self-rolled-up nanotubes. The process involves an oxidation reaction using metal as a catalyst on the surface of the semiconductor and preferential etching of the semiconductor material directly beneath the metal. Thus, metallic patterns can be transferred onto a semiconductor with appropriate choice of oxidizing agent, acid, and suitable metal. In the case of InGaAs/GaAs nanotubes, a dilute solution of potassium permanganate (KMnO$_4$) and HF was used in the ratio KMnO$_4$:HF:H$_2$O = 1:2:10 to etch patterned gold particles on the surface of the tube.

2.5 Characterization Techniques

The topography of the rolled-up semiconductor nanotubes was analyzed by scanning electron microscopy (SEM) in a Hitachi S4800 microscope. The SEM images depicting the tube shape, profile, number of rotations, and diameter were recorded and studied in detail.

2.6 Simulation

Simulations supporting the geometry effect were performed by Brad Derickson and Huan Li in Professor K. Jimmy Hsia’s group from the Department of Mechanical Science and Engineering, University of Illinois. The rolling behavior of nanotubes was simulated using a finite element model (FEM) in ABAQUS v6.5 software program. The bilayer of InGaAs and GaAs was simulated to incorporate and vary properties based on thickness using an eight-node thick shell plate.
The epitaxial strain mismatch between the bilayer was introduced using an in-plane thermal coefficient for the InGaAs layer and varying the temperature during simulation. The concept of symmetry was invoked and only one quadrant of the plate was modeled to reduce the computational time.
2.7 Figures

Figure 2.1 Schematic showing an example of a planar stack structure grown by metal organic chemical vapor deposition (MOCVD) for fabrication of strain-induced semiconductor tubes. The stack contains an In$_{0.3}$Ga$_{0.7}$As strained layer along with a GaAs quantum well (QW) layer grown between Al$_{0.3}$Ga$_{0.7}$As barrier layers on an Al$_{0.75}$Ga$_{0.25}$As sacrificial layer.
Figure 2.2 Mask pattern designed in LASI software consisting of $19 \times 50 \, \mu m$ rectangles oriented 45° to the wafer cleaving edge $<110>$ used for defining rectangular mesas on the planar structure.
CHAPTER III: EFFECT OF MESA GEOMETRY

3.1 Geometry Dependence Plot

For large area fabrication, it is important to ensure uniformity in diameter along the individual tube and throughout the sample in every tube. It was reported that nanotubes rolled along the <100> crystal orientation direction, irrespective of the stripe pattern orientation, because rolling is influenced by the anisotropy of Young’s modulus in A$^3$B$^5$ cubic crystals [13, 29, 49]. For In$_x$Ga$_{1-x}$As-GaAs materials, the crystal is less stiff along <100> directions than along <110> directions. Any variance from the <100> direction would result in a nanotube with a non-uniform diameter. Thus, crystal direction was an important parameter in the formation of strain-induced self-rolling nanotubes.

This thesis presents details on another parameter significant for the ordered large area assembly of nanostructures. The self-rolling process of detached hetero-structures leading to micro- and nano-objects has been demonstrated earlier for various geometries such as helices, spirals, tubes, rings [22, 45, 50]. While a rectangular geometry is proposed for fabricating nanotubes, the effect of stripe dimensions on the tube structure has not been studied.

Nanotubes required for the study were fabricated as described in Section 2.3 of Chapter II of this thesis. Experiments were performed using stripe patterns with lengths and widths in the range of 5–100 µm on samples with diameters between 1–10 µm. The diameter can be precisely predetermined in the epitaxial growth process in the range of a few hundreds of micrometers to several nanometers. The experimental results were systematically studied and plotted on the basis of tube circumference, rolling behavior, and mesa dimensions. Shown in Figure 3.1 are the
2-D and 3-D plots for the rolling behavior as a function of three parameters: the circumference $c$, the length $b$, and the width $a$ of the rectangular stripe pattern.

Based on the trends observed, the rolling behavior can be broadly classified into the following two categories. One, when the circumference ($c$) of the tube is much larger than the width ($c/a > 1$) or the aspect ratio of the mesa rectangle is large ($b/a > 1$), rolling always occurs from the long side ($b$ side). In the 2-D plot, this region is highlighted in yellow and extends along the far right and top portion of the graph.

When the circumference is smaller than or equal to the width ($c/a < 1$), rolling can take place from the long side, short side, or meta-stable, depending on the aspect ratio ($b/a$) of the mesa pattern, as shown in the 2-D plot.

The meta-stable region in the 2-D plot is indicated for patterns that have a very low aspect ratio or a tube circumference that is much smaller than the pattern width. In this region, rolling occurs short side on one end and long side on the other end which produces a deadlock ‘meta-stable’ state where further rolling from either direction is restricted and tubes lose their tendency to roll. The samples obtained in the meta-stable region contained either tubes with mixed transitions or a blend of tubes rolled up at the short and long sides.

In the 3-D plot shown on the right in Figure 3.1, a clear demarcation in the rolling trends was observed by introducing the width of the stripe pattern as the third axis. While long-side rolling seems to be limited to shorter widths, mixed- and short-side rolling occurred for higher values for the same aspect ratio. As the width of the strip pattern increases, a larger area is accessible to the etchant and hence the possibility of short-side or even mixed rolling is fairly intuitive. For
shorter widths, the amount of area etched along the length is greater compared to the width; thus, a larger area exposed to the etchant instigates long-side rolling.

From the plot, it can be derived that rolling largely depends on two factors, the width of the stripe and the circumference of the tube. For tubes where \( c < a \), the width of the pattern decides the rolling behavior, whereas for \( c > a \), rolling occurs generally long side. Very high aspect ratio (b/a) patterns also are found to result in long-side rolled up tubes. A lot depends on the circumference of the tube that is formed as a result of the strain between the bilayer structures. The rolling direction is determined by the amount of strain in the bilayer and the energy released when the bilayer is detached from the substrate. However, it is important to remember that the strain in the bilayer solely governs the final diameter of the tube and, hence, is independent of the stripe geometry or dimensions. Accordingly, rolling in either direction should and will not change the diameter of the resultant tube.

3.2 General Trends: Long-Side, Short-Side and Mixed Rolling

With the rectangular stripes oriented along \(<100>\) (the soft direction), tubes with different lengths and different numbers of rotations were formed. In the soft rolling direction, all four sides of the rectangular mesa are crystallographically equivalent and, hence, have an equal tendency to roll up during etching of the sacrificial layer. Depending on the length, width, and the diameter of the bilayer (and concurrently the built-in strain and thickness), different trends are observed. Figure 3.2, shows the three types of rolling trends obtained. Part (a) shows the predominantly long-side rolled-up tubes fabricated with a \( 10 \times 25 \ \mu m \) stripe pattern on an \( \text{In}_{0.3}\text{Ga}_{0.7}\text{As/GaAs} \) stack. The diameter of these tubes is about \( 3 \mu m \). Shown in part (b) are the short-side rolled-up tubes formed with a \( 19 \times 50 \ \mu m \) rectangular stripe on an \( \text{In}_{0.2}\text{Ga}_{0.8}\text{As/GaAs} \)
stack with a diameter close to 4 µm. Part (c) displays a yield of long-side and short-side rolled-up tubes fabricated using a $13 \times 50$ µm mesa pattern on an $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}/\text{GaAs}$ stack.

3.3 Time Evolution

In order to understand the mechanism involved in the rolling process, a time evolution of rolling behavior was studied. Several samples of the same composition and mesa pattern were fabricated and etched in dilute HF solution for different times, as described in the experimental section earlier. The samples consisted of tubes fabricated using an $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}/\text{GaAs}$ stack with the $10 \times 25$ µm strip to study long-side rolling behavior and an $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}/\text{GaAs}$ stack with $19 \times 50$ µm strip pattern to study the rolling behavior occurring from short side. The dimensions of the rectangular mesa were chosen so that the resultant tubes clearly pertained to the long-side and short-side rolling regime. In the first example, $10 \times 25$ µm gave a $b/a$ ratio of 2.5 and a $c/a$ ratio of 0.98. In the second example, $19 \times 50$ µm gave a $b/a$ ratio of 2.63 and a $c/a$ ratio of 0.66. These two points are indicated on the 2-D plot (Figure 3.1) discussed earlier.

Shown in Figures 3.3 and 3.4 are the snapshots for both long-side and short-side rolled-up behavior in nanotubes at the specified etching time. As discussed earlier, all four sides have an equal tendency to roll up. Initially, after 30 s of etching, all four sides of the bilayer film tend to curve as it starts to partially detach from the substrate during selective etching of the sacrificial layer. This is shown in frame (a) in Figure 3.3 and Figure 3.4. The part of the sacrificial layer that is still restricted to the substrate is the dark rectangular area visible in the centre of the strip. In the case of long-side rolling (Figure 3.3), a stage (b) occurs where the bilayer film tends to curve up from both the short side and the long side.
On further increasing the etching time to 55 s, a stage (c) occurs where long-side rolling supersedes the short side. This provides a useful insight about the behavior of strained thin films and the energy minimization process at the nanoscale level. The transition from stage (b) to stage (c) can be explained on the basis of thermodynamic control. Since the length of the strip is longer than the width, more area is etched along the length than the width at a particular time. This in turn allows better access to the etchant and, thus, the tube assumes the length of the long side at the end of 60 s, as shown in Figure 3.3 (d). It is presumed that the short side must have flattened down in the process of energy minimization. In the case of short-side rolling (Figure 3.4), a similar behavior is observed during initial etching of 30 s when all the four sides tend to curve up (a). Interestingly enough, the etching times in this case are longer because of the larger pattern (19 × 50 μm). Here, rolling from the short side predominates after 120 s of etching, as shown in stage (b), and continues to form multi-turn tubes on each end until they are against each other as the sacrificial layer is etched further for (c) 140 s and (d) 180 s, respectively. As the sacrificial layer strip became narrower, a small tear in the tubular structure (i.e., a part of the bilayer film was still attached to the substrate) was observed in almost all short-side rolled-up tubes.

3.4 Specific Ratio

In the size range studied, an interesting phenomenon occurred in samples with the specific ratio of 2.63 (b/a = 2.63). All the tubes formed with this specific ratio rolled up from the short side. A comparison between samples of three different diameters fabricated with the same mesa dimensions was done. Shown in Figure 3.5 (a, b, and c), are short-side rolled-up tubes fabricated using a 19 × 50 μm pattern on samples with diameters of 4, 7, and 9 μm. For an even comparison, the diameters were varied only by altering the thickness of the barrier layer in the stack and not the built-in strain. All samples consisted of an In_{0.2}Ga_{0.8}As/GaAs bilayer. Figure
3.5 (d, e, and f) shows short-side rolled-up tubes formed using patterns 19 × 50, 29 × 75, 38 × 100 µm on three samples with a diameter of 4 µm. Thus, regardless of the absolute a and b dimensions, and diameter, at this ratio, the patterned membranes resulted in short-side rolled-up nanotubes. The specific aspect ratio for the short side observed in Figure 3.5 does not have a significant physical meaning, other than the fact that it seems to lead to an intermediate state that favors short-side rolling. Although a definite hypothesis of this phenomenon could not be proposed, the observation could be useful for practical applications.

However, it is important to remember that the strain in the bilayer solely governs the final diameter of the tube and, hence, is independent of the stripe geometry or dimensions. Accordingly, rolling in either direction should and will not change the diameter of the resultant tube.

3.5 Comparison with Simulated Results

To support these experimental results, collaboration with Professor Hsia’s group was initiated to simulate the rolling behavior of nanotubes. Based on the finite element model created, a comparison between the energy states associated with the two rolling directions was performed. Figure 3.6 shows the energy states associated with three kinds of rolling behavior. It is calculated that rolling along the long side has the lowest energy state and is preferable, because built-in strain in the bilayer is most relaxed in this case. Note that for the simulations, the driving force for rolling is analogous to the release of the bilayer with time. Promisingly, the enlarged view in Figure 3.6 (b) shows energy states during initial time and indeed is consistent with the experimental results in that mixed rolling is the lowest energy state. This is in good agreement with our experimental results in the time evolution study, where both long and short side rolling
begin with all four sides curving up (Figure 3.3). Figure 3.7 shows the rolling behavior for two mesa patterns (a) using the $19 \times 50 \, \mu\text{m}$ pattern and (b) using the $4 \times 25 \, \mu\text{m}$ pattern etched to 40% of the total area. While it is true that rolling begins to occur from both sides for either geometry, the extent of etching differs along the length and width of the mesa, which ultimately decides the rolling direction. The simulation and experimental results shown here imply that the rolling history could very well drive the rolling along a different direction depending on the intermediate state of the deformation. In Figure 3.7 (a), it is seen that the extent of etching along the width of the mesa is greater than along the length. The edge along with the width is lifted completely, whereas a part of the length and corner still remain pinned, which naturally leads to short side rolling behavior.

By contrast, Figure 3.7 (b) shows an almost opposite rolling behavior in that the corner and length are etched more than the width of the mesa, which leads to a long-side rolled-up tube. These simulations again support experimental data that indicate short-side rolling for $19 \times 50 \, \mu\text{m}$ and long-side rolling for $4 \times 25 \, \mu\text{m}$ strip dimensions as plotted in earlier Figure 3.1.

3.6 Forced Rolling

This study is noteworthy because it can be used to predict the rolling behavior in the design stages prior to on-site fabrication. While this is true, at certain times it is necessary to defeat the naturally occurring thermodynamic energy release process to achieve specific shape, length, and tube orientation. In this case, the nanotube structure can be engineered to roll up short or long side by introducing etch holes on the mesa pattern to induce a fraction of anisotropy. By introducing etch holes, the concept of kinetics comes into play. During etching, the areas surrounding the holes are etched faster and provide better passage to the etchant, thereby creating
an asymmetry in etching between the penetrable (with holes) and sealed areas (without holes). Shown in Figure 3.8 (a) is one such example where the rectangular mesa dimensions yield a short-side rolled-up tube. By introducing an array of uniform holes on one side of the rectangular stripe, the tube rolls up along the length as shown in Figure 3.8 (b). Note that the patterning of the rolled-up bilayer does not affect the diameter. It would be counter-intuitive that the introduction of holes had little or no effect on the internal strain in the bilayer or cause atypical, non-uniform rolling. However, this can be explained on the basis of discrete and uniform strain distribution. It is for the same reason that the introduction of quantum wells or quantum dots uniformly in the microtube wall does not cause uneven rolling or strain. The idea is to introduce an asymmetry in etching, which causes rolling to dominate in one direction. While holes were used to demonstrate this idea, other uniform geometries such as squares, rectangles, and ellipses can also be implemented. Conversely, a restrain can be used on the rectangular mesa that restricts etching in that direction. As a result, the etchant tries to circumvent the restrain and etches the bilayer mesa from an accessible region. Figure 3.8 (c) shows, for instance, how a circular area on the rectangular mesa evaporated with a thin layer (5 nm) of metallic gold, holds down the film in that direction. The tube rolls up from the opposite end, as shown in the figure.

3.7 Patterning Tubes using Metal-Assisted Chemical Etching

Metal-assisted chemical etching [51-53] is a contemporary top-down and scalable approach for fabricating three-dimensional nanostructures. Using this method, nanotubes can be patterned to form functional structures that can be used in plasmonic enhancement, photonic device crystals, DBR (distributed bragg reflectors) lasers. It has been demonstrated earlier [16, 34, 36] that geometry can play an active role in varying the optical properties of the nanotube.
Modifying the edge geometry to create a bottle-like triangular notch or to etch periodic structures such as DBR gratings on the mesa enhances the optical confinement in nanotubes. Moreover, nanotubes with a periodic array of holes can be designed to be used in 3-D photonic crystals. Although this can be done using traditional lithographic techniques as described in the previous section, the resolution and scalability becomes a limiting factor. With metal-assisted chemical etching, tubes can be patterned in the nanoscale range. During metal-assisted chemical etching, the metal on the semiconductor surface acts as a local cathode and a catalyst to promote the oxidation reaction [54, 55]. The etch rate depends directly on the rate at which the oxidant (KMnO$_4$) can generate holes under the metal particle and indirectly the concentration of the solution. Shown in Figure 3.9 (a), is an SEM image of a 30% InGaAs QW semi-porous long-side rolled-up tube fabricated using metal-assisted etching. Gold (Au) nanoparticle islands obtained by evaporation of 1.5 nm of Au on the rolled-up tube are etched in a solution of HF:KMnO$_4$:DI (2:1:10) for 10 s. The detailed fabrication procedure has been described in Section 2.3 of this thesis. Figure 3.9 (b) presents a magnified view of the surface of the nanotube. It is seen that the etching only occurred in dense areas where metal nanoparticles were found in clusters. A non-uniform deposition of Au on the surface of the nanotube could be the possible reason for site-selective etching. Subsequently, evaporation was performed before rolling up the tube structure to ensure a uniform distribution on the surface of the rectangular mesa. Shown in Figure 3.10 (b) is an SEM image of a 20% InGaAs QW uniform and porous short-side rolled-up tube after metal-assisted chemical etching for 15 s in HF:KMnO$_4$:DI (2:1:10) solution. In comparison with Figure 3.10 (a), the tube appears to be collapsed due to the porosity.

The potential of metal-assisted chemical etching to create nanostructures extends far from the preliminary results demonstrated in this work. The versatility in the process allows the formation
of various patterned nanostructures merely by controlling the etching concentration, etching time, and type of metal used. Such a process could be used to pattern nanotubes with ordered periodic arrays of holes to design photonic crystal cavity structures, which could be future extensions of this project.
3.8 Figures

Figure 3.1 (a) 2-dimensional and (b) 3-dimensional plots summarizing rolling behavior as a function of circumference of the tube (c), width (a), and length (b) of the rectangular strip pattern. Each unique rolling behavior is color coded (blue: long side, red: short side, green: mixed) on the plot. Patterns with very high aspect ratios (far right) always roll long side as shown in the yellow highlighted region of the 2-D plot. Rolling depends on the tube diameter for smaller aspect ratios, as indicated in the bottom left of the 2-D plot. For the same aspect ratio (3-D plot), long side rolling seems to be limited to shorter widths, and mixed and short side rolling are more likely to occur for bigger stripes.
Figure 3.2 SEM images showing the three kinds of rolling behavior observed in self-rolling semiconductor tubes. (a) 30% InGaAs/GaAs QW long-side rolled-up tubes 25µm in length and 3µm diameter fabricated using a 10 × 25 µm pattern, (b) 20% InGaAs/GaAs QW short-side rolled-up tubes 19µm in length and 4µm diameter fabricated using a 19 × 50 µm pattern, and (c) 30% InGaAs/GaAs QW mixed rolling behavior obtained using a 13 × 50 µm pattern.
Figure 3.3 SEM images of 30% InGaAs/GaAs QW long-side rolled-up tubes 25µm in length and 3µm diameter fabricated using a 10 × 25 µm mesa pattern, formed after sacrificial Al$_{0.75}$Ga$_{0.25}$As layer etching for (a) 30 s, (b) 45 s, (c) 55 s and (d) 60 s in 1:2 HF:DI solution. As the bilayer is released from the substrate, the bilayer lifts up in both directions. Increasing etch times shows preferential long-side rolling.
Figure 3.4 SEM images of 20\% InGaAs/GaAs QW short-side rolled-up tubes 19\mu m in length and 4\mu m diameter fabricated using a 19 \times 50 \mu m mesa pattern, formed after sacrificial Al_{0.75}Ga_{0.25}As layer etching for (a) 30 s, (b) 120 s, (c) 140 s and (d) 180 s in 1:2 HF:DI solution. As the bilayer is released from the substrate, the bilayer lifts up in both directions. Increasing etch times shows preferential short-side rolling. Note that a tear developed during the rolling process.
Figure 3.5 SEM images of short-side rolled-up tubes obtained using a 19 × 50 μm pattern on 20% InGaAs/GaAs QW samples with diameter (a) 4 μm, (b) 7 μm and (c) 9 μm after sacrificial layer etching for 3 mins in 1:2 HF:DI solution. SEM images of short-side rolled-up tubes using (d) 19 × 50 μm, (e) 28 × 75 μm and (f) 38 × 100 μm patterns on samples with diameter of 4 μm.
Figure 3.6 (a) Graph showing the simulation results of the energy state of a strained membrane, where the x-axis is the driving force for rolling and the y axis is the total energy of the geometry. Long-side rolling is the lowest energy state at equilibrium condition. (b) Enlargement of area shown in (a), showing the relationship between strain energy and rolling force during initial etching time. Mixed rolling is the lowest energy state initially.
Figure 3.7 Simulation results showing a comparison between etching behavior of two different patterns. (a) 19 × 50 μm pattern, showing the corner and a small area along the length are pinned compared to the width. (b) 4 × 25 μm pattern, showing the corner is lifted and most of the length is etched compared to the width. These behaviors reflect opposing rolling directions and are experimentally verified for short- and long-side rolling, respectively.
Figure 3.8 SEM images showing the control of rolling direction using the lithographic patterning technique. Holes patterned on one side of the rectangular mesa cause the tube to roll up in the opposite direction from the natural. (a) SEM image of a 30% InGaAs QW short-side rolled-up tube fabricated using a $19 \times 50 \, \mu\text{m}$ pattern after sacrificial layer etching for 3 min in dilute HF solution. (b) SEM image of long-side rolled up 30% InGaAs QW tube fabricated using a $19 \times 50 \, \mu\text{m}$ pattern with etch holes on one side along the length. (c) SEM image showing forced short-side rolling behavior of a 30% InGaAs QW sample when a 5 nm spot of Au is evaporated on top of the rectangular mesa before etching the sacrificial layer in HF solution to roll up the tube.
Figure 3.9 (a) SEM images of 30% InGaAs QW semi-porous long-side rolled-up tube fabricated using metal-assisted etching. Gold (Au) nanoparticle islands obtained by evaporation of 1.5 nm of Au on the rolled-up tube are etched in a solution of HF:KMnO$_4$:DI (2:1:10) for 15 s. (b) Magnified view of (a) showing porosity in regions where Au nanoparticles are dense.
Figure 3.10 (a) SEM image of 20% InGaAs QW short-side rolled-up tube fabricated using $19 \times 50 \ \mu m$ pattern before metal-assisted etching. Gold (Au) layer of $1.5 \ \text{nm}$ is evaporated on the rectangular mesa before sacrificial layer etching in HF solution to roll up the tube. (b) SEM image of a 20% InGaAs QW uniformly porous short-side rolled-up tube after metal-assisted etching for 15 s in HF:K$\text{MnO}_4$:DI (2:1:10) solution.
CHAPTER IV: CONCLUSION

4.1 Summary

In conclusion, yet another parameter significant for ordered large area assembly of self-rolled-up semiconductor tubes, i.e., the effect of the geometry of bilayer mesa on the tube shape, has been discussed in detail. The rolling direction is a result of an energy minimization process that the planar film undergoes at equilibrium condition when it is detached from the substrate. The dimension of the stripe pattern plays an important role because it determines the extent of anisotropic etching of the bilayer. Depending on the extent of anisotropy in etching, the energy associated with the release differs along the length and width of the rectangular strip; thus, a different rolling behavior is observed.

The rolling behavior can be broadly classified into two categories: tubes that roll up along the length of the mesa and short-side rolled-up tubes. Usually, patterns with a high aspect ratio tend to roll up long side, while patterns with comparable widths and lengths exhibit a different rolling behavior, depending on the circumference of the tube. Simulation results of strained bilayers for various geometries demonstrated that rolling along the long side is the lowest energy state, probably because the built-in strain in the bilayer is most relaxed in this state. The simulated and experimental results consistently establish the fact that rolling begins from all four sides of the mesa despite the geometry and later takes over either direction, depending on the anisotropy in etching. By means of this analysis, the factors affecting the rolling behavior can be carefully controlled to ensure uniformity in tubes for large area fabrication.

This thesis also provides insights on engineering tube geometry through forced rolling or patterning. The characteristic rolling behavior of semiconductor tubes can be remodeled by
introducing etch holes or through metal-assisted chemical etching. Indeed, it is critical to be able to conceive and control the rolling direction in order to design tubes for a specific length and number of rotations.

4.2 Potential Application and Future Directions

Nanotechnology has the potential to create many new structures and devices with a vast range of applications in medicine, electronics, and energy production. An emerging class of 3-D nanostructures and objects can be fabricated by strain-driven engineering: a combinatorial approach that uses top-down and bottom-up fabrication methods to form self-rolling structures. One such example is semiconductor tubes, which can be embedded with versatile hetero-junctions and active materials in the tube wall that make them unique in the world of nanostructures. By providing an additional third dimension, the out-of-plane curvature, the functionality of microtube optics extends beyond the conventional planar structures.

This additional dimension helps to control the internal strain in the system, thereby intrinsically modifying the optical properties of the microtube. Much can be done in this area by designing the tube geometry to produce low-threshold optical devices such as resonators or lasers. By including a metal layer in the tube wall, the concept of plasmonics can be introduced to design a wide range of possible plasmonic-based devices and low detection limit sensing applications. Biological materials such as DNA or chromosomes can be embedded inside the tube wall and can be used as drug delivery vehicles or for cancer detection. By patterning semiconductor tubes with a periodic array of holes, the possibility they can be used as photonic crystals can be explored.
REFERENCES


