Indium Nitride Nanostructures Prepared by Various Growth Techniques

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ABSTRACT

In the last few years the interest in the material properties of Indium Nitride (InN) semiconductor has been remarkable. There have been significant improvements in the properties and growth methods of InN nanowires (NWs). High quality single crystalline InN NWs with a high-growth rate are regularly obtained. InN NWs exhibit a highly conducting quasi-two-dimensional electron gas (2DEG) on their surface, which causes nearly metallic conductivity even at low temperatures. The newly verified narrow bandgap (~0.69 eV) of InN extends the spectral range covered by III-nitrides to near-infrared, which offers a great advantage of nitrides for optoelectronic applications. In this article, the work accomplished in the InN NW research has been reviewed from the evolution. The growth, characterization and recent developments in InN NW research has been focused mainly. The most popular growth techniques, Metal-organic chemical vapor deposition (MOCVD) and Molecular beam epitaxy (MBE) were discussed in detail. Important phenomena in the growth of InN NWs, as well as the problems remaining for future study, were also addressed.

Keywords: Metalorganic deposition; Nanostructures; Optical properties; X-ray diffraction.

1. INTRODUCTION

Semiconductor nanowires deserve over-growing attention and increasing interest of the scientific community both because of their potential to build future electronic systems and because of intriguing new physical effects, which can lead to new functionalities (Taniyasu *et al.* 2006; Ariyawansa *et al.* 2006; Adachi *et al.* 2010; Zhao *et al.* 2011; Babichev *et al.* 2013; Adachi *et al.* 2014; Pust *et al.* 2015). Nanomaterials are the basic building blocks for nano-devices (Li *et al.* 2006; Li *et al.* 2012; Yu *et al.* 2013; Le *et al.* 2014). Therefore, research about nanomaterials is important for both fundamental physics and device applications (Li *et al.* 2006; Li *et al.* 2012; Yu *et al.* 2013; Le *et al.* 2014). Due to the distinctive properties of nanostructures, various kinds of InN nanostructures have been synthesized, such as nanowires (Yu *et al.* 2015), nanotubes (Wang *et al.* 2015), nanorods (Jiang *et al.* 2009), and nanotips (Schley *et al.* 2007) by solvothermal (Sakalauskas *et al.* 2010), chemical vapor deposition (CVD) (Xu *et al.* 2010; Huang *et al.* 2010; Lei *et al.* 2012), metalorganic chemical vapor deposition (MOCVD) (Chen *et al.* 2009), localized laser-assisted metal-organic vapor phase epitaxy (Kim *et al.* 2019), chemical beam epitaxy (CBE) (Chao *et al.* 2006),

molecular beam epitaxy (MBE) (Richter *et al.* 2009) and plasma-assisted molecular beam epitaxy (PAMBE) (O'Leary *et al.* 1998). Although many methods have been employed to synthesize one-dimensional (1D) InN nanostructures, these methods have their limitations and drawbacks. InN, however, is not all that easy, even considering the general difficulties encountered in the nitride semiconductor system to synthesize. The somewhat intractable problem with InN is the enormous difference in the ionic size of its constituent atoms; the atomic radii for In and N are largely different, which leads to highly distorted interatomic distances, interatomic bonding charges, tendency to form metallic clusters of the group III constituent and inhomogeneous strain.

2. PROPERTIES OF InN

Indium nitride is a very important III-nitride semiconductor material with many potential applications. The recent publications have shown that the blue light-emitting diodes (LEDs), photodetectors, laser diodes (LDs) and high electron mobility transistors proved the benefits of nitride material system (Taniyasu *et al.* 2006; Li *et al.* 2006; Ariyawansa *et al.* 2006;

Adachi *et al.* 2010; Zhao *et al.* 2011; Li *et al.* 2012; Babichev *et al.* 2013; Adachi *et al.* 2014; Le *et al.* 2014; Pust *et al.* 2015). Moreover, due to the large piezoelectricity of III-nitrides, these materials were considered potential candidates for flexible optoelectronic devices (Yu *et al.* 2013; Wang *et al.* 2015; Yu *et al.* 2015). Additionally, III-nitride materials are direct bandgap semiconductors with a wide bandgap range (0.68 – 6.2 eV) (Schley *et al.* 2007; Jiang *et al.* 2009; Sakalauskas *et al.* 2010). The In GaN quantum wells are very much essential for light-emitting devices because the addition of a small amount of indium in the active GaN layer greatly increases the luminesce efficiency. The ensemble Monte Carlo method reveals that InN exhibits an extremely high drift velocity at room temperature (O'Leary *et al.* 1998; Bellotti *et al.* 1999; Foutz *et al.* 1999). It was found that the transport characteristics of InN are superior to those of GaN and GaAs, over a large range of temperatures from 150 to 500 K. It was identified that InN-based field-effect transistors (FETs) have extremely high speeds with a cut-off frequency of over 1 THz for 0.1 μm gates. Thus, InN proves to be a highly potential material for the fabrication of high-speed, high-performance, hetero-junction FETs.

The most commonly mentioned band gap value for InN is 1.89 eV at room temperature (Tansley *et al.* 1986). The LEDs, laser diodes (LDs), and transistors commonly involve InGaN, with low infractions. However, incorporating a large fraction of In into InGaN leads to great advantages over various applications. The use of InN-based optoelectronic devices extends the potential of an environmental-friendly red emitter with no toxic element, and it may replace GaAs-based devices. In addition to that InN is a promising material for low-cost solar cells with high efficiency. In optimum fabrication conditions, the combination of InN and Si with band gap energies 1.9 eV and 1.1 eV leads to obtain a conversion efficiency of over 30%. The InN/Si solar cell material contains no poisonous materials such as arsenic, and it does not need phosphine during the fabrication process. Qiann *et al.* reported that InN is a good plasma filter material for the largely used GaSb and GaInAsSb photovoltaic cells in the thermophotovoltaic system (Qian *et al.* 2002). Several groups studied the photoluminescence property of single-crystalline InN and reported that the bandgap energy of InN is lower than 1.0 eV (Matsuoka *et al.* 2002; Wu *et al.* 2002; Johnson *et al.* 2004; Grandal *et al.* 2005). The newly reported small band gap values are compatible with the wavelength of the optical fiber. Despite the careful scientific effort, one of the basic properties of InN - the bandgap, is still under debate. Based on many studies reported it was concluded that the optical properties of InN are very much dependent on the deposition method and the intrinsic doping concentration.

3. DEPOSITION TECHNIQUES OF InN NANOWIRES

Unfortunately, high-quality InN is very difficult to synthesize. The growth process involves very low decomposition temperature (~630 °C) and high equilibrium vapor pressure of nitrogen. The growth of nanowires generally occurs above the eutectic temperature of the metal catalyst and the semiconducting material. There are many reports on InN films and especially on one-dimensional InN nanostructures. In recent years, the number of reports concerning InN research has increased significantly. Vapor-solid growth of InN nanowires using a mixture of In metal/In_2O_3 powder and ammonia was reported by Zhang *et al.* 2002. Other InN nanostructure synthesis methods use a Solvo-thermal method, halide chemical vapor deposition (CVD) using single-source precursors $N_3In[(CH_2)_3NMe_2]_2$ and amonolysis of indium oxide. Among many other growth techniques, metalorganic vapor phase epitaxy (MOVPE), Metalorganic chemical vapor deposition (MOCVD), and molecular beam epitaxy are the most popular growth techniques. The VLS mechanism has proven to be highly flexible and allows for the controlled growth of complex nanostructures. Nanowires generally grow in the crystal direction that minimizes the total free energy, which, in many cases, is dominated by the surface free energy of the interface between the semiconductor and metal catalyst.

Guosheng Cheng *et al.* 2005 fabricated indium nitride nanowires by a catalyst-free method. They have synthesized InN nanowires at a large scale in a quartz tube containing the reagents of indium and indium oxide powders placed in a central part of the hot wall chemical wall deposition (CVD) chamber. Argon gas was initially flown at a constant flow rate of 1-atmosphere base pressure, and then the tube was heated up to 700 °C. Upon reaching the temperature of 700 °C, 100 sccm (standard cubic centimeter per minute) ammonia was introduced and allowed to flow for 30 minutes continuously. Then the furnace was allowed to cool down naturally at room temperature in the ammonia atmosphere.

Li *et al.* 2016 focused upon InN nanowire growth on metal substrates. Due to the vast application of InN nanowires on electronic devices, near-infrared optoelectronics, high-efficiency solar cells and

nanogenerators, growing InN nanowires on a cheap metal substrate is very attractive; also, this will create a path for various low-cost InN based opto-electronic devices. In this work, the authors designed a homemade metal-organic chemical vapor deposition (MOCVD) system to grow InN nanowires. The furnace was kept in atmospheric pressure during the entire growth process and brass foils were used as substrates. Trimethylindium and ammonia were used as precursors, and the flow rates were maintained at 14 µmol/min and 3 SLM, respectively. The authors were able to grow high-density InN nanowires on brass substrates *via* MOCVD method.

Chen *et al.* 2012 prepared the InN epitaxial films and nanorods on GaN template by radio-frequency metalorganic molecular beam epitaxy (RF-MOMBE) method. The authors claim that the growth rate of RF-MOMBE was much higher than the conventional Molecular Beam Epitaxy method; moreover, it was capable of producing high-quality InN nano-rods. They prepared the InN films/nanorods on GaN deposited c-plane sapphire substrate by MOMBE system with radio frequency (RF) source to activate the nitrogen. Initially, the authors grew 4 µm thick GaN template using a commercially available MOCVD system. A molecular turbo pump was used to evacuate the growth chamber with a base pressure of 1×10^{-9} torr. During the InN nano-rod growth, the flow rate of N_2 was fixed at 1 sccm and V/II ratio was adjusted to 0.4 sccm. The thermal cleaning was carried out by heating the substrate for 600 °C and then cooled down to 500 °C for nitradation. The authors used a mass flow controller to control the V/III flow rate.

Lei *et al.* 2012 synthesized InN nanowires in a vertical tubular furnace with straight alumina tube. They used high-purity InN powders and 4H–SiC single crystals as source material and substrates, respectively. In their experiment, 4H–SiC substrates were first eroded by NaOH at 400 °C for 10 min. Then, the eroded substrates and InN powders were put into the furnace. The remaining air molecules were removed by flushing Ar three times into the furnace. After flushing Ar into the furnace, the mixed atmosphere of NH_3 and N_2 was maintained at 1400 °C temperature for 1 hour. Finally, the furnace was cooled to room temperature. The authors could observe gray color products on the 4H–SiC substrates. The authors observed that the nanowire growth process followed the self-catalytic vapor-liquid-solid (VLS) mechanism. In the VLS method, the InN nanoparticles acted as catalysts for the 1D growth process.

Song *et al.* 2019 synthesized nanotubes and nanobelts by Chemical vapor deposition (CVD) method. The authors reported various InN nanostructures on silicon (100) substrate using CVD method. During the growth process, they varied the temperature in a very small range between 700 to 735 °C. The morphological studies of the as-grown nanotubes and nanobelts confirmed the wurtzite crystallographic structure with the preferred orientation along [0001]. Room temperature photoluminescence (RT-PL) spectrograph revealed a blue shift. The authors concluded that temperature was a very sensitive factor that affected the morphological evolution of the as-prepared InN and other nitride nanostructures.

Stress-free InN nanowires were grown on graphene substrates using the sublimation method by Chen *et al.* 2018. The authors used In-metal sublimation CVD method at normal atmospheric pressure. They adopted this method due to its low-cost and effective growth rate. In this process, Indium was evaporated and allowed to pass through a small gap; then it was allowed to deposit on an inverted substrate in NH_3 atmosphere. The authors fabricated InN nanowires on graphene/GaN/sapphire, Au/GaN/sapphire and Au/graphene/GaN/sapphire substrates. In this paper, the authors confirmed that the growth of InN nanowires was assisted by Au catalyst and that it was free from stress by introducing graphene interlayer. This graphene layer was prepared on GaN/Sapphire substrate and heated at 120 °C for 15 minutes to improve the bonding between the substrate and graphene.

Dwivedi *et al.* 2017 demonstrated the synthesis of the InN nano-pine array using Oblique Angle Deposition (OAD) technique on Si substrate. They have found that the average length of the as-synthesized nano-pins were ~2 µm. The authors proposed a special technique to synthesis well-aligned InN nanostructures using a customized horizontal quartz tube by OAD method. They mixed high purity Indium Oxide (In_2O_3) powder and Indium (In) powder in a quartz boat in 1:2 wt. ratio. One end of the quartz boat was placed in a hot furnace; in the other end, RCA-cleaned p-type Si <100> substrate was placed at an orientation of 85 °C. After evacuating the quartz tube, they flushed the high-purity nitrogen gas into the tube for 30 min. The hot zone was kept at an evaporation temperature of 920-930 °C with a heating rate of 30°C/min. The temperature of the cold zone was kept at 525-530 °C, a slow heat increase rate of 15 °C/min. The entire growth process was continued for 30 minutes under the flow of high-purity ammonia gas. During the growth process, the furnace pressure was maintained at 0.3 mbar. Finally, the authors achieved catalyst-free high-quality InN nano-pine structures in a horizontal quartz tube by assembling InN nano-pillars of ~50 nm average diameter.

Kumar *et al.* 2014 reported the growth of InN nano-rods by Plasma Assisted Molecular Beam Epitaxy (PAMBE) method. Before the initiation of InN growth, an electron beam-evaporated Au (~2 nm) layer was deposited in n-Si (III) substrates. The base pressure was maintained below 1×10^{-10} mbar. The Si substrate was chemically cleaned, and the deposited Au film was annealed for 600 ℃, and then the substrate temperature was decreased to 500 ℃ to fabricate the nanorods. During the growth process, the nitrogen gas pressure was maintained at 5.8×10^{-5} mbar. The entire nano-rod growth has taken place in 2 hours. The annealed Au film was converted into small dots with an average size range of 15-25 nm. The length of the nano-rods grown in this process was in the range of 200-350 nm with an average base diameter of ~42 nm.

Barick *et al.* 2015 reported the structural and electronic properties of InN nanowire network synthesized by CVD method. The authors have prepared InN nanowires on Au-coated quartz substrate using a vapor-liquid-solid mechanism. They deposited the gold layers for different thicknesses (5, 15 and 52 nm) using four target electron beam evaporators. These gold-plated substrates were placed side-wise on a quartz boat together with high purity indium metal. Mass flow controllers were used to control argon and ammonia gas. The flow rates of argon and ammonia gases were maintained at a rate of 20 sccm and 40 sccm, respectively. Argon gas was allowed to flow through the quartz tube for about 20 minutes before the growth process to remove the unwanted gas residues. Their study revealed the multi-nucleation growth; each site acted as the origin of several nanowire growth, resulting in long and thin InN nanowires. Further, the authors concluded that this type of nanowire growth was possible during a specific growth temperature and gold layer thickness. The authors have observed that the nanowires prepared in this method grow along [11$\bar{2}$0] direction (a-plane) to form a dense nanostructure network.

Zhao *et al.* 2013 reported the Mg-doped growth of InN nanowires. Before the nanowire growth, a thin In seeding layer was deposited. At higher growth temperatures, the In seed layer turns into In droplets, which can activate the formation of Mg-doped InN nanowires. The growth parameters include a nitrogen flow rate of 1 sccm, a nitrogen plasma power of 350 W, an Influx of 6×10^{-8} torr and a substrate temperature of 480 ℃.
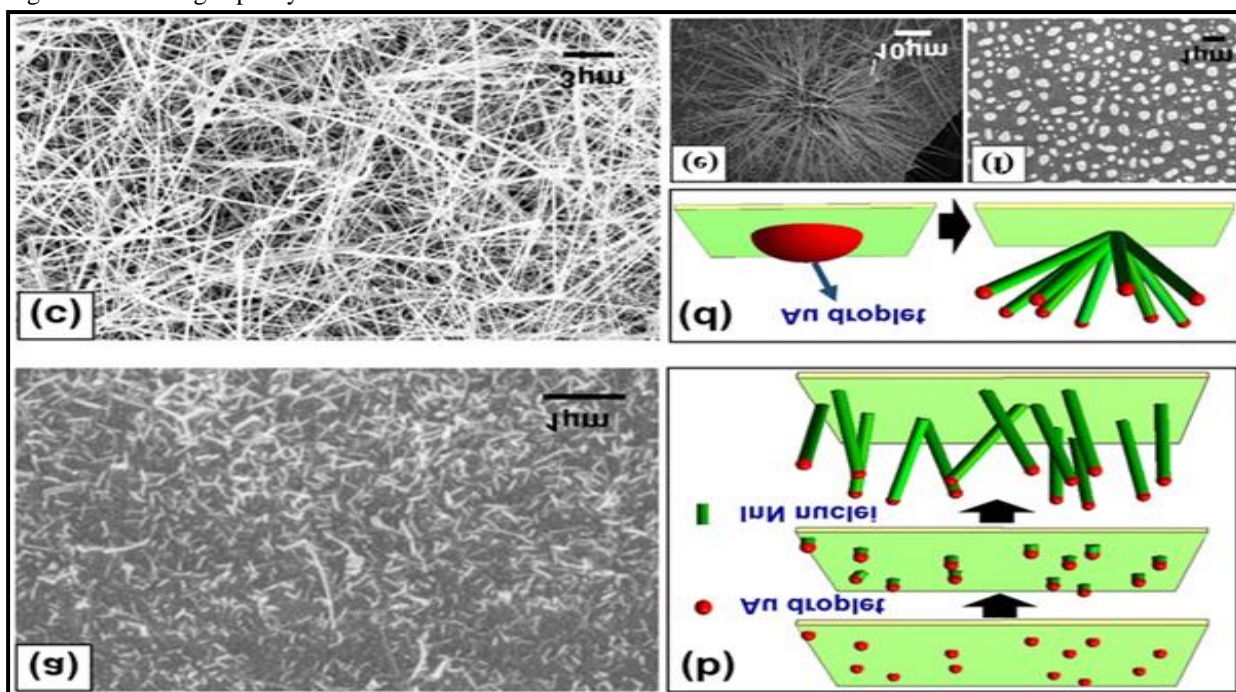

Fig. 1: (a) SEM micrograph of InN grown on 3 nm Au-coated quartz substrate at 550 ℃, (b) schematic diagram of mononuclear growth of nanowires, (c) SEM micrograph of InN grown on 15 nm Au-coated quartz substrate at 550 ℃, (d) schematic diagram of multiple nucleations of nanowires, (e) portion of the SEM micrograph shown in Fig. 1 (c), where multiple nucleations of InN nanowires are visible and (f) SEM image of 15 nm Au-coated Si substrate after annealing at 550 ℃ for 30 minutes (Barick *et al.* 2015)
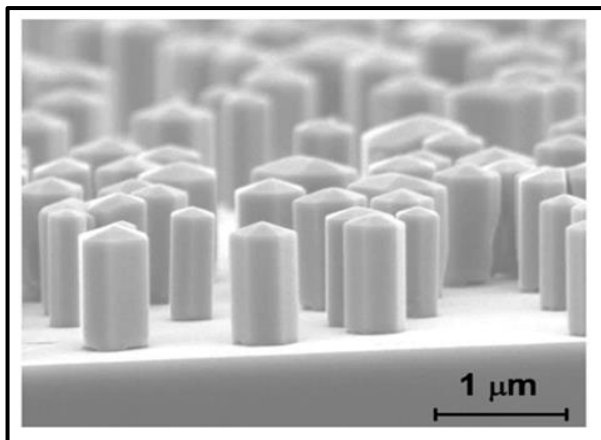
Fig. 2: SEM image of Mg-doped InN nanowires (Zhao *et al.* 2013)

From Fig. 2 revealed the formation of hexagonal-shaped nanowires. Their characterization further revealed that InN nanowires synthesized in this method were free of stacking faults and misfit dislocations.

Teker *et al.* 2017 fabricated the InN nanowires by hot-wall 25 mm Low-Pressure Chemical Vapor Deposition (LPCVD) method. They have used Si and SiO_2/Si substrates along with Ni catalysts during the growth process. They have ultrasonically cleaned the substrate in acetone, isopropyl alcohol and de-ionized water, and finally, the substrates were dried with nitrogen. A quartz boat containing both the substrate and In were loaded into a CVD reactor and heated to target growth temperature. After that, ammonia and hydrogen gases were applied through the reactor for about 80 min. Mass flow controllers controlled the gas flow rates, and at the end, the reactor was cooled down under hydrogen flow to 250 ℃, and then cooled down to room temperature. The authors have observed significant changes in Ni-coated Si substrate and Ni-particle applied SiO_2/Si substrate. They have noticed that the high-density nanowire growth was achieved at 1100 ℃. Moreover, the growth rate was found to be high in Ni film catalyst.

Winden *et al.* 2013 reported the site-controlled growth of InN nanostructures using metalorganic vapor phase epitaxy (MOVPE). The authors explained how to obtain selective area growth of InN nanostructures on patterned SiO_2/GaN(0001)/c-plane $\alpha - Al_2O_3$ substrates. The authors have used a reactor equipped with *in situ* optical sensors for temperature determination. GaN layers on c-plane sapphire substrates were masked using SiO_2. Standard electron beam lithography and Reactive ion etching (RIE) were used, which revealed a hexagonally arranged hole array defined on the sapphire substrate. It was evident from the selective area growth

that the mask geometry influences the surface diffusion as well as vapour phase depletion. The authors used trimethylindium (TMIn) and ammonia (NH_3) as the precursors and pure nitrogen (N_2) as the carrier and ambient gas. They maintained a chamber pressure of 400 mbar and the growth temperature was varied between 520-720 ℃. The optimized growth conditions were employed to study the nanostructure evolution. The authors have concluded that pyramidal-shaped nanostructures were obtained. The gradual reduction of growth times has given clear information on how the nanostructures have evolved inside 100 nm mask.

4. CONCLUSION

III-nitride nanostructures have been identified as critical foundations for several nanoscale opto-electronic devices due to their excellent optical and transport properties. InN was the last-studied III-nitride material among the alloy family. Among many other nanostructures, InN stood out as the best in high fabrication efficiency solar cells, terahertz emitters and detectors with greater performance due to its large drift velocity at room temperature. Initial growth of InN was performed using powder or small crystals by the method of interacting indium compound with ammonia, or thermal decomposition of complex compounds containing In and N. From the review of InN nanostructures synthesized by different methods, we could observe that InN NWs fabricated using a catalyst-free molecular beam epitaxy method or a catalyst-assisted CVD process have a high crystalline quality. The deep knowledge acquired in this field thus will pave way for confidently addressing the challenge of growing very complex heterostructures for future nanodevices.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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