**Fabrication of In$_x$Ga$_{1-x}$N Nanowires on Tantalum Substrates by Vapor-Liquid-Solid Chemical Vapor Deposition**

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1. Introduction

In$_x$Ga$_{1-x}$N alloys are important optoelectronic materials, which have been widely used in light emitting diodes and laser diodes due to their tunable band-gaps [1]. In$_x$Ga$_{1-x}$N alloys are also very promising for energy conversion applications such as solar water splitting [2], photocatalytic reduction of CO$_2$ [3], lithium ion batteries [4], and supercapacitors [5]. In$_x$Ga$_{1-x}$N nanowires (NWs), especially, have drawn great attention due to their large surface area and improved carrier collection efficiency. A variety of In$_x$Ga$_{1-x}$N NW devices have been prepared and exhibited excellent performances [6–10]. Another advantage of In$_x$Ga$_{1-x}$N NWs is that they can accommodate lateral lattice mismatch with the substrate and, therefore, suppress the formation of threading dislocations. Other than conventional substrates such as sapphire, SiC, and Si, In$_x$Ga$_{1-x}$N NWs have been successfully grown on Ti [11,12], Mo [13], Ta [14], Hf [15], Ni [16], copper [17], stainless steel [18], carbon paper [4,5], etc. [19]. These new substrates have offered the In$_x$Ga$_{1-x}$N NW devices with better thermal conductivity, electric conductivity, and mechanic flexibility. Most of these growths were implemented by molecular
beam epitaxy (MBE) or metallorganic chemical vapour deposition (MOCVD). Compared to MBE and MOCVD, chemical vapor deposition (CVD) is less expensive, making it more attractive to fabricate large-area devices at a lower cost.

In this article, a vapor-liquid-solid (VLS)-CVD technique was employed to grow In$_x$Ga$_{1-x}$N NWs on a pure tantalum (Ta) substrate. Ta was chosen as a substrate based on the fact that after CVD growth Ta surface would form tantalum oxides, oxynitrides, and tantalum nitrides, such as Ta$_2$O$_5$, TaON, Ta$_3$N$_5$, and Ta$_x$N$_y$ subnitrides. These tantalum oxides or nitrides are either conductive themselves, or have matched energy band structures with In$_x$Ga$_{1-x}$N. Ta$_2$O$_5$, for example, is a potential coating material, catalyst, capacitor, resistor, and optical device itself. Ta$_2$O$_5$ can be synthesized by thermal oxidation or electrochemically anodic oxidation [20,21]. Electrochemical anodic oxidation of Ta has been carried out to obtain a highly ordered dimpled Ta surface as a template for Au nanoparticle arrays [22], which can be used as catalyst arrays for VLS-CVD growth of nanowires. Furthermore, ammonolysis of Ta$_2$O$_5$ can achieve TaON and Ta$_3$N$_5$, both of which are promising visible-light photocatalysts [23,24]. Besides, Ta$_2$O$_5$, TaON, and Ta$_3$N$_5$ all have matched energy band structures with In$_x$Ga$_{1-x}$N [25]. Nitridation of the Ta$_2$O$_5$ film would also result in Ta$_x$N$_y$ subnitrides, which can work as conductive layers between the film and the Ta substrate [26]. Based on the above analysis, we employed both thermal oxidation and electrochemical oxidation to obtain Ta$_2$O$_5$ films on the surfaces of the Ta substrates. After a post-nitridation process, CVD growth of In$_x$Ga$_{1-x}$N NWs was carried out on the surface of the Ta substrates. It was found that In$_x$Ga$_{1-x}$N NWs can only be successfully fabricated on a specifically pretreated tantalum surface when Ni-Au alloys with certain compositions were used as catalysts. The microstructures of the obtained In$_x$Ga$_{1-x}$N NWs/Ta samples were extensively characterized.

2. Materials and Methods

Three surface states of polished Ta (99.95 %) plates were employed as substrates for the NWs growth: native, oxidative, and nitrided. Oxidative state was obtained by the heat-treatment of the polished Ta plates under atmosphere at 500 °C for 4 h. Nitrided Ta surface was achieved by a high voltage anodization followed by a high temperature nitridation. The polished Ta plates were electrochemically anodized in a two-electrode system using a platinum foil (10 × 10 mm) as the counter-electrode. The anodization was operated at 80 V in a stirred solution of concentrated H$_2$SO$_4$ (95–98%), H$_2$O, and HF (48%) in a volume ratio of 9:4:1 for around 5–10 min until the oxide film was peeled off. The residue white films were further removed by sonication in the deionized water. The samples were then nitridated at 950 °C under 1 atm for 2 h with NH$_3$ of 100 standard-state cubic centimetres per minute (sccm).

The treated tantalum plates were then coated with mixed salts of Ni(NO$_3$)$_2$ and HAuCl$_4$·4H$_2$O as catalysts, with a volumetric percentage of the Ni(NO$_3$)$_2$ ethanol solution as 100%, 50%, 33.3%, 20%, and 0%, respectively. The catalysts were specified as Ni, Ni$_{1}$Au$_{1}$, Ni$_{1}$Au$_{2}$, Ni$_{1}$Au$_{4}$, and Au, accordingly. The treated tantalum plates with catalysts were dried at 40 °C in air before CVD growth.

The CVD growth was carried out in a horizontal gliding furnace as shown in Figure 1 to realize fast heating and cooling. A mixture of gallium acetylacetonate (Ga(ACac)$_3$) and indium acetylacetonate (In(ACac)$_3$) (99.99%) were used as the metallic precursors to react with NH$_3$ to produce In$_x$Ga$_{1-x}$N NWs [27]. The furnace has two heating zones, which were set as 400 °C and 800 °C, respectively. When the desired temperatures were attained, the furnace was glided from right to the left to align the edge of zone 1 with the metallic source, which is a mixture of 0.15 g Ga(ACac)$_3$ and 0.06 g In(ACac)$_3$. The center of zone 2 would correspond to the location of the treated Ta plate. H$_2$ (20 sccm) was introduced to reduce Ni catalyst when the temperature was ramping up. Once the desired temperatures were recovered, H$_2$ was turned off and N$_2$ (20 sccm) was used as a carrier gas along an inner quartz tube (Φ30 mm) to transport metallic precursors to react with NH$_3$ (80 sccm), which flowed through a separate inner quartz tube (Φ10 mm) to prevent parasitic reactions. The pressure
was kept as 2000 Pa for 30 min. Upon the completion of the CVD growth, the furnace was glided away, and N\textsubscript{2} and NH\textsubscript{3} were switched to the outer quartz tube (Φ100 mm) to accelerate the cooling process.

Figure 1. Schematic diagram of the CVD growth apparatus.

The morphologies of the obtained samples were examined using a field emission scanning electron microscope (SEM, Zeiss Sigma 500, Netherland). The structures were identified by X-ray diffraction (XRD, Philips, PANalytical X’pert, Netherland, Cu Ka radiation (λ = 1.5417 Å)). The microstructure characterization of the obtained NWs was performed with Transmission Electron Microscope (TEM) using a TEM/STEM system (FEI Talos F200X, Hillsboro, OR, USA) equipped with 2 Super-X SDDs. Low temperature photoluminescence (RT-PL) was performed at 77 K with an excited wavelength of 267 nm (Horiba JobinYvon Fluorlog 3-21, USA) to evaluate the optical properties of the obtained NWs.

3. Results and Discussion

3.1. CVD Growth on the Native and Oxidative Ta Substrates

After the CVD growth, neither native nor oxidative Ta surfaces can lead to the growth of NWs. On the surface of the oxidative Ta substrates, only metallic clusters, polish scratches, and film cracks can be observed in the SEM images as shown by Figure 2a–c. XRD spectra in Figure 2d showed that a crystalline β-Ta\textsubscript{2}O\textsubscript{5} phase formed on the Ta surface, whose peak intensities decreased with increasing substrate temperatures in the order of 700 °C, 725 °C, and 750 °C. The observation indicated that the volatile nature of the Ta\textsubscript{2}O\textsubscript{5} films could be the reason why the NWs growth failed on the native and oxidative Ta substrates. The other possibility could be originated from the reductive reaction between the Ta\textsubscript{2}O\textsubscript{5} film and the hydrogen and ammonia during the high temperature CVD growth.

Figure 2. (a–c) The top-view SEM images and (d) the corresponding XRD spectra of the oxidative Ta samples after CVD growth using a catalyst of Ni\textsubscript{1}Au\textsubscript{1} with the substrate temperatures of (a) 700 °C, (b) 725 °C, and (c) 750 °C.
3.2. CVD Growth on the Nitrided Ta Substrates

The above results indicated that rather than an oxidative Ta surface, a nitrided Ta surface is more likely to be a substrate for the CVD grown In$_x$Ga$_{1-x}$N NWs. Nitridation of the thermally oxidative Ta substrates, however, resulted in a peeled film off the Ta substrate. As a consequence, electrochemical anodic oxidative Ta was used for the post-nitridation process. Short-time anodization of the Ta plates usually produced a thick sealed tubular oxide film with many cracks as shown by the inset of Figure 3a. On the top of the thick tubular oxide film existed a continuous passive film, which has been demonstrated to work as surface recombination centers of the electron-hole pairs and to deteriorate the device properties [28]. To solve these problems, a prolonged anodization process was used instead, which exfoliated the tubular film and exposed the underlying surface. The exposed surface contained a thin continuous oxide layer that exhibited a morphology of regular dimples ~250 nm in diameter (Figure 3a). After nitridation, the dimpled morphology was maintained and no cracks appeared on the surface (Figure 3b). XRD (Figure 3c) suggested the existence of Ta$_3$N$_5$, TaN$_{0.83}$, and TaN$_{0.43}$ on the nitrided Ta surface. The distributions of the phases were drawn schematically in Figure 3d, where it was proposed that the post-nitridation transformed the thin dimpled oxide layer into a thin dimpled Ta$_3$N$_5$ layer, with intermediate layers of TaN$_{0.83}$ and TaN$_{0.43}$ formed between the surface Ta$_3$N$_5$ layer and the Ta substrate.

After VLS-CVD growth, homogeneous distributions of long NWs were achieved only for the catalysts of Ni and Ni$_1$Au$_1$ (Figure 4a–e). SEM images also show that the more the content of Au, the less the density of NWs. The large particles distributed on the surface of the nitrided Ta substrates (Figure 4c–e) were revealed to be Au particles dissolved with a large amount of Ga and In (Figure 4f). It has been reported that Au atoms can detach and migrate along GaN nanowires grown on Si during the plasma-enhanced CVD process [29]. In our case, it seems that Au atoms were dewetting on the nitrided Ta surface and coalesced to form particles, whose sizes were too large to catalyze NWs growth.

![Figure 3.](image-url)
The Ta3N5 intermediate layer should be the main reason for the successful fabrication of nitride NWs on the Ta surface. EDS/STEM mapping in Figure 6d also revealed a core-shell structure for the NW with an InxGa1−xN core around 20–30 nm in diameter and a GaN-shell around 10–20 nm in thickness.

Figure 3. (a) The top-view SEM image of the Ta sample anodized at 80V for 2 min. (b) Cross-section schematic diagram of the nitrided sample from a thin dimple Ta surface after CVD growth using catalysts of (a) Ni, (b) Ni1Au1, (c) Ni1Au2, (d) Ni1Au4, and (e) Au. (f) The EDS spectrum (spectrum 1) from a large particle in the inset which is the top-view SEM image of a nitrided dimpled Ta surface after CVD growth using catalysts of Ni1Au4.

For the catalyst of Ni1Au1, the obtained NWs had rough facets on the sidewalls and droplets on the top (Figure 5a). The later clearly indicated a VLS growth mechanism of the NWs. For the nitrided dimpled Ta surface with NWs, XRD spectrum in Figure 5b clearly revealed the presence of phases of GaN, TaN0.83, TaN0.43, and Ta, while the Ta3N5 phase was obviously missing. When a thick tubular Ta3N5 layer was used as the substrate (Figure 5c), the Ta3N5 phase remained in the XRD spectrum as shown by Figure 5d, indicating a stable Ta3N5 phase under a pressure of 2000 Pa at 800 °C. The absence of Ta3N5 peaks in Figure 5b could thereby be ascribed to the reduction of the thin dimpled Ta3N5 layer by hydrogen during the CVD growth. Although not detected by XRD, a small amount of Ta3N5 should still be present to bond the NWs and the TaN0.83 layer together, as shown schematically by Figure 5e. The survived Ta3N5 could be owing to the protection of the catalyst droplets. The stable Ta3N5 intermediate layer should be the main reason for the successful fabrication of nitride NWs on the Ta surface.

Figure 4. Top-view SEM images of the InxGa1−xN NWs grown on the nitrided Ta surfaces using catalysts of (a) Ni, (b) Ni1Au1, (c) Ni1Au2, (d) Ni1Au4, and (e) Au. (f) The EDS spectrum (spectrum 1) from a large particle in the inset which is the top-view SEM image of a nitrided dimpled Ta surface after CVD growth using catalysts of Ni1Au4.

Figure 5. (a) The enlarged top-view SEM image and (b) XRD spectrum of the nitrided sample from a thin dimpled Ta surface; (c) the top-view SEM image and (d) XRD spectrum of the nitrided Ta sample with a thick tubular oxide film after CVD growth using catalysts of Ni1Au1. (e) The cross-section schematic diagram of the nitrided sample from a thin dimple Ta surface after the CVD growth.
3.3. TEM Characterization of In$_x$Ga$_{1-x}$N NWs on Nitrided Ta Substrates

TEM characterizations were performed on the NWs, which were removed from the Ta substrate by sonication in a methanol solution and collected by the copper grids with a carbon film. Bright-field (BF) TEM images showed that the diameter of the NWs ranges from 50 to 100 nm, and their length is on the order of microns. The converged beam electron diffraction (CBED) patterns viewed along three different directions all verified a wurtzite structure with a NW growth direction of [1100] (Figure 6a–c). According to the element mappings (Figure 6d) and the EDS spectra (Figure 6e,f), neither droplets nor NWs contained Ta atoms. Instead, the droplet consisted of nitrogen and three metallic elements with a relative concentration of 75.8 at.% Ni, 22.5 at.% In, and 1.7 at.% Au (Figure 6e). The ratio of Au in the droplet was much less than the original catalyst solution (Ni1Au1), which is consistent with the SEM observation that Au atoms tend to migrate on the nitrided Ta surface. EDS/STEM mapping in Figure 6d also revealed a core-shell structure for the NW with an In$_x$Ga$_{1-x}$N core around 20–30 nm in diameter and a GaN-shell around 10–20 nm in thickness. Repeated EDS/STEM measurements were conducted on the core regions in different NWs and a typical EDS spectrum was shown in Figure 6f. Quantification results showed that the In$_x$Ga$_{1-x}$N cores had “x” varying between 1.3 to 3.5 at.%. It should be noted that since the In$_x$Ga$_{1-x}$N core was wrapped with a GaN shell, the obtained indium composition “x” is actually an average composition of the NW and could be lower than the true value of the core. High resolution TEM (HRTEM) of the NWs found no dislocations but showed the presence of the basal stacking faults (BSFs) inside the In$_x$Ga$_{1-x}$N cores (Figure 6g).

Low-temperature (LT) PL was employed to characterize the NWs as shown by Figure 7. The sample showed a weak PL shoulder (P1) between 355–362 nm, which could correspond to the neutral donor bound A exciton (D0, XA) recombination (358 nm/3.472 eV) and free A exciton (XA) recombination (357.5 nm/3.477 eV) of the thin GaN shells in the NWs [14,15,30,31]. The second peak P2 is located at 373 nm, which can be attributed to the XA peak from In$_x$Ga$_{1-x}$N core. Based on P2, the band gap energy of the In$_x$Ga$_{1-x}$N cores in the NWs can be estimated as 3.32 eV. The average indium concentration $x$ in the NW cores can be obtained by using equation $E_g = 0.7x + 3.477(1 - x) - bx(1 - x)$, where $E_g$, 0.7, and 3.477 are the bandgap energy of In$_x$Ga$_{1-x}$N, InN, and GaN, respectively, and $b$ represents the bowing parameter [32]. The indium concentration $x$ in the NW cores can therefore be calculated to be 2.4 at.% or 3.8 at.%, depending on the choice of bowing parameter $b$ as 3.6 eV [33] or 1.4 eV [32]. The indium concentration calculated from P2 peak in PL matches quite well with those measured from EDS/STEM. The third peak P3 at 392 nm could arise from two reasons. The first one is the XA peak from In$_x$Ga$_{1-x}$N with higher indium composition, which is vetoed since a thorough TEM examination has been carried out and neither NW nor quantum dots with high indium concentration can be found. Therefore, it is more likely to be true to associate the relatively broad P3 peak with the structural defects such as basal stacking fault in the In$_x$Ga$_{1-x}$N core [14,15,30,31,34], which has been observed in the In$_x$Ga$_{1-x}$N core as shown in Figure 6g.
is the XA peak from In \(_{x}\)Ga \(_{1-x}\) measured from EDS/STEM. The third peak P3 at 392 nm could arise from two reasons. The first one is the XA peak from In\(_{x}\)Ga\(_{1-x}\) with a concentration calculated to be 2.4 at.% or 3.8 at.%, depending on the choice of bowing parameter. The second peak corresponds to the structural defects such as basal stacking faults in the In\(_{x}\)Ga\(_{1-x}\) cores.

Repeated EDS/STEM measurements were conducted on the core regions in different NWs and a typical EDS spectrum was shown in Figure 6f. Quantification results showed that the In\(_{x}\)Ga\(_{1-x}\) concentration can be found. Therefore, it is more likely to be true to associate the relatively broad P3 peak with the structural defects such as basal stacking faults in the In\(_{x}\)Ga\(_{1-x}\) cores.

To summarize, our investigation showed that tantalum oxide is not stable under the CVD growth conditions. Instead, tantalum nitrides were demonstrated to be more suited as a substrate for work as an intermediate layer to bond the In\(_{x}\)Ga\(_{1-x}\) NWs and the substrate. The underlying band gap energy of the In\(_{x}\)Ga\(_{1-x}\) NWs can be estimated as 3.32 eV. The average indium content in the NW cores can be obtained by using equation 1–x) − bx(1 − x), where \(E_g\), 0.7, and 3.477 are the bandgap energy of In\(_{x}\)Ga\(_{1-x}\), InN, and GaN, respectively, and \(b\) represents the bowing parameter [32].

Figure 6. (a–c) BF TEM images and the corresponding CBED patterns (insets) of the In\(_{x}\)Ga\(_{1-x}\)N NWs using the NiAu1 as a catalyst; (d) STEM/EDS element mappings of the region enclosed in Figure 4a; STEM/EDS spectra of the NW from the locations (e,f) in Figure 4d. (g) The HRTEM image and the corresponding SADP pattern viewed along [11\(\overline{2}\)0] for the region enclosed in Figure 4c.

Figure 7. PL measurement at 77 K of In\(_{x}\)Ga\(_{1-x}\)N NWs on a pretreated Ta substrate using Ni as a catalyst.
To summarize, our investigation showed that tantalum oxide is not stable under the CVD growth conditions. Instead, tantalum nitrides were demonstrated to be more suited as a substrate for In$_x$Ga$_{1-x}$N NWs. The nitrided dimpled Ta surface contained a thin continuous Ta$_3$N$_5$ layer, which can work as an intermediate layer to bond the In$_x$Ga$_{1-x}$N NWs and the substrate. The underlying substrate contained TaN$_{0.83}$, TaN$_{0.43}$, and Ta, all of which are conductive. Therefore, in terms of the transport of the photo-generated carriers, Ta should be superior to the conventional Si substrate because of an insulated Si$_x$N$_y$ layer formed on the surface of Si [19]. On a dimpled surface of tantalum nitrides, Au atoms tend to coalesce rather than form nano-droplets to catalyze NW growth. Ni was proved to be the suitable catalyst for the VLS-CVD growth of In$_x$Ga$_{1-x}$N NWs. The obtained core-shell structure for the NWs could be explained by an initial VLS-growth of the In$_x$Ga$_{1-x}$N core and a subsequent VS-growth of the GaN shell, which will be investigated and discussed in the future. Although not affected by the Ta substrate, the composition of In$_x$Ga$_{1-x}$N NWs should be further adjusted by changing the CVD growth parameters, such as temperature and pressure. Once higher indium content can be achieved in In$_x$Ga$_{1-x}$N NWs, a matched energy band structure can be established between the In$_x$Ga$_{1-x}$N NWs and the Ta$_3$N$_5$ surface layer. Furthermore, the BSFs in the m-In$_x$Ga$_{1-x}$N NWs could also help to accelerate the separation of photogenerated hole-electron pairs in the devices such as solar cells and photo-detectors. Therefore, VLS-CVD fabrication of the In$_x$Ga$_{1-x}$N NWs/Ta electrode holds great promise as a large-scale manufacture method for solar cells, photo-detectors, and photo-catalytic devices.

4. Conclusions

In$_x$Ga$_{1-x}$N NWs were successfully grown on Ta substrates via VLS-CVD. A Ta surface pretreatment method was developed by a combination of electrochemical anodization and high temperature nitridation, leading to a dimpled Ta$_3$N$_5$ surface layer with underlying intermediate layers of TaN$_{0.83}$ and TaN$_{0.43}$. Ni rather than Au was demonstrated as the suitable catalyst for VLS-CVD growth of NWs on the Ta templates. The obtained In$_x$Ga$_{1-x}$N NWs grew along a [100] direction and processed an In$_x$Ga$_{1-x}$N core/GaN shell structure with the presence of basal SFs in the In$_x$Ga$_{1-x}$N core.

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