

Dnr 2014-0063 92

Projektnr P40119-1

Energimyndighetens titel på projektet – svenska Utspädda nitridbaserade nanotrådar för effektiva solceller		
Energimyndighetens titel på projektet – engelska Dilute-nitrides-based nanowires for efficient harvesting of solar energy		
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Nanowire, solar cell, dilute nitrides, nanotechnology		

# Förord (Preface)

This research was conducted thanks to the financial support from the Swedish Energy Agency. It was also partly supported from Linköping University.

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## Sammanfattning.

Att utvinna energi från solljus ses just nu som en nödvändig del av framtida energiproduktion. Solceller innehållande nanotrådar har flera möjliga fördelar jämfört med plana motsvarigheter, dels på grund av hur ljuset fångas upp i nanotrådarna vilket maximerar ljusabsorptionen, förbättrade möjligheter att bestämma bandgapet, en lägre grad av strain, en kortare extraktionslängd för laddningsbärarna, en högre tolerans för defekter, samt en lägre materialkonsumtion vilket leder till en lägre kostnad. Solcellseffektiviteten kan ytterligare ökas genom att utnyttja mellanliggande band (intermediate band, IB).

I detta projekt har vi utforskat möjligheterna att använda IB-metoden med nanotrådar av utspädda nitridlegeringar. Då de första nanotrådarna av utspädda nitridmaterial tillverkades mycket nyligen (2012), så har forskningen huvudsakligen fokuserat på materialrelaterade problem. Utfallet av forskningen inkluderar (men är inte begränsat till) att uppnå kontrollerad nanotrådstillverkning på billiga Si-substrat, optimering av tillverkningsparametrar för förbättrad optisk effektivitet, förstå rekombinationsprocesser i nanotrådar, förstå defektformation. Vi har också studerat och förstått den elektroniska strukturen hos utspädda nitridnanotrådar och hur den beror på kvävekoncentration i GaNAs(P)-legeringar och deras kristallstruktur. Dessutom så har försök till optimering av den optimala solcellsdesignen utförts, vilket resulterat i en optimal kärna/multiskal GaNPbaserad nanotrådsstruktur, med reducerade förluster via icke-ljusemitterande rekombinationer och en förbättring i ljusutvinning genom att utnyttja defekter och ljusuppfångning (light trapping) i nanotrådsuppställningar. Dessa resultat bidrar med nya vetenskapliga insikter och kunskap inom processer som styr hur utspädda nitridnanotrådar fungerar som nästa generation av billiga och effektiva IB-solceller.

## Summary

Harvesting energy from sunlight using photovoltaic technology is currently being recognized as an essential component of future global energy production. Nanowire solar cells provide potential advantages over planar counterparts, due to light trapping within nanowire arrays that maximizes light absorption, improved band gap tuning, relaxed strain restrictions, a reduced carrier extraction length, increased defect tolerance, lower material consumption and, therefore, lower costs. The solar cell efficiency could be further increased by utilizing the intermediate band (IB) approach.

In this project we explored the possibility of realizing the IB approach in the nanowire geometry using novel dilute nitride semiconductor alloys. Since the first dilute nitride nanowires were fabricated only most recently (in 2012), the conducted research was largely focused on solving material-related issues. The research outcome of these efforts includes (but is not limited to) achieving controllable nanowire growth on cheap Si substrates, optimization of growth parameters for improved optical efficiency, understanding recombination processes in NWs, understanding defect formation. We have also studied and understood electronic structure of dilute nitride nanowires and its dependence on the nitrogen composition in the GaNAs(P) alloys and their lattice structure. In addition, efforts in developing an optimal solar cell design were undertaken and resulted in development of an optimal core/mutishell GaNP-based NW structures with reduced losses via non-radiative recombination, and improvements in light harvesting efficiency by utilizing defect engineering and light trapping in nanowire arrays. The obtained results provide new scientific insights and knowhow regarding processes that govern performance of nanowires from dilute nitride semiconductors, en route towards a new generation of intermediated band solar cells with low costs and high efficiencies.



Photovoltaics represents an essential component of future global energy production. Wide-spread utilization of photovoltaics requires achieving a higher solar cell efficiency at lower costs. For these purposes, nanowire (NW) solar cells provide potential advantages over planar counterparts [1-4], due to e.g., improved band gap tuning, facilitated strain relaxation, a reduced carrier extraction length, increased defect tolerance, lower material consumption and, therefore, lower costs, etc. For a conventional NW solar cell made of a single semiconductor, however, the aforementioned benefits are not expected to increase the maximum conversion efficiency beyond the Shockley–Queissier limit but relax requirements to approach it.

The solar cell efficiency can be further improved (to up to 62%) using the socalled intermediate band (IB) approach [5], where IB is used as a stepping stone for low energy photons to transfer electrons from the valence band (VB) to the conduction band (CB). The proposals for the IB states include confined energy levels in a quantum dot [6] and, more recently, electronic states in dilute nitride alloys [7]. Dilute nitrides are derived from conventional III-V semiconductors such as (Ga,In)(P,As) by the insertion of N into the group V sublattice. The large electronegativity mismatch between the N atom and the group V anion it replaces leads to profound effects on electronic properties of the resulting alloys and allows widely extended band structure engineering. Unlike conventional III-V alloys, dilute nitrides exhibit a huge bowing in the band gap energy accompanied by many intriguing properties [8]. One of them is splitting of the CB states into two well separated subbands:  $E^+$  and  $E^-$ . The lowest subband is narrow in energy and can act as IB. Energy positions of these bands can be adjusted by choosing proper alloy composition(s) and, in the case of NWs, by lattice engineering. The first proof-of-principle of this approach was obtained using planar GaNAs as an active material [7], though the achieved solar cell efficiency was substantially lower than the theoretical limit, likely due to non-optimized design.

In this project we have suggested to explore the IB approach in solar cells based on dilute nitrides and to optimize and enhance their performance utilizing defect engineering and nanowire architecture. Considering that knowledge of fundamental physical properties of dilute nitride NWs, development and optimization of structural design, and also development of a reliable and reproducible fabrication process were only marginal at the beginning of the project, all these issues were planned to be addressed in the project.

# Genomförande (Implementation)

The program included three inter-connected work packages: solving materialrelated issues with the aim of optimization of material quality (WP1); understanding fundamental physical properties of dilute nitrides in the NW geometry (WP2); and design optimization required to improve efficiency of light absorption and conversion (WPs). Growth optimization was performed in close collaboration with Prof. C. W. Tu (Jacobs School of Engineering at UC at San Diego, USA) and Prof. F. Ishikawa (Ehime University, Japan). All characterization studies were conducted at LiU, Sweden. In addition to the main PI, the team in LiU included Prof. W. M. Chen, Asso. Prof. J. E. Stehr and PhD students S. Filippov (PhD 2016) and M. Jansson (planned PhD in 2019).

Structural quality of the NWs was examined by using high resolution X-ray diffraction (XRD), scanning electron microscopy (SEM), cathodoluminescence and transmission electron microscopy (TEM). Point defects were identified by employing a range of defect-sensitive techniques based on optical and spin resonance spectroscopies like photoluminescence (PL) and optically detected magnetic resonance (ODMR). Optical techniques such as cw- and time-resolved PL and Raman spectroscopies were also employed to evaluate optical quality and mechanisms of carrier recombination. All characterization measurements provided feedback to growth, assisting its optimization.

#### **Resultat (Results)**

Device applications of semiconductor materials require careful optimization of the growth process. This is of crucial importance for dilute nitride semiconductors studied in this project, as nitrogen incorporation can only be achieved at relatively low growth temperatures, i.e. at conditions that facilitate defect formation. Considering that the fabrication of dilute nitrides in the NW geometry was first achieved in 2012 [9], a significant part of the conducted research was devoted to development of a reliable and reproducible fabrication process, i.e. concentrated

within WP1. The main results of this part of the project can be briefly summarized as follows:

- Together with our partners, we have developed controllable growth of dilute nitride NWs based on GaNP, GaNAs and GaNAsP alloys and optimized growth conditions (e.g. growth temperature, and III-V ratio) required to achieve high optical and structural quality. These NWs were grown by self-catalyzed molecular beam epitaxy (MBE) on Si substrates. In all material systems, the NWs form dense arrays, and can even be fabricated as



Fig.1 SEM of GaAs/GaNAs core-shell NWs grown on a patterned Si substrate (Appl. Phys. Lett. 111, 072106 (2017)). Copyright of AIP Publishing

patterned arrays- see figure 1. They can be grown in several geometries including uniform, core/shell and core/shell/shell radial heterostructures – see Figure 2. The achievable range of nitrogen compositions is 0-2.5% for the GaNP alloys, 0.1-2.3% for the GaNAs alloys and 0.12 -0.16% for the GaNAsP alloys. The nitrogen composition can be reliably controlled by varying parameters of RF nitrogen plasma. Statistical measurements performed on individual NWs show only small

variations in the N content both along an individual NW and also between different NWs within the array (e.g.  $[N] = 0.53 \pm 0.3\%$  for the GaNAs NWs and  $[N] = 0.14 \pm 0.02$  for the GaNAsP NWs). (Applied Physics Letters 105, 072107 (2014); Applied Physics Letters 105, 253106 (2014); Journal of Physical Chemistry C, 121, 7047 (2017); Applied Physics Letters 111, 072106 (2017); Nanotechnology 30, 244002 (2019)).



Figure 2. Representative SEM images of arrays of the GaNP NWs (a), GaP/GaNP core/shell NWs (b) and GaP/GaN<sub>y</sub>P<sub>1-y</sub>/GaN<sub>x</sub>P<sub>1-x</sub> core/shell/shell NWs (c), grown on (111) Si substrates. The scale bar displayed in (a)-(c) is 300 nm. (J. E. Stehr et al Nano Lett. 15, 242 (2015)). Copyright 2015 American Chemical Society.

- We have also developed a top-down approach for fabrication of NW/nanopillar arrays using nanosphere lithography combined with inductively coupled plasma reactive ion etching. Using this approach, we have fabricated an array of GaNAs nanodisks-in-GaAs nanopillars from a GaAs/GaNAs multiple quantum well structure. The array had a hexagonal structure with uniform pillars spaced by about 460 nm, as defined by the nanosphere mask. A representative SEM image of such array is shown in Figure 3. The fabricated nanopillars have an excellent optical



Figure 3. (a) SEM image of the GaNAs array. (b) Side-view SEM image of an individual GaNAs nanopillar, where the seven GaNAs nanodisks are shown in red color. The energy profile of the CB edge along the NP axis is indicated on the left side. The scale bars in (a) and (b) denote 400 nm and 100 nm, respectively. (S. L. Chen et al Nat. Commun. 9, 3575 (2018)).



quality, which demonstrates the great potential of this approach for fabrication of photovoltaic devices (Nat. Commun. 9, 3575 (2018)).

- Based on comprehensive optical measurements, we have demonstrated that alloying with nitrogen causes alloy disorder. Amplified by the giant bowing in the bandgap energy, this leads to strong localization of photo-generated carriers at low temperatures and also restricts carrier diffusion. The presence of alloy disorder, however, has also a positive effect as it reduces impacts of structural defects in carrier recombination. This relaxes requirements on structural purity (i.e. presence of structural defects such as rotational twins) of dilute nitride NWs. The impacts of alloy disorder become less severe at room temperature due to activation of photo-generated carriers to extended band states. (Applied Physics Letters 105, 072107 (2014); Scientific Reports 5, article number: 11653 (2015); Journal of Physical Chemistry C, 121, 7047 (2017); Journal of Physical Chemistry C 122, 19212 (2018); Nanotechnology 30, 405703 (2019)).

- By employing the ODMR spectroscopy combined with PL measurements, we have shown that nitrogen incorporation in GaNAs leads to the formation of gallium vacancies acting as non-radiative recombination centers. The defects are concluded to be formed within bulk regions of the nanowire and do not have the decisive effect on their optical efficiency. (Applied Physics Letters 109, 203103 (2016)).

- Based on temperature dependent cw- and transient PL measurements, we have revealed that GaNAs-based and GaNAsP-based NWs are superior to conventional GaAs and GaAsP NWs because of suppressed detrimental surface recombination via in-situ surface nitridation. This is very beneficial for photovoltaic devices based on these materials, as the suppressed surface recombination means reduced recombination losses even without deliberate surface passivation (Scientific Reports 5, article number: 11653 (2015); Journal of Physical Chemistry C, 121, 7047 (2017)).

- Using PL excitation spectroscopy, we have evaluated effects of the shell formation on carrier transport in GaAs/GaNAs core/shell NWs. It is found that the presence of the shell induces localized potential in the core, which at low temperatures hampers transfer of photo-excited carriers between these layers. This knowledge is important for designing photovoltaic structures from radial heterostructures (J. Vacuum Science & Technology B 34, 04J104 (2016)).

<u>Within WP2</u>, we have also performed studies of fundamental physical properties of dilute nitride alloys with the general aim to identify the material system (i.e alloy composition, crystalline structure, with/without deliberately introduced defects) with the best figure of merits for solar energy conversion. The main results of this part of the project can be summarized as follows.

- We have shown that alloying with nitrogen represents a very efficient way to tailor the bandgap energy of GaNAs, GaNP and GaNAsP alloys. For example, the

bandgap energy of the GaNAsP nanowires can be tuned from 1.81 down to 1.68 eV (at 4 K) by incorporating only 0.16% N. This range of energies represents the optimum range for IB solar cells. This is accompanied by a reduction in the temperature dependence of the bandgap energy, which is about 30% within the temperature range of 4.5 - 300K. Moreover, alloying with nitrogen dramatically enhances oscillator strength of optical transitions in GaNP, allowing efficient absorption of solar light even in very thin NWs. Our studies, therefore, confirm that alloying Ga(As,P) with nitrogen represents a viable and attractive approach of bandgap engineering required for future photovoltaic applications.

- The NW architecture allows one to extend band structure engineering beyond quantum confinement effects by utilizing formation of different crystal phases that are thermodynamically unfavorable in bulk materials. It is therefore of crucial importance to understand the influence of variations in the NWs crystal structure on their fundamental physical properties. We have investigated effects of structural polytypism on the optical properties of GaP and GaP/GaNP core/shell NW structures by a correlative investigation on the structural and optical properties of individual NWs. It was found that structural defects, such as rotational twins in zinc blende (ZB) GaNP, have detrimental effects on light emission intensity at low temperatures by promoting non-radiative recombination processes. On the other hand, formation of the wurtzite (WZ) phase does not notably affected the emission intensity neither in GaP nor in the GaNP alloy - see Figure 4. This suggested that zone folding in WZ GaP does not enhance its radiative efficiency, consistent with theoretical predictions. We have also shown that the change in the lattice structure have negligible effects on the bandgap energies of the GaNP alloys, at least within the range of the investigated nitrogen compositions of < 2 %. Both WZ and ZB GaNP are found to have a significantly



Figure 4. (a) TEM image of a GaP/GaN<sub>0.019</sub>P<sub>0.981</sub> NW with a large WZ inclusion. The scale bar is 200 nm. (b) Cathodoluminescence (CL) line-scan image measured at 50 K along the axis of the same NW as shown in (a). The CL emission intensity is displayed as a function of the wavelength and emission position along the line-scan. The linear color scale is given in arbitrary units. (c) The corresponding CL spectra collected from the regions of the NW as indicated by the horizontal dashed lines. (Dobrovolsky et al. Nano Lett. 15, 4052 (2015)). Copyright (2015) American Chemical Society.

higher oscillator strength of optical transitions as compared with that in parental GaP, promising for potential applications of GaNP NWs as efficient nanoscale solar cells. (Nano Letters 15, 4052 (2015)).

- By employing the PL spectroscopy, we have shown that coaxial GaNP NWs grown on Si substrates can harvest infrared light, in spite of the fact that the bandgap energies of GaNP alloys lie within the visible spectral range (i.e. within 540 – 650 nm for the currently achievable [N]< 3%). This energy upconversion can be monitored via anti-Stokes near-band-edge PL from GaNP, visible even from a single NW. The dominant process responsible for this effect is identified as being due to two-step two-photon absorption (TS-TPA) via a deep level lying at about 1.28 eV above the valence band, based on the measured dependences of the anti-Stokes PL on excitation power and wavelength. The formation of the defect participating in the TS-TPA process is concluded to be promoted by nitrogen incorporation. The revealed defect-mediated TS-TPA process can boost efficiency of harvesting solar energy in GaNP NWs, beneficial for applications of this novel material system in third-generation photovoltaic devices. (Small 10, 4403 (2014)).

- Based on comprehensive optical and spin resonance measurements, we have demonstrated that spin-dependent recombination in GaNAs alloy can be employed to increase carrier lifetime in GaNAs-based nanopillars. This can be achieved by employing only 2-3 defects per nanopillar, demonstrating very high efficiency of this process. Our results show that the unconventional spin blockade approach can be utilized in novel defect-engineered NW solar cells with the purpose to spin block the defect-mediated non-radiative shunt paths for improved efficiency.

<u>Within WP3</u>, our efforts were focused on developing an optimum prototype design for IB solar cells with the NW architecture. The main results of these optimization studies are as follows.

- In an effort to significantly increase optical efficiency of GaNP-based nanostructures, we have identified important non-radiative recombination centers in GaNP coaxial NWs grown on Si substrates. A point defect complex, labeled as DD1 and consisting of a P atom with a neighboring partner aligned along a crystallographic <111> axis, was identified by ODMR as a dominant non-radiative recombination center that resides mainly on the surface of the NWs and partly at the hetero-interfaces- see Figure 5. The formation of DD1 was found to be promoted by the presence of nitrogen and can be suppressed by reducing the strain between the core and shell layers, as well as by protecting the optically active shell by an outer passivating shell. Growth modes employed during the NW growth were shown to play a role. Based on these results, we singled out the GaP/GaN<sub>y</sub>P<sub>1-y</sub>/GaN<sub>x</sub>P<sub>1-x</sub> (x<y) core/shell/shell NW structure, where the GaN<sub>y</sub>P<sub>1-y</sub> inner shell with the highest nitrogen content serves as an active light-emitting/absorbing layer, as the optimized and promising design for efficient light emitters and solar cells based on GaNP NWs. (Nano Letters 15, 242 (2015)).



Figure 5. (a) A representative PL spectrum measured at 5K from the GaP/GaNP NW array. (b) A model illustrating a possible configuration of the DD1 defect on the NW surface, taking as an example a  $P_{Ga}$  antisite forming a complex with a partnering nitrogen atom. (c) and (d) ODMR spectra of the GaP/GaNP NWs measured at 4 K (the open circles) at X- band (9.4 GHz) and Q-band (34 GHz), respectively. The solid lines are the simulated spectra of DD1 using the spin-Hamiltonian parameters for the DD1 defect. (Stehr J E et al Nano Lett. 15, 242 (2015)). Copyright 2015 American Chemical Society.

- The efficiency of NW-based solar cells is often limited by light absorption as surface coverage of the active material determines, within a ray optics approximation, the maximum fraction of sunlight that could be absorbed and converted to a photocurrent. We have shown that the fraction of light absorbed by the GaNAs NW/nanopillars can be significantly enhanced via the so-called light trapping, as was also observed in other NW systems [9-12]. This was demonstrated for arrays of GaNAs nanopillars fabricated by ion lithography.

#### **Diskussion (Discussion)**

To summarize, dilute nitrides are a novel addition to the family of III-Vbased NWs with great potential for solar cell applications. A major advantage of alloying with nitrogen is the increased flexibility in the bandgap engineering provided by the bandgap bowing that can be utilized in both intermediate band and multi-junction solar cells. This allows efficiently tuning of the alloy bandgap and, therefore, the spectral range of light absorption by minor addition of nitrogen. In addition, introduction of nitrogen can improve optical properties of the NWs. In the case of GaN(As)P, where the parental N-free material has an indirect bandgap, this improvement is related to the N-induced transition to the quasi-direct bandgap in the N-containing alloys that occurs at very low alloy compositions of only 0.4% and dramatically enhances light absorption. For direct bandgap GaNAs(P)-based NWs, the improvement is caused by suppression of detrimental surface recombination, likely due to the N-induced nitridation of the NW surfaces during the growth.

In all dilute nitride NWs, the presence of nitrogen causes localization effects that are amplified by the giant bowing in the bandgap energy and are the most pronounced at low measurement temperatures. This leads to strong localization of photo-generated carriers and restricts carrier diffusion, i.e. is harmful for solar cell performance. The presence of alloy disorder, however, has also a positive effect as it reduces impacts of structural defects in carrier recombination.

Similar to the case of bulk dilute nitrides, alloying with nitrogen promotes formation of several point defects. Some of the defects, such as  $V_{Ga}$ ,  $Ga_i$  complexes and a complex defect involving a P atom, act as competing NR centres degrading the solar cell efficiency. The defect formation, however, also leads to a number of useful properties. In GaNP NWs, defect-mediated energy upconversion extends the spectral range of energy harvesting by about 0.7 eV. Spin-dependent recombination via Ga<sub>i</sub>-related defects can lead to suppression of non-radiative recombination in GaNAs-based solar cells.

Despite of the great promises and attractive properties, device applications of the dilute nitride NWs are very far from commercialization, which is probably not surprising considering their infant stage. The current challenges for the realization of the full potential of these materials on route towards the real-life applications are manifold. The growth of dilute nitride NWs is so far restricted to MBE, which creates difficulties in controlling their crystalline structure to the same extent as is currently achieved for conventional III-V NWs. All existing dilute nitride NWs contain a large number of crystalline defects, such as twinning planes, and have predominantly ZB structure. Achieving the well-controlled crystalline structure of the NWs - the prerequisite for the lattice engineering, therefore, requires further efforts. Other challenges include but are not restricted to: (i) achieving higher N compositions; and (ii) designing strategies of controllably introducing the desired defects during the growth without compromising the material quality. In spite of these challenges, we are convinced that this novel NW system, with its extraordinary physical properties uncommon to conventional semiconductors, will make significant contributions to the advances in the area of photovoltaics.

### Publikationslista (Publication list)

The project results were published in the following refereed journal articles and book chapters.

 S. Sukrittanon, Y. J. Kuang ), A. Dobrovolsky, Won-Mo Kang, Ja-Soon Jang, Bong-Joong Kim, W. M. Chen, I. A. Buyanova and C. W. Tu: Growth and characterization of dilute nitride GaNxP1-x nanowires and GaNxP1-x/GaNyP1-y core/shell nanowires on Si (111) by gas source molecular beam epitaxy. Appl. Phys. Lett. 105, 072107 (2014)



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The obtained results were also presented at numerous international conferences including 9 invited talks.

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### **Bilagor (Attachments)**

Administrativ bilaga