

Nanofils Semiconducteurs



Journées Nationales des Nanofils Semiconducteurs (J2N) Grenoble – lundi 13 / mercredi 15 Novembre 2017

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PARTENAIRE PRIVE



Horaire	Lundi 13 novembre 2017
13:30 13:45	Accueil à l'ENSE3, bât. GreEn-ER, 21 rue des Martyrs 38031 Grenoble cedex 1 Introduction
14:00	Session 1 : Dispositifs pour l'Energie et la Détection (chairman : Y. Coffinier)
14:00 - 14:30	Invité 1: T. Pauporté, Chimie Paris Tech, « Electrochemical Growth of ZnO Nanowires Applied to Nanosensors »
14:30 - 14:50	<u>Oral 1</u> : T. Le, <i>CEA-INAC</i> , « Étude électrogravimétrique du stockage électrochimique dans des électrodes à base de nanofils de silicium »
14:50 – 15:10	Oral 2 : M. Al-Ghzaiwat, LPICM, « Mini-modules based on radial junction silicon nanowire solar cells grown on FTO/glass substrates »
15:10 - 15:30	<u>Oral 3</u> : O. Burchak, ENWIRES, « Mass synthesis of silicon nanowires for their application in Li-ion batteries »
15:30	Pause-café
16:00	Session 2 : Caractérisations et Propriétés 1 (chairman : J. Pernot)
16:00 - 16:30	Invité 2 : J. Claudon, <i>CEA-INAC</i> , « Une boîte quantique intégrée dans un nanofil semiconducteur : une nouvelle plateforme pour l'optique quantique et la nanomécanique hybride »
16:30 – 16:50	<u>Oral 4</u> : L. Mancini, <i>C2N</i> , « Optical properties of grown-on-graphene GaN nanowires »
16:50 - 17:10	<u>Oral 5</u> : F. Donatini, <i>Institut Néel</i> , « Exciton diffusion coefficient in ZnO nanowires »
17:10 - 17:30	<u>Oral 6</u> : M. Amato, <i>C2N</i> , « Crystal phase effects in group IV nanowire polytypes and their homojunctions »
17:30	Pause
17:40	Session 3 : Dispositifs pour la Nanoélectronique (chairman : B. Salem)
17:40 – 18:10	Invité 3 : S. Barraud, CEA-LETI, « Enjeux et avancées technologiques des transistors MOSFET à nanofils »
18:10 - 18:30	<u>Oral 7</u> : E. Eustache, <i>LTM</i> , « Fabrication and characterization of horizontal and vertical GeSn nanowires using a top-down approach for future CMOS generations
18:30 – 18:50	» <u>Oral 8</u> : E. Azrak, <i>GPM</i> , <i>LPICM</i> , « Growth of in-plane Ge-Sn nanowires by solid- liquid-solid mechanism »
18:50	Fin des présentations

Horaire	Mardi 14 novembre 2017
08:50	Accueil à l'ENSE3, bât. GreEn-ER, 21 rue des Martyrs 38031 Grenoble cedex 1
9:00 9:00 - 9:30 9:30 - 9:50 9:50 - 10:10 10:10 - 10:30	Session 4 : Croissance 1 (chairman : S. Plissard) Invité 4 : B. Alloing, <i>CRHEA</i> , « GaN nanowires by selective area approaches » Oral 9 : F. Glas, <i>C2N</i> , « Comparison of modeling strategies for the growth of heterostructures in III-V nanowires » Oral 10 : J. Cibert, <i>Institut Néel</i> , « Croissance et sublimation de nanofils : boîtes quantiques CdTe dans ZnTe » Oral 11 : F. Oehler, <i>C2N</i> , « Growth dynamics of self-catalyzed GaP nanowires arrays grown by MBE »
10:30	Pause-café
11:00 11:00 – 11:30	Session 5 : Dispositifs pour la Détection et l'Energie (chairman : V. Sallet) <u>Invité 5</u> : Y. Coffinier, <i>IEMN</i> , « Fabrication et fonctionnalisation de nanofils de silicium pour des applications santé et environnementales »
11:30 – 11:50 11:50 – 12:10	<u>Oral 12</u> : X. Guan, <i>INL</i> , « GaAs/TiO ₂ core-shell nanowires for solar water splitting » <u>Oral 13</u> : T. Nguyen, <i>LMGP</i> , « Nanonets semiconducteurs : De la conception à l'intégration en dispositifs électroniques pour la détection de biomolécules »
12:10 - 12:30	Oral 14 : A.C. Salaün, <i>IETR</i> , « Résistances à base de nanofils de silicium pour la détection de bactéries »
12:30	Repas Buffet : salle de restauration
14:00 14:00 – 14:20	Session 6 : Caractérisations et Propriétés 2 (chairman : M. Tchernycheva) Oral 15 : G. Brémond, INL, « Measurement considerations on doping profiles by scanning probe microscopy on semiconductor nanowires and nanostructures »
14:20 - 14:40	<u>Oral 16</u> : A.M. Siladie, <i>CEA-INAC</i> , « p-n junction visualization and quantitative characterization on single $Al_xGa_{1-x}N$ nanowires »
14:40 - 15:00	Oral 17 : N. Chauvin, <i>INL</i> , « Optical polarization properties of wurtzite InP and InAs/InP nanowires grown on silicon (111) »
15:00 – 15:20	<u>Oral 18</u> : D. Beznasiuk, <i>Institut Néel</i> , « Strain distribution along an axial InGaAs/GaAs nanowire heterostructure »
15:20 - 15:40	<u>Oral 19</u> : M. Den Hertog, <i>Institut Néel</i> , « In-situ biasing and off-axis electron holography of a ZnO nanowire »
15:40	Pause-café

16:15 16:15 - 16:45 16:45 - 17:05 17:05 - 17:25 17:25 - 17:45	Session 7 : Dispositifs pour l'Optoélectronique (chairman : J. Eymery)Invité 6 : P. Gilet, ALEDIA, « Nanofils : d'un objet scientifique à la création d'uncomposant électronique grand public »Oral 20 : N. Guan, C2N, « Flexible LEDs based on III-Nitride core-shell nanowireLEDs »Oral 21 : T. Cossuet, LMGP, « ZnO / Sb ₂ S ₃ core shell nanowire heterostructures forETA solar cells »Oral 22 : A. Fave, INL, « HETONAN project: High efficiency tandem solar cells basedon III-V nanowires on silicon »
17:45	 Session Posters et cocktail dinatoire P1: M. Vettori, <i>INL</i>, « Ordered arrays of GaAs/AlGaAs nanowires for photovoltaic cells » P2: M. Foldyna, <i>LPICM</i>, « In-situ study of nanowire growth by spectral ellipsometry » P3: J. Tang, <i>C2N</i>, « Growth of anisotropic quantum dots in thin III-V nanowires » P4: X. Guan, <i>INL</i>, « GaAs Core / SrTiO₃ shell nanowires grown by molecular beam epitaxy » P5: C. Verrier, <i>LMGP</i>, « Tunable morphology and pH-dependent doping of ZnO nanowires grown by chemical bath deposition » P6: S. Hassani, <i>GEMaC</i>, « Structural properties and ordering studies in Zn_{1-x}Mg_xO alloys grown by MOVPE » P7: P. Chenevier, <i>CEA-INAC</i>, « Mass production of silicon nanowires » P8: G. Amiri, <i>GEMaC</i>, « MOCVD growth and optical characterization of ZnTe/ZnMgTe core-shell nanostructures » P9: T. Cossuet, <i>LMGP</i>, « Polarity-dependent selective area growth and physical properties of ZnO nanorods by chemical bath deposition » P10: R. Fons, <i>CEA-INAC</i>, « Vertical CdSe/ZnSe nanowire-quantum dots and enhanced photon extraction with a bottom-up photonic shell » P11: K. Kheng, <i>CEA-INAC</i>, « Vertical CdSe/ZnSe nanowire-quantum dots and enhanced photon extraction with a bottom-up photonic shell » P13: G. Brémond, <i>INL</i>, « Cartographie de la polarisation d'une couche d'amorce de nanofils de ZnO par les techniques de la microscopie en champ proche » P14: B. Gautier, <i>INL</i>, « Cattographie de la polarisation de nanofils Ge_xSn_{1-x} par CVD via le mécanisme VLS » P15: T. Laffner, <i>LTM</i>, « Elaboration et caractérisation de nanofils Ge_xSn_{1-x} par CVD via le mécanisme VLS » P17: C. Durand, <i>CEA-INAC</i>, « GaN/InAlN multiple quantum well tubes »

P18 : T. Cerba, LTM, « DSA-based process for InAs horizontal nanowires in sub-
10nm FET »
P19: A. Kapoor, CEA-INAC, « Green emission from In-rich core-shell InGaN/GaN
multi-quantum wells along GaN nanowires »
P20 : C. Justeau, GREMAN, « Influence des paramètres de synthèse sur la structure
des nanofils de ZnO pour la fabrication de nanogénérateurs piézoélectriques »
P21 : M. Parmar, IMEP-LaHC, « Development and characterization of flexible/non-
flexible energy transducers based on ZnO nanowires »
P22 : A. El Kacimi, CEA-LETI, « Capteurs piézoélectriques capacitifs souples à base
de fils de GaN verticaux : design et réalisations »
P23 : N. Pelekanos, CEA-INAC, Univ. of Crete, IESL/FORTH, « Random and periodic
arrays of GaAs/InGaAs core-shell nanowires for photovoltaic applications »
P24 : Y. Leprince-Wang, ESYCOM, « ZnO nanowire based microfluidic system for
photocatalytic water purification »
P25 : Y. Leprince-Wang, ESYCOM, « Transition-metal doped ZnO nanowires for
photocatalytic water purification »
P26 : M. Capochichi-Gnambodoe, ESYCOM, « Gas sensing performance
enhancement of transition-metal doped ZnO nanowires »
<u>P27</u> : E. Bano, IMEP-LaHC, « Investigating the long-term stability of Si and SiC
nanowires under physiological conditions »
P28 : K. Boche, Imina Technologies, « Characterization of the piezoelectric
response of GaN nanowires for the design of flexible force sensors »

Horaire	Mercredi 15 novembre 2017
08:50	Accueil à l'ENSE3, bât. GreEn-ER, 21 rue des Martyrs 38031 Grenoble cedex 1
9:00	Session 8 : Croissance 2 (chairman : C. Durand)
9:00 - 9:30	Invité 7 : G. Patriarche, C2N, « Observation in-situ dans un microscope
	électronique à transmission de la croissance par jets moléculaires de
	nanostructures III-V »
9:30 – 9:50	Oral 23 : M. Zeghouane, Institut Pascal, « Homogeneous and high-quality InGaN
	nanowires with different indium compositions »
9:50 - 10:10	<u>Oral 24</u> : S. Plissard, <i>LAAS</i> , « Defect-free Bi _{1-x} Sb _x nanowires on Si by MBE »
10:10 - 10:30	<u>Oral 25</u> : V. Sallet, <i>GEMaC</i> , « ZnO nanowire facets transformation induced by Ga
	doping »
10:30	Pause-café
11:00	Session 9 : Dispositifs Piézoélectriques (chairman : V. Consonni)
11:00 - 11:30	Invité 8 : N. Gogneau, C2N, « Piezo-electric III-N nanowires for energy harvesting »
11:30 – 11:50	Oral 26 : S. Boubenia, GREMAN, « Optimisation géométrique des nanogénérateurs
	à base de nanofils d'oxyde de zinc »
11:50 – 12:10	Oral 27 : Y. Leprince-Wang, ESYCOM, « ZnO nanowire-based piezoelectric
	nanogenerator »
12:10 - 12:30	Oral 28 : E. Saoutieff, CEA-LETI, « High-Resolution fingerprint sensing with vertical
	piezoelectric nanowire matrices: main achievements of the PiezoMAT project »
12:30	Bilan et discussion collégiale (animateur : S. Plissard)
13:00	Repas Buffet : salle de restauration
14:00	Réunion du Bureau Nanofils Semiconducteurs
16:00	Fin des J2N

LUNDI 13 NOVEMBRE

SESSION 1 : DISPOSITIFS POUR L'ENERGIE ET LA DETECTION

Electrochemical Growth of ZnO Nanowires Applied to Nanosensors

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Keywords: ZnO, Electrodeposition, Nanowires, Doping, Nanosensors

ZnO, a wide bandgap II-VI semiconductor (Eg = 3.4 eV) with a large exciton binding energy (60 meV), is studied in a large extent for various optical and electronic applications. ZnO nanostructures can be grown from solutions by several techniques such as hydrothermal deposition, chemical bath deposition, electrochemical deposition (ECD)... The latter technique is of utmost interest for integration of the structures in optoelectronic devices since it is low-temperature and low-cost. It can be easily scaled-up for preparations on large surface area substrates. Moreover, due to the electron exchange process at the origin of the oxide deposition, a good electrical continuity is ensured between the substrate and the oxide layer, and no seed layer is required.[1,2]

We will describe the technique of the electrochemical deposition of ZnO nanowires with a high structural quality. The growth of nanocolumn and nanowire arrays can be achieved by the method with controlled aspect ratio, density and optical properties.[2-5] The final structure can be finely tuned by adjusting the precursor concentration in the bath, the deposition time, the electrochemical potential, the substrate and so on. Moreover, NWs can be doped by various transition metals such as Cu, Cd, Ag, Pd... with a controlled concentration and surface metal nanoclusters can also be one-step obtained.[6] These nanowires have been detached, electrically connected and used as single functional nanosensors. We will focus mainly on highly selective and sensitive H₂ gas nanosensors and UV photodetectors. We will show their significantly changed detection properties and improved selectivity by controlling the dopants and their concentrations.

Références :

[1] T. Pauporté, E. Jouanno, F. Pellé, B. Viana P. Aschehoug, J. Phys. Chem. C., 2009, 113, 10422–10431. [2] T. Pauporté, G. Bataille, L. Joulaud, F.J. Vermersch, J. Phys. Chem. C., 2010, 114 194-202.). [3] O. Lupan, T. Pauporté, T. Le Bahers, B. Viana, I. Ciofini, Adv. Funct. Mater., 2011, 21 3564. [4] O. Lupan, L. Chow, T. Pauporté, L.K. Ono, B. Roldan Cuenya, G. Chai, Sensors and Actuators B, 2012, 173, 772. [5] O. Lupan, V. Cretu, V. Postica, M. Ahmadi, B. Roldan Cuenya,L. Chow, I. Tiginyanu, B. Viana, Th. Pauporté, R. Adelung, Sensors Actuators B, 2016, 223, 893.[6] O. Lupan, T. pauporté et al., Sensors Actuators B, 2018, 254, 1259–1270.

Étude électrogravimétrique du stockage électrochimique dans des électrodes à base de nanofils de silicium

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Mots clés : Nanofils de Silicium, Stockage d'énergie, Supercondensateurs, Micro-balance à quartz.

Les électrodes élaborées avec des architectures nanométriques sont de plus en plus étudiées pour améliorer les performances des batteries et supercondensateurs. En particulier les nanofils de silicium dopés permettent d'obtenir de hautes valeurs de puissance et d'énergie grâce à de larges surfaces développées et une bonne conductivité jusqu'au collecteur de courant [1]. Cependant les mécanismes d'échanges ioniques et les phénomènes de décomposition de l'électrolyte ayant lieu à l'interface des nanofils varient en fonction de l'électrolyte utilisé et restent largement méconnus.

Une meilleure compréhension de ces mécanismes peut être obtenue avec la microbalance à quartz électrochimique (EQCM) qui couple l'analyse électrochimique à la mesure des variations de masse de l'électrode, ce qui permet de distinguer les espèces mises en jeu et la cinétique des échanges ioniques [2].

Pour réaliser cette étude, la croissance de nanofils de silicium a été mise au point sur un substrat piézoélectrique en GaPO4 compatible avec la température de croissance par CVD à 600°C (Fig. 1.a).

Des mesures électrogravimétriques effectuées dans divers milieux organiques et des mélanges de liquides ioniques ont permis de distinguer différents mécanismes de stockage électrochimique et leurs cinétiques associées (Fig. 1.b).

a) 2,um





Références :

 Aradilla, D.; Gentile, P.; Bidan, G.; Ruiz, V.; Gómez-Romero, P.; Schubert, T. J.; Sahin, H.; Frackowiak, E.; Sadki, S. *Nano Energy* **2014**, *9*, 273–281.
 Escobar-Teran, F.; Arnau, A.; Garcia, J.; Jiménez, Y.; Perrot, H.; Sel, O. *Electrochemistry Communications* **2016**, *70*, 73–77.

Mini-modules based on radial junction silicon nanowire solar cells grown on FTO/glass substrates

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Index Terms: Mini-modules, Radial junction, a-Si:H, silicon nanowire, VLS method, PECVD, laser scribing.

Over the last decade, the installed solar photovoltaic (PV) panels increased significantly worldwide. Furthermore, over 90 % of solar PV market is based on silicon. Thin film technology takes part for the requirements of a low cost and increased flexibility. Here we study radial junction silicon nanowire (RJ SiNW) solar mini-modules fabrication to take advantage of strong light trapping of nanowires. In addition, the fabrication process of RJ SiNWs is fully compatible with wellestablished industrial thin film technology. An open-circuit voltage of 4.5 V for cells connected with the assistance of the laser scribing technique, has been achieved for 6 cells of $4.3 \times 0.4 \text{ cm}^2$ area each. The process starts with laser scribing of the SnO_2 : F/glass substrates (5x5 cm²) (step P1). Then, after introducing the samples inside the plasma-enhanced chemical vapor deposition (PECVD) reactor, Sn catalyst was produced by a hydrogen plasma treatment of the SnO₂:F substrates at temperature of 250 °C (above the melting temperature of Sn). Afterwards, p-type SiNWs were grown using plasmaassisted vapor-liquid-solid (VLS) growth [1]. Subsequently, the intrinsic a-Si:H layer was deposited with a thickness around 100 nm. For the finalization of the RJ, the samples were moved to another PECVD reactor where n-type µc-SiO_x:H was deposited. After the SiNW radial junction solar cell deposition, the nanowire forest underwent a second laser scribing step (step P2). Finally, an ITO top electrode was deposited using sputtering technique and ITO lines (step P3) were obtained using a lift off technique. The most recent result on RJ SiNW solar mini-modules will be discussed as well as the prospects for this technology and the challenges still lying ahead.



Figure 1: Schematics showing the geometry of laser scribed mini-modules. The yellow dashed line illustrates the electrical current path through the interconnected cells.

References: [1] S. Misra, et al., Journal of Physics D: Applied Physics, 2014, 47, 393001.

Mass synthesis of silicon nanowires for their application in Li-ion batteries

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Silicon nanowires, industrial fabrication process, anode materials for Li-ion batteries

Lithium-ion batteries, supercapacitors, photo- and thermo-voltaics: all these energy storage and conversion devices can strongly benefit from unique properties of silicon nanowires (SiNWs). In the case of lithium batteries, it was found that silicon nanowire electrodes could accommodate large strain without pulverization, provide good electronic contact and conduction, and display short lithium insertion distances.¹ Industrial scale fabrication of nanowire-containing devices needs an efficient mass production of high quality nanowires. Intensive research efforts have been devoted to safe and inexpensive nanowire fabrication processes. But serious challenges still remain before the SiNWs can be deployed cost-effectively and on a large scale.

We develop a novel bottom-up synthesis of ultra-thin SiNWs (Fig. 1a). The SiNWs can be doped by introducing a source of dopant, either P or B, and the diameter of the SiNWs can be kept highly homogeneous. Several grams per batch of these high quality SiNWs have been produced using our first industrial reactor. The measured performances of the obtained nanowires, used as anode material in half-cells against Li electrode, indicate their high potential for increasing both the capacity and cycling stability of LIBs (Fig 1b). In summary, low-cost, safe and high-throughput production, together with precise control of all nanowire parameters, makes this manufacturing procedure highly attractive for industrial scale fabrication of SiNWs.



Figure 1: a) SEM image of SiNWs; b) Galvanostatic cycling of a half-cell with SiNW electrode.

[1] C. K. Chan, H. Peng, G. Liu, K. McIlwrath, X. F. Zhang, R. A. Huggins, Y. Cui, *Nature Nanotechnology*, **2008**, *3*, 31-35.

SESSION 2 : CARACTERISATIONS ET PROPRIETES 1

Une boîte quantique intégrée dans un nanofil semiconducteur : une nouvelle plateforme pour l'optique quantique et la nanomécanique hybride

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Mots clés : boîte quantique, nanofil semiconducteur, guide d'onde, antenne optique, nanomécanique hybride

Les boîtes quantiques semi-conductrices sont des atomes artificiels qui présentent – à température cryogénique – des propriétés optiques exceptionnelles. Leur intégration dans un nanofil, lui aussi semiconducteur, ouvre de riches perspectives pour l'optique quantique et dans le domaine émergent de la nano-mécanique hybride.

Dans un premier temps, je décrirai le développement d'antennes optiques basées sur un nanofil, dont on exploite ici les propriétés diélectriques. Ce guide d'onde permet de canaliser l'émission de la boîte quantique vers un unique mode guidé. L'intégration d'un miroir et d'un *taper* permettent ensuite d'obtenir un diagramme de rayonnement Gaussien et directif. De telles antennes trouvent des applications directes à la réalisation de sources brillantes d'états non-classiques de la lumière [1-3], et plus généralement à réalisation d'expériences d'optique quantique.

D'un point de vue mécanique, un nanofil est aussi un oscillateur. Lorsque ce dernier vibre, la contrainte mécanique appliquée sur la boîte vient moduler fortement son énergie d'émission [4]. Grâce à ce couplage, on peut par exemple utiliser la boîte quantique pour sonder finement les vibrations mécaniques du nanofil [5]. A plus long terme, ce système pourrait aussi permettre de préparer des états quantiques de vibration mécanique. Ces travaux sont menés en étroite collaboration avec des collègues de l'Institut Néel (Grenoble) et de l'Université de Bâle (Suisse).

Références :

- [1] J. Claudon et al., Nature Photonics 2010, 4, 174.
- [2] M. Munsch et al., Phys. Rev. Lett. 2013, 110, 177402.
- [3] P. Stepanov et al., Appl. Phys. Lett. 2015, 107, 141106.
- [4] Y. Yeo et al., Nature Nanotechnology 2014, 9, 106.
- [5] M. Munsch et al., Nature Communications 2017, 8, 76.

Optical properties of grown-on-graphene GaN nanowires

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Key words: GaN nanowires, growth on Graphene, Micro-photoluminescence

Two-dimensional atomic layer materials such as graphene have been recently proposed as a novel substrate for GaN growth. Graphene can be transferred on any carrier substrate and can impose an epitaxial relationship for the grown GaN nanowires (NWs) [1,2]. Plasma-Assisted Molecular Beam Epitaxy growth of good crystal quality GaN NWs was demonstrated on graphene transferred to SiO_2 layer [2]. The NW growth on graphene is strongly selective with respect to the SiO_2 surface for optimized growth conditions, which is very promising for the development of highly ordered NW arrays. Still, a systematic study of the optical properties of GaN NWs grown on graphene has not been performed yet.

We address, by micro-photoluminescence (μ PL), the optical properties of grown-on-graphene GaN NWs and compare them with those of NWs grown on SiO₂/Si(100), SiN/Si(111) and AlN/Si(111). The μ PL study of grown-on-graphene NWs arrays points out the absence of photoemission at energies which are typically ascribed to the presence of inversion domain boundaries (defect commonly observed in NWs grown on Si [3]). The assessment of the optical properties of dispersed and free-standing single NWs allows for further insight into radiative recombination processes.



Figure 1: Micro-photoluminescence spectra acquired from GaN NWs (a) grown on graphene on $SiO_2/Si(100)$ and (b) on traditional Si(111) with AlN buffer substrates.

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Exciton diffusion coefficient in ZnO nanowires

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Mots clés : Nanowire, exciton, diffusion coefficient, cathodoluminescence, lifetime.

In semiconductor nanowires the exciton diffusion coefficient can be determined using a scanning electron microscope fitted with a cathodoluminescence system. High spatial and temporal resolutions cathodoluminescence experiments are needed to measure independently the exciton diffusion length [1] and lifetime in single nanowires. However, both diffusion length and lifetime could be affected by the electron beam bombardment during observation and measurement. Thus, in this work the exciton lifetime in a ZnO nanowire is measured versus the electron beam dose via a time-resolved cathodoluminescence experiment with a temporal resolution of 50 ps. The behavior of the measured exciton lifetime is consistent with our recent work [2] on the electron beam dose dependence of the exciton diffusion length in similar nanowires investigated under comparable SEM conditions. Combining the two results, the exciton diffusion coefficient in ZnO is determined and is found constant over the full span of electron beam dose.



Figure : Electron beam dependence of measured exciton lifetime τ , surface recombination velocity S, diffusion length L_D from our previous work [1] and diffusion coefficient D.

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Crystal phase effects in group IV nanowire polytypes and their homojunctions

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Mots clés : band offset; density functional theory; electronic structure; Silicon nanowires; silicon polytypes

Crystal structure and interface engineering are acquiring an increasing importance in nanoscience because of their enormous potential to conceive new properties and functionalities [1]. As for nanowires (NWs), the emergence of new stable polytypes of common semiconductors promises to have a crucial impact in materials design [2].

Recent experimental investigations have confirmed the possibility to synthesize and exploit polytypism in group IV nanowires. Indeed, while the crystal structure of Si and Ge nanowires (NWs) at standard conditions usually takes a well-defined ned cubic-diamond phase (as for their bulk counterparts), in the last few years several experimental observations of NWs exhibiting other phases — i.e. the hexagonal-diamond one — have been reported [3-4]. Other studies have instead succeeded in growing Si and Ge NWs containing both the cubic-diamond and the hexagonal-diamond phases opening the way to the fabrication of well-ordered group IV polytype homojunctions [5].

Driven by this promising evidence, here I will discuss recent first-principles calculations of the electronic and optical properties of hexagonal-diamond and cubic-diamond Si and Ge NWs as well as their homojunctions [6-7]. I will outline how a change in the crystal phase can strongly modify the electronic structure and optical response of the NW inducing novel and fascinating properties. Furthermore, I will show that, in the case of homojunctions, playing on crystal phase, size and length of the junction is an efficient tool to modulate band offsets and electron-hole separations.

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SESSION 3 : DISPOSITIFS POUR LA NANOELECTRONIQUE

Enjeux et avancées technologiques des transistors MOSFET à nanofils

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Mots clés : CMOS, nanofils, intégration Gate-Last, mobilité électronique

Depuis de nombreuses années, les transistors MOSFET à nanofils de silicium sont considérés comme le dispositif ultime de la technologie CMOS. En effet, ces nanostructures unidimensionnelles entourées par une grille high-κ/métal possède une excellente immunité aux effets de canaux courts permettant de poursuivre la réduction d'échelle bien connue sous le nom de *loi de Moore*. Si les premières démonstrations de nanofils superposés et à grille enrobante ont été réalisées il y a une dizaine d'année [1], des avancées majeurs ont été réalisées depuis quelques années [2]. Certaine industrie de la microélectronique inscrive désormais cette nouvelle architecture dans leur feuille de route pour le nœud 4nm [3]. Les enjeux et les avancées technologiques des transistors à nanofils fabriqués par une approche *Gate-Last* seront discutés. Les principales étapes technologiques de la route *Gate-Last* sont montrées sur la figure 1. Nous expliciterons la manière de les dessiner et de les fabriqués pour les rendre compétitif par rapport aux technologies MOSFET existantes. Nous montrerons que cette nouvelle architecture offre plus de liberté aux *designers* pour optimiser la performance et la faible consommation des circuits intégrés grâce à un ajustement adéquat de la largeur des fils de Si.



Figure 1: Images obtenues par microscopie électronique en transmission montrant les étapes clés d'une intégration *Gate-Last* des transistors à nanofils. (a) formation d'un empilement (Si/Si_{0.7}Ge_{0.3}) par épitaxie, (b) gravure de la couche active (Si/Si_{0.7}Ge_{0.3}), (c) formation des espaceurs internes, (d) nanofils de Si superposés et à grille enrobante.

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Fabrication and characterization of horizontal and vertical GeSn nanowires using a "top-down" approach for future CMOS generations

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Key words: germanium tin nanowire transistor

Germanium Tin ($Ge_{1-x}Sn_x$ or GeSn) alloy material has emerged as a promising candidate for high performance and low-power consumption CMOS devices. By varying the Sn concentration, GeSn bandgap can be tuned [1] and low effective mass [2] and high carriers mobility [3] can be achieved. To reach the market, GeSn technology must outperform silicon. On the one hand, a number of process bottlenecks must be overcome, such as the interface quality between the channel and the gate-oxide and source/drain contacts optimization. On the other hand, GeSn multi-gate structures (tri-gate and gate-all-around architectures) must be realized to improve electrostatic control of the channel [4].

In this context, this work focus on the optimization of the dry etching process of vertical and horizontal GeSn nanowires with different Sn concentration (from 6% Sn to 15% Sn). L. Milord & al. [5] used Cl_2 , N_2 and O_2 to achieve anisotropic etching of micro scale pattern for optical application. However, to our knowledge, no systematic study of Sn concentration dependence of the etching profile nor nano scale pattern (sub 20 nm) achieved by a single-step process with a vertical profile have been reported in the literature. In this study, we discuss the impact of the different experimental conditions on etching rate and quality of the sidewalls. Interestingly, optimal etching parameters differ with the Sn concentration. Finally, we use ICP etching with CF_4 [6] to selectively remove the Ge buffer and release horizontal GeSn nanowires. Controlled and smooth etching of nanostructures is a crucial step towards fabrication of complete transistors based in GeSn material.



Figure 1: vertical $Ge_{0.94}Sn_{0.06}$ nanowires (a), five $Ge_{0.90}Sn_{0.10}$ horizontal nanowires (b) and a $Ge_{0.85}Sn_{0.15}$ horizontal nanowire (c)

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Growth of in-plane Ge-Sn nanowires by solid-liquid-solid mechanism

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Keywords: optoelectronics, pseudo-direct gap, Ge nanowires, PECVD, nanostructures

Due to the inefficient light emission of Si, most of optoelectronics uses direct band-gap materials i.e. group III-V, which are highly toxic and expensive. An urge to replace this hybrid approach by a monolithic Si integration with a group IV element has risen. Germanium-Tin (GeSn) alloy is an alternative pseudo-direct gap candidate that undergoes an indirect-to-direct transition for sufficient Sn concentrations (6% -11%) [1], and its compatibility with Si-based technologies [2]. Major problems arise from the large lattice mismatch between Ge and Sn (15 %), the low solubility limit of Sn in Ge (< 1% at.), and a high phase-separation tendency. This work reports the fabrication of germanium-tin nanowires (Ge-Sn NWs) by a mechanism known as Solid-Liquid-Solid (SLS) [3]. The SLS fabrication process consists of i) deposition of catalyst (In or Sn) nanodrops on a given substrate such as Si wafer, ii) hydrogen plasma treatment of catalyst nanodrops in a Plasma-Enhanced Chemical Vapor Deposition reactor, iii) deposition of an amorphous Ge layer and iv) annealing the sample at a temperature above the eutectic point to activate the growth. The influence of different growth conditions on the NWs synthesis will be presented. In particular we will show that this fabrication process allows to obtain Ge-Sn nanowires with a uniform distribution of Sn, with concentrations in the range of 10 to 20%. Further characterization will be made to understand the structural, electrical and luminescent properties of these nanostructures.

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MARDI 14 NOVEMBRE

SESSION 4 : CROISSANCE 1

GaN nanowires by selective area approaches

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Mots clés: GaN nanowires, selective area growth, GaN sublimation, MOCVD, MBE

GaN nanowires have been proposed as alternative building blocks for optoelectronic devices and sensors in order to improve their efficiency and sensitivity. Whenever precise positioning of the eventual nanoemitters, nanotransducers, etc. is required, selective area growth can be a means to achieve the necessary spatial accuracy. In this presentation we will describe two distinct and complementary techniques developed at CRHEA for manufacturing site-controlled GaN nanowires. The first approach concerns the growth by bottom up of GaN nanowires by MOCVD. The second is a top-down approach and uses the sublimation of a GaN epitaxial layer by high-temperature annealing in MBE. The optical and structural properties of the obtained one-dimensional heterostructures, characterized by photoluminescence and cathodoluminescence, will be presented and compared.



Figure 1: SEM images of GaN nanowires grown on masked GaN-on-sapphire templates [1]



Figure 2: (a-d): Sketches of different steps of the selective area sublimation as a function of time [2]

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COMPARISON OF MODELING STRATEGIES FOR THE GROWTH OF HETEROSTRUCTURES IN III-V NANOWIRES

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Mots clés : nanowires, heterostructures, modeling, nucleation, III-V compounds

We present two models of the vapor-liquid-solid growth of nanowires of alloyed compound semiconductors [1]. These models are tested against experiments previously carried out on axial heterostructures in self-catalyzed (Al,Ga)As nanowires [2]. Rather than adopting a simple generic description of the thermodynamics of the system (*e.g.* the regular solution model), we make use of the appropriate CALPHAD-determined bulk thermodynamic functions.

With a growth rate set by the group V dynamics, the key question is to find the relationship between the compositions of liquid catalyst droplet and solid. Our first model assumes the equilibrium relationship. It predicts heterostructure profiles in excellent agreement with the experiments [1,2]. Our second model acknowledges that nanowires grow via nucleation at the solid-liquid interface. We find the size and composition of the critical nucleus at the saddle point of the surface describing the work of formation of the nucleus (Fig. 1). Assuming a fixed nucleus edge energy, the critical composition is virtually independent of the As concentration in the liquid, so that the profiles are also well predicted. With a composition-dependent edge energy, the profiles may differ significantly. We clarify why the effects of the groups III and V atoms may be decoupled and why the reservoir effect is weak, and discuss possible extensions to other systems.



Figure 1: Variation of the work of formation of a solid (Al,Ga)As nucleus from liquid (Al,Ga,As) (with Al and As atomic concentrations of 0.002 and 0.02), as a function of its size r and fractional AlAs content x. Nucleus edge energy is 0.123 J m⁻² independently of x. T = 883 K.

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Croissance et sublimation de nanofils: boîtes quantiques CdTe dans ZnTe.

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Mots clés : energy dispersive x-ray spectrometry, nanowires, quantum dots, electron microscopy, Kirkendall effect

Lors de la croissance de nanofils de semiconducteurs en épitaxie par jets moléculaires à partir d'un catalyseur (nanoparticule ou gouttelette d'or), la sublimation du composé et l'évaporation des éléments à partir de la nanoparticule d'or peuvent être importants. C'est le cas de CdTe : la vitesse de croissance dépend fortement de la température et elle est réduite au-dessus d'une température limite. En retour, cela permet d'insérer des boîtes quantiques CdTe dans des nanofils ZnTe avec des interfaces abruptes; de plus, on minimise ainsi la formation d'agrégats de CdTe par croissance latérale sur les facettes du nanofil, ceux-ci rendant les spectres de photoluminescence difficiles à exploiter. Les résultats obtenus par reconstruction 3D quantitative utilisant l'EDX (energy dispersive x-ray spectrometry, Fig. 1a-b) ou l'analyse GPA (geometrical phase analysis) des images de microscopie électronique en transmission, sont ajustés par un modèle de croissance permet une éventuelle redissolution du CdTe dans la nanoparticule, lui conférant une forme de lampe (ou de goutelette pendante, Fig. 1d) que nous attribuons à l'effet Kirkendall : la diffusion du Cd dans la nanoparticule induit un déplacement de lacunes et la forme finale est stabilisée par l'énergie de surface (effet Kirkendall inverse).



Figure 1: (a) cartographie EDX d'un nanofil ZnTe contenant des insertions CdTe réalisées avec le même temps de croissance à des températures différentes ; (b) profil EDX du Cd et du Zn le long du nanofil précédent ; (c) vitesses de croissance de ZnTe et CdTe, en fonction de la température (symboles : mesures ; lignes : calcul) ; (d) forme de la nanoparticule d'or résultant de la sublimation de CdTe en fin de croissance.

Growth dynamics of self-catalyzed GaP nanowires arrays grown by MBE

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Mots clés : nanowire, MBE, growth, shadowing

Semiconductor nanowires (NWs) are now emerging as technologically relevant for devices such has III-V solar cells. GaAs and GaP nanowires can be grown on patterned silicon to replace the expensive III-V substrates. However much of the focus has been on the mask preparation and growth parameter[4][5], but not on the dynamics of the growth. Here we present study of the growth of arrays of self-catalyzed GaP nanowires. We have obtained near perfect hexagonal arrays using patterned (111) Si substrates with a silica mask. Markers are inserted during growth to determine the dynamics of the system. The nanowire array is then observed by cross-sectional TEM. Measurements of the axial spacing between the markers and of the marker width, as a function of position along the NW axis, allow us to evaluate precisely and simultaneously the axial and radial growth rates of individual NWs. We then develop a growth model that accounts quantitatively for the time evolution of these two growth rates.



Figure 1: SEM view (20° tilt) of the edge of an hexagonal array of GaP nanowires on patterned Si 111 grown by MBE.

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SESSION 5 : DISPOSITIFS POUR LA DETECTION ET L'ENERGIE

Fabrication et fonctionnalisation de nanofils de silicium pour des applications santé et environnementales.

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Les nanosciences et les nanotechnologies font depuis plus de vingt ans l'objet de travaux de recherche à la confluence de nombreuses disciplines scientifiques et technologiques. Fabriquer, observer et manipuler des nano-objets; les étudier afin de comprendre leurs propriétés et leurs interactions avec leur environnement permet d'envisager le développement des applications dans tous les champs technologiques. De la nanoélectronique, aux nanomatériaux en passant par la nanomédecine ou encore les nanobiotechnologies, les champs d'applications sont très vastes.

En matière de remédiation en particulier, les problématiques principales se focalisent sur la restauration de la qualité des sols ou de l'eau (eaux usées, contaminées...). Le développement de nouveaux outils issus des nouvelles technologies notamment des biotechnologies, de la chimie et des nanotechnologies associées au génie écologique est donc crucial pour la détection et l'élimination de certains types de polluants (nanofiltration, photocatalyse de polluants...). Cependant, on ne peut parler d'environnement sans discuter du problème de la gestion des ressources énergétiques disponibles. En effet, les inadéquations entre les besoins énergétiques et les ressources disponibles demandent un effort considérable pour la mise au point de dispositifs robustes, efficaces et fiables de stockage et de conversion. Là encore, les nanotechnologies sont impliquées dans les dispositifs énergétiques du futurs : cellules photovoltaïques, microbatteries, supercapaciteurs, récupération d'énergie (thermoélectricité, microfluidique).

D'un autre côté, l'avènement des nanotechnologies dans le domaine de la santé a permis le développement de nouveaux outils performants pour le domaine médical. Dans ce cas, l'exploitation des propriétés physiques, chimiques ou biologiques de certains nanomatériaux autorisent des approches très novatrices ouvrant la possibilité de détecter plus tôt certaines maladies, d'améliorer de façon drastique le diagnostic (*in vivo* et *in vitro*), les thérapies et les approches de médecine régénératrice. On parle alors de nanomédecine aux potentialités multiples.

Lors de cette présentation, je m'attacherai à présenter la fabrication, la fonctionnalisation et l'utilisation de nanofils de silicium pour des applications santé et environnementales développées au sein de mon laboratoire.

GaAs/TiO₂ core-shell nanowires for Solar Water Splitting

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Mots clés : GaAs NWs, self-catalyzed, MBE, TiO₂ passivation, PEC

Concerning the eco problem and energy crisis we are facing now, the energy harvested from sunlight offers a desirable approach [1]. Photoelectrochemical (PEC) hydrogen production from water provides a way to directly convert the solar energy into a storable clean fuel [2]. GaAs nanowire (NW) configuration is preferred for the high specific surface area as well as the good light absorption [3]. It can serve as a model system to demonstrate the quality of NW-array light absorber. However GaAs suffers the photo-corrosion in aqueous electrolytes [4]. TiO₂ is mainly used to passivate semiconductor electrodes for its remarkable stability [4, 5], which may well enhance the stability of GaAs based electrodes.

Self-catalyzed GaAs NWs were grown on Si(111) substrate by molecular beam epitaxy (MBE). Then, a 5 nm-thick TiO₂ layer was also deposited via MBE on the GaAs NWs (Figure 1 a). For the bare GaAs NW array, the current desnstiy is about -0.135 mA/cm² at 0.033 V vs RHE, and the V_{onset} is -0.47 V. After passivated by the amorphous TiO₂ layer, only a small transient current is detected (Figure 1 b). However, the photocurrent also decreases due to the poor conductivity of TiO₂. The optimization of the PEC performances of TiO₂ passivated GaAs NW array is being worked on.



Figure 1: a) SEM image of the GaAs / TiO₂ NW array, b) PEC responses (on-off curve) of the GaAs NWs array and GaAs / TiO₂ NW array in NaOH solution (pH=13).

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Nanonets semiconducteurs : De la conception à l'intégration en dispositifs électroniques pour la détection de biomolécules

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Mots clés : Nanostructure, nanonet, transistor à effet de champs, résistor, propriétés électriques, détection des biomolécules, ADN, acétone

Avec leur facteur de forme et leur surface spécifique élevés [1] alliés à leur taille nanométrique, les nanomatériaux unidimensionnels, nanotubes et nanofils (NFs), sont très sensibles à leur environnement surfacique, notamment à la présence de biomolécules, ce qui les rend attractifs dans le domaine du diagnostic médical et environnemental. Aujourd'hui, l'intérêt de ces nanostructures pour la biodétection a été révélé par de nombreuses études expérimentales. Toutefois, malgré ce fort potentiel, la technologie d'intégration reste à ce jour coûteuse et il est difficile de fabriquer des dispositifs aux propriétés reproductibles en raison d'importantes fluctuations des propriétés physiques d'un NF à l'autre. Ces points d'achoppement entravent par conséquent la production en masse des dispositifs à NFs, uniques ou en parallèles. Pour contourner ces difficultés, le NANONET (Nanostructured Network - NN) a été développé. Défini comme un réseau aléatoirement orientés des NFs semiconducteurs, le NN présente de nombreux avantages: électriquement actif, peu sensible aux défauts et aux fluctuations des propriétés des NFs donc reproductible, aisément fonctionnalisé. Cette dernière caractéristique permet d'y ajouter de nouvelles propriétés pour, par exemple, favoriser la biodétection, le tout en profitant des mêmes avantages que les NFs individuels le constituant.

Notre étude repose actuellement sur les NN de silicium et d'oxyde de zinc (ZnO). Alors que ce dernier est exploité, en configuration résistor, pour la détection d'acétone dans l'haleine des patients diabétiques, les NNSi sont intégrés au sein de transistors, base des capteurs à ADN. Les technologies utilisées sont toutes compatibles avec une intégration à grande échelle. Les résultats obtenus sont tous très prometteurs. Les nanonets sont assemblés de manière contrôlée par des techniques peu coûteuses, ils sont homogènes et transférables sur tout type des substrats (transparent, opaque, flexible, rigide...) ce qui permet d'envisager de nombreuses applications [2]. Le procédé d'intégration mis au point est simple, reproductible et compatible avec la technologie CMOS. Les dispositifs obtenus, notamment les transistors à NNSi présentent de bonnes caractéristiques électriques, même avec des canaux dont la longueur est plus longue que celle des NFs de plus d'un ordre de grandeur. Concernant la détection d'ADN et d'acétone, nos premiers prototypes de biocapteurs montrent des réponses homogènes du courant lors de la détection.

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Résistances à base de nanofils de silicium pour la détection de bactéries

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Mots clés : Nanofil de silicium, résistance, bactéries

La détection des bactéries pathogènes dans les aliments, l'eau et l'air est une question importante en raison de son impact critique sur la santé publique, en particulier avec l'émergence de bactéries multirésistantes. Les méthodes microbiologiques standard de culture bactériennes imposent des délais qui peuvent ralentir la mise en place de moyens de lutte contre les contaminations. Dans ce contexte, une stratégie consiste à développer des capteurs ultrasensibles à base de réseaux de nanofils de silicium pour une détection rapide de bactéries. Les nanofils possèdent en effet une grande surface de contact permettant des interactions préférentielles avec les bactéries. Des résistances à base de nanofils en silicium utilisées comme capteurs de bactéries sont fabriquées à l'aide des technologies classiques du silicium. Les nanofils sont obtenus par la méthode VLS (Vapeur Solide Liquide) utilisant de l'or comme catalyseur et les électrodes sont constituées de silicium polycristallin. Les bactéries sont préférentiellement accrochées dans le réseau de nanofils (fig.1a) et la résistance électrique des dispositifs diminue de manière significative en présence de bactéries (fig.1b). Les résultats montrent l'utilisation potentielle des résistances correspondantes, avec les nanofils comme unités sensibles pour la détection de bactéries.



Figure 1. (a) Cliché MEB illustrant des bactéries accrochées dans le réseau de nanofils de silicium, (b) caractéristiques électriques I-V de la résistance électrique avec et sans bactéries (Escherichia coli ATCC 35128, concentration 8 × 106 CFU/mL).

SESSION 6 : CARACTERISATIONS ET PROPRIETES 2

MEASUREMENT CONSIDERATIONS ON DOPING PROFILES BY SCANNING PROBE MICROSCOPY ON SEMICONDUCTOR NANOWIRES AND NANOSTRUCTURES

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Mots clés : NW, ZnO, doping, SPM, core-shell, SCM, SSRM

Measurement methods to quantitatively determine the doping in semiconductor nanowires (NW) or nanostructures (NS) are strongly requested in order to control the doping incorporation in such onedimensional structures and so far, to develop technology which would integrate them.

In the last two decades, scanning capacitance microscopy (SCM) and scanning spreading resistance microscopy (SSRM) based on atomic force microscopy (AFM), have emerged as promising tools for two-dimensional high resolution carrier/dopant profiling. Because the principle of these techniques is based on an AFM tip contact, not only the signal-on-noise ratio, but also a lot of parameters and environment measurement conditions have to be fulfilled properly. Generally, these techniques need for an accurate calibration method to achieve a quantitative doping analysis. These particularities are required to make sure that the results on the free carrier concentration are reliable.

Based on different examples obtained on the characterization of doping profile on semiconductor NWs and NS mainly on ZnO [1,2], this communication will aim to point out the different parameters of the structures and the measurement conditions to be controlled in order to define a method of calibration of the free carrier profiles in this type of NS.

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P-N JUNCTION VISUALIZATION AND QUANTITATIVE CHARACTERIZATION ON SINGLE AL_xGA_{1-x}N NANOWIRES

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Mots clés : electron beam induced current, p-n junction, aluminium gallium nitride

Actual 2D AlxGa1-xN LEDs exhibit low efficiency due to extended defects that affect the optical and electrical properties by trapping carriers or gettering point defects. The requirement for improved efficiency of actual devices could benefit from the use of nanowire heterostructures presenting the advantage of plastically relaxing the constraints during growth. One of the biggest challenges when

developing such devices is the assessment of the p-n junction. The present work combines complete optical and electrical characterization techniques adapted for studying single nanowires, allowing the visualization and characterization of the axial junctions in terms of abruptness and interdiffusion of carriers. Evidence of dopant incorporation coming from the different Raman signatures for single Al_xGa_{1-x}N NW junctions over a wide range of AlN molar fraction was obtained by using µ-Raman technique. High-resolution nanocathodoluminescence gave an estimation of junction width by mapping Si- and Mg-related optical signatures. Electrical characterizations provide information on the quality of materials and allow extracting the electrical parameters of the junction. Using Kelvin Probe AFM, electrical evidence of the Al_xGa_{1-x}N NW p-n junction through a clear CPD voltage offset has been observed [1]. Similarly to individually contacted GaN NW junctions [2], Al_xGa_{1-x}N NWs were studied allowing the extraction of minority carriers' diffusion lengths and depletion region width from the EBIC signal.



Figure 1. pn junction EBIC signature in a 60% AlGaN junction

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OPTICAL POLARIZATION PROPERTIES OF WURTZITE INP AND INAS/INP NANOWIRES GROWN ON SILICON(111)

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Mots clés : Nanowires, III-V semiconductor, optical spectroscopy

The integration of InAs/InP quantum dot-nanowires (QD-NWs) in more complex photonic structures requires to understand and to control the orientation of the emitting dipole. A specific room temperature microphotoluminescence (μ -PL) setup was built to investigate the polarization resolved μ -PL from the same NW array along two perpendicular orientations (from the NW side and along the NW axis). This setup is used to investigate two issues: 1) to determine the crystal field splitting Δ_{CF} and the spin orbit coupling Δ_{SO} values of wurtzite (Wz) InP and 2) to probe the hole mixing in different Wz InAs/InP QD-NW geometries. The investigated NW samples were grown on a Si(111) substrate by Vapor-Liquid-Solid assisted solid source Molecular Beam Epitaxy, using Au-In droplets as catalyst in-situ deposited at 500°C.¹ Axial and radial growth conditions were optimized to control the InP Wz phase, the NW diameter and the QD geometry.²

1) Excitation PL spectroscopy is usually used to extract Δ_{CF} and Δ_{SO} parameters of Wz InP. However, the equations expressing the valence band splitting are symmetric with respect to these two parameters and a choice $\Delta_{CF} > \Delta_{SO}$ or $\Delta_{CF} < \Delta_{SO}$ has to be taken into account in order to assign the numerical values. The investigation of the NW side emission (Fig 1.a) allows us to conclude that $\Delta_{CF} > \Delta_{SO}$ in Wz InP.

2) The polarization resolved μ -PL of InAs/InP QD-NWs emitting in the telecom band was performed from the NW side and along the NW axis. This allows us to investigate the mixing between the A and B holes of the Wz InAs QD as a function of the InAs/InP heterostructure geometry (Fig 1.b).



Figure 1: Polarization resolved µPL of Wz NWs collected along the NW side direction: a) A and B transitions of Wz InP NWs, b) ground state emission from two different QD geometries.

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STRAIN DISTRIBUTION ALONG AN AXIAL InGaAs/GaAs NANOWIRE HETEROSTRUCTURE.

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Key words: nanowire heterostructures, strain, dislocation-free interface

Advanced electronic and optoelectronic devices based on semiconducting nanowires (NWs) were recently successfully developed [1,2]. NWs receive much attention thanks to their unique onedimensional morphology. Indeed, their diameter, length and density are readily controllable by adjusting the growth parameters and the catalyst diameter. Moreover, NWs can host material combinations of very different lattice parameters since it can relax laterally and elastically release the strain caused by the mismatch. This enables to realize dislocation-free interfaces with no equivalent in traditional two dimensional thin film epitaxy. In this context, it is important to understand how the strain distributes at the vicinity of the interface in axial nanowire heterostructures. Indeed, strain deeply influences the band structure of semiconductor heterojunctions, and consequently the electronic and optical properties of the final device.

Here, we use transmission electron microscopy along two crystalline orientations to investigate the strain distribution of an $In_{0.85}Ga_{0.15}As/GaAs$ nanowire heterostructure [3]. We combine high-resolution scanning transmission electron microscopy (HRSTEM) together with image processing (Geometrical Phase Analysis (GPA)). We observe that a region of 20 nm-long around the $In_{0.85}Ga_{0.15}As/GaAs$ interface is strained and that the crystalline planes bend in this region close to the side walls. The interface between $In_{0.85}Ga_{0.15}As$ and GaAs is free of dislocations despite a 6% lattice mismatch, revealing full elastic relaxation (Figure 1).



Figure 1: HRSTEM images of the $In_{0.85}Ga_{0.15}As$ -on-GaAs nanowire from a) $\langle 2-1-10 \rangle$ and c) $\langle 01-10 \rangle$ zone axis. GPA colour maps (obtained by 0002 and -2110 spots from Fast Fourier Transformation (FFT)) and Moire patterns superposition corresponding to the lattice constant c/2 (b) and a (d) distribution along the nanowire axis.

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In-situ Biasing and Off-axis Electron Holography of a ZnO Nanowire

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Keywords: ZnO Nanowire; doping; surface charge; in-situ off axis electron holography; cathodoluminescence.

Quantitative characterization of electrically active dopants and surface charges in nano-objects is challenging, since most characterization techniques using electrons, ions or field ionization effects study the chemical presence of dopants, which are not necessarily electrically active. We perform cathodoluminescence and voltage contrast experiments on a contacted and biased ZnO nanowire with a Schottky contact and measure the depletion length as a function of reverse bias [1]. We compare these results with state-of-the-art off-axis electron holography in combination with electrical in-situ biasing on the same nanowire. The extension of the depletion length under bias observed in scanning electron microscopy based techniques is unusual as it follows a linear rather than square root dependence, and is therefore difficult to model by bulk equations or finite element simulations. In contrast, the analysis of the axial depletion length observed by holography may be compared with three-dimensional simulations, which allows estimating an n-doping level of 1×10^{18} cm⁻³ and negative sidewall surface charge of 2.5×10^{12} cm⁻² of the nanowire, resulting in a radial surface depletion to a depth of 36 nm. We found excellent agreement between the simulated diameter of the undepleted core and the active thickness observed in the experimental data. By combining TEM holography experiments and finite element simulation of the NW electrostatics, the bulk-like character of the nanowire core is revealed.

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SESSION 7 : DISPOSITIFS POUR L'OPTOELECTRONIQUE

Nanofils : d'un objet scientifique à la création d'un composant électronique grand public.

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Mots clés : Nanowires, LED, micro LEDs

Que ce soit pour l'éclairage ou pour la réalisation de displays au sens large, le marché des LEDs est en constante croissance. Parallèlement à l'augmentation des performances électro optiques (pour l'éclairage) ou à la pixellisation à très petit pas (pour les displays) par exemple, la réduction des coûts reste un des éléments importants pour l'accessibilité au marché. Afin de suivre les feuilles de route, il sera nécessaire soit de pousser les technologies existantes, soit de développer des technologies en rupture tout en gardant une maîtrise des coûts de fabrication.

Cinquante ans de microélectronique nous ont appris que l'augmentation de la taille du wafer de fabrication permettait une diminution régulière des coûts de fabrication. Les fabricants de LED l'ont également compris avec une volonté forte de travailler avec des substrats de saphir de plus en plus grands. Le passage sur des substrats de silicium 6 ou 8 pouces longtemps annoncé a toujours été repoussé principalement pour des problèmes de rendement de fabrication dus à la différence de paramètres de maille et des coefficients de dilatation thermique des deux matériaux utilisés.

L'utilisation des nanofils de GaN épitaxiés sur silicium permet de changer ce paradigme et apporte en plus de nouvelles opportunités pour la réalisation de dispositifs électroluminescents. Au cours de cet exposé, après avoir décrit les différents avantages des nanofils en fonction des applications visées, nous présenterons leurs caractéristiques avant de discuter des performances obtenues que ce soit au niveau d'un fil ou d'une assemblée de fils. Nous discuterons également des problèmes liés à l'industrialisation de cette technologie.



Figures : image d'un substrat 200mm post épitaxie de nanofils (à gauche) , image d'un composa 2x2cm² à base de nanofils (à droite)

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Flexible LEDs based on III-Nitride core-shell nanowire LEDs

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Key words: nanowire, nitride, flexible LED

Flexible light sources have attracted a lot of attention in research as well as in industry thanks to their numerous applications such as curved screens, rollable displays and implantable sources. The dominant technology in the market is based on organic light emitting diodes (OLEDs), which however suffer from the low luminance, low quantum efficiency and short lifetime, especially in the short wavelength range. The III-nitride p-n junction LEDs are free from these drawbacks, but cannot be directly used for the flexible applications because of the mechanical rigidity of the nitride material. Instead of conventional 2D thin films, nano-structured MOCVD core-shell nanowires [1] are used by our group as an alternate active material to realize flexible LEDs. By embedding the active nanowire LED array into a flexible polymer supporting layer, we demonstrated blue, green, blue/green bi-color [2] and white [3] flexible LEDs. This technology benefits from the high flexibility of the polymer and the outstanding opto-electronic properties of the nitride nanowires. The optic and electronic characterizations of the flexible LEDs will be presented.



Figure 1: Top: flexible nanowire blue, green and white LEDs under operation. Bottom: schematic and electroluminescence spectra of a bicolor LED.

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ZnO / Sb₂S₃ core shell nanowire heterostructures for ETA solar cells

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Keywords: ZnO nanowires, Sb₂S₃, core shell heterostructures, ETA solar cells.

ZnO nanowire (NW) arrays are promising building blocks for extremely thin absorber (ETA) solar cells. A ZnO NW-based core shell p-n heterojunction is typically formed using a direct p-type semiconductor as absorber layer and following the type-II band alignment. Increasing interest has been dedicated to this radial architecture owing to efficient light trapping and charge carrier management together with the use of a low amount of materials. Antimony trisulfide (Sb_2S_3) , as a p-type semiconductor with a 1.7 eV band gap energy and a high absorption coefficient, has been integrated into mesoporous-TiO₂-based dye-sensitized solar cells, showing power conversion efficiency (PCE) as high as 7.5 %. It is usually grown by low-cost, low-temperature chemical deposition techniques, which still make its combination with ZnO NWs difficult owing to their instability in acidic conditions. In this work, the crystallization process of Sb₂S₃ thin films is investigated by *in situ* x-ray diffraction and in situ Raman spectroscopy, revealing the intermediate formation of a metallic antimony phase and showing the optimal annealing temperature of 270°C [1]. Furthermore, an 8 nm-thick TiO₂ protective layer is grown by atomic layer deposition onto ZnO NW arrays grown by chemical bath deposition. Sb_2S_3 , as an absorbing shell, is subsequently deposited by spray. The Sb_2S_3 conformal shell with high crystalline quality covers the ZnO/TiO₂ NW arrays from the bottom to the top. The photovoltaic performance of the ZnO/TiO₂/Sb₂S₃ core shell NW heterostructures using P3HT as hole transporting material results in a promising PCE of 2.3 % [2]. The use of low-cost, surface scalable chemical deposition techniques for their whole fabrication opens the way for improving the performances of ZnO NW-based ETA solar cells.



Figure 1: J(V) measurements of ETA solar cells (left) with its schematic diagram (right). The insets are HAADF-STEM and FESEM images as well as EDS-STEM elemental map.

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"HETONAN" project:

High efficiency tandem solar cells based on III-V nanowires on silicon

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Mots clés : GaAs/AlGaAs, GaP/GaAsP nanowires, ordered array, silicon substrate, tandem solar cell, tunnel junction

In the past 15 years, the photovoltaic (PV) industry has drastically reduced the fabrication cost of silicon solar cell modules and the further improvement mainly relies on better cell efficiencies at low cost. However, the record efficiency of crystalline silicon (c-Si) single junction solar cell is approaching the maximum limit (\sim 30%) which is due to different physical loss mechanisms. In the HETONAN project we are designing and developing a tandem solar cell structure able to convert the solar spectra very efficiently by combining a planar Si bottom cell with a III-V cell based on coreshell nanowires (NWs) on top of it. The planar Si solar cell is used as a substrate for the growth of p-n core-shell III-V NWs. The two subcells are connected in series by a low-resistance tunnel junction elaborated in the planar Si structure using proximity rapid thermal diffusion. A nanopatterning (network of holes) on the Si substrate is realized in a thin PECVD SiO₂ layer in order to define the NW array whose parameters (diameter, period) were optimized thanks to optoelectronic simulations. Two different types of III-V NWs with the optimal bandgap value (1.7 eV) are evaluated: $GaAs_{0.7}P_{0.3}$ and Ga_{0.8}Al_{0.2}As NWs which are grown by MBE using the self-catalyzed VLS method. The results obtained in the different parts of the project will be presented: modelization of the tandem structure with a Jsc of 17.1mA/cm² in both subcells, measurement of a low resistive tunnel Si structure, evidence of a radial n-p core-shell junction.



Fig. 1: a) Scheme of the aimed tandem cell, **b)** Ordered array of core-shell GaAs/AlGaAs NWs with the p-n junction in the AlGaAs shell.

SESSION POSTER

Ordered arrays of GaAs/AlGaAs nanowires for photovoltaic cells

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Mots clés : GaAs/AlGaAs nanowires, ordered array, solar cell, photovoltaics, EBIC

Solar energy is for sure one of the many fields in which semiconductor nanowires (NWs) can play a key-role. In particular, one of the subjects receiving close review in the late years is the development of NW-based tandem solar cells (TSC)^[1,2], which appears particularly interesting since it offers the possibility to increase the photovoltaic (PV) efficiency.

In this context, we developed a project ^[3] whose aim is to obtain a TSC based on a Si(111) bottom cell coupled to a top one consisting in a ordered array of $Al_{0.2}Ga_{0.8}As$ NWs with radial p-n junction. Due to the difficulty to grow Ga-catalysed AlGaAs NWs on Si patterned substrates with a SiO₂ mask (because of the oxidation of aluminium adatoms), we chose to grow a GaAs p-doped core by VLS axial growth followed by the radial growth of a PV-active p-n AlGaAs shell. The process to realize these devices requires many different steps, but one of the most challenging tasks we focused on is the growth of NWs in ordered array on Si(111) patterned substrates. In order to fulfil this objective we started from the growth optimization of self-catalyzed GaAs NWs (Fig. 1a), then we continued with the growth of p-doped GaAs core/ p-n doped Al_{0.2}Ga_{0.8}As shell NWs with a final AlGaAs/GaAs passivation layer (Fig. 1b). In both cases results were positive, showing the possibility to obtain high yield of vertical NWs. TEM image in Fig. 1c reveals the different parts of a typical core-shell NW. Electron Beam Induced Current (EBIC) microscopy and photocurrent measurements realized on similar p-n core/shell GaAs NWs provided evidences of a good diodic behaviour of the radial p-n junction, showing the remarkable PV response from these structures.



Fig. 1: a) Ordered array of GaAs NWs. b) Ordered array of core-shell GaAs/AlGaAs NWs. c) TEM image of a core-shell NW.

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In-situ study of nanowire growth by spectral ellipsometry

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Index Terms: Silicon nanowire, solar cells, in-situ characterization, spectral ellipsometry.

Silicon nanowire solar cells have attracted the attention of the scientists for few decads already. Nevertheless, it is much more recently that the applications of nanowires in devices started to reach performance of their thin film counterparts in the fields such as light emitting diodes or photovoltaics. The increased efficiency of nanowire based solar cells requires more effort to be put into a process control and capabilities to characterize samples directly in-situ. We have focused on the in-situ characterization of nanowires grown by plasma-enhanced chemical vapor deposition (PECVD) using vapor-liquid-solid process by spectral ellipsometry. The samples are being monitored from the introduction of the substrate composing of thin layer of Sn evaporated over 1 μ m thick ZnO film on Corning glass substrate until the end of the nanowire growth. The whole process of nanowire growth has been described using appropriate models for the measured spectral range (wavelengths of 450-850 nm) based on effective medium theory. Results of the in-situ nanowire characterization provide us with the information on the material growth and indirectly also about the estimated density of nanowires [2]. The new possibilities for our upgraded PECVD reactor, including one pump-down fabrication and access to more materials, will be discussed in the context of the performance improvements enabled by removing the risk of surface oxidation in intermediate fabrication steps.

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Growth of anisotropic quantum dots in thin III-V nanowires

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Mots clés : Quantum dots, III-V nanowires, self-catalyzed growth, molecular beam epitaxy

Semiconductor quantum dots (QDs) are promising building blocks for the next generation of photonic and spintronic devices, such as single photon sources and quantum bits. Traditionally, the (QDs) are fabricated by a self-assembly method. This method has intrinsic limitations. Firstly, it is challenging to engineer the geometry of the QDs, which influences the quantum confinement effect (in particular, III-V QDs tend to adopt a lens shape). Secondly, the dot material must have a large lattice mismatch with its substrate to achieve the self-assembly of the dots. Thus, few materials can be used and the strain state of the QDs cannot be tuned flexibly. Moreover, the built-in strain in the dots leads to a heavy ground hole state.

Growth of QDs in nanowires (NWs) offers a more flexible way to engineer the geometry and the strain state. With this method, the aspect ratio of the dots can be tuned easily by changing the NW core growth time, and the strain adapted by varying the composition and the thickness of the NW shell. However, it is challenging to achieve sub-20 nm wide III-V NW cores using the self-catalyzed growth method. In this study, thin GaP and GaAs NWs were grown on Si(111)/SiO_x substrates by self-catalyzed molecular beam epitaxy. By tailoring the amount of pre-deposited Ga and the group V to group III flux ratio, GaP and GaAs NWs with diameters below 20 nm have been obtained reliably. We also inserted GaAs in GaP NWs. Transmission electron microscopy (TEM) shows that the GaAs/GaP interface in the radial direction is defect-free. The interface width in the axial direction is between 3 and 10 monolayers. The defects in the GaAs core are mainly twins in the zinc blende structure (wurtzite segments are also present). Energy dispersive X-ray spectroscopy (EDX) indicates that the insertion is nearly pure GaAs and the shell nearly pure GaP. The NW density is about 0.2 μ m², which permits the detection of photoluminescence from single NWs kept on their substrates.



Figure 1: (a) Mean diameter of the GaP NWs as a function of pre-deposited amount of Gallium and V/III atomic flux ratio. (b) High resolution TEM image of a thin Gap NW with GaAs insertion. (c) High angle annular dark-field image of a thin GaP NW with GaAs insertion. (d) Composite EDX map of the same NW as in (c) (blue: P signal, green: As signal).

GaAs Core / SrTiO₃ Shell Nanowires Grown by Molecular Beam Epitaxy

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Mots clés: Core / shell nanowires, hybrid nanowires, functional oxides, semiconductor, interface

Perovskite oxides possess a wide range of properties [1, 2] which can lead to the development of multifunctional devices when coupling with III-V semiconductor nanowires (NWs). However the epitaxial growth of perovskite oxides on III-V NWs is still rather challenging. By the As capping / decapping method (Figure 1a), the GaAs NWs (or GaAs / AlGaAs NWs) could be transferred among different separated reactors without oxidizing or contaminating the GaAs (or AlGaAs) facets [3], which is of great necessity for the further functional oxide shell growth. Then, by means of the two-steps SrTiO₃ growth method [4], we have obtained a partially oriented SrTiO₃ shell covering the GaAs NWs (Figure 1b), proving the possibility to fabricate the monocrystalline epitaxial shell of functional oxides integrated on semiconducting NWs [5]. As revealed by XPS spectra and TEM images, the interface is not abrupt, which probably prevents a perfect epitaxial growth. The GaAs / SrTiO₃ NWs also showed a good thermal tolerance to an annealing up to 500 °C, benefiting the further growth of another functional oxide, such as the ferroelectric BaTiO₃, to construct the core / multi-shells NW array and the further practical devices.





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Tunable morphology and pH-dependent doping of ZnO nanowires grown by chemical bath deposition

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Keywords: ZnO nanowires, pH, doping, physico-chemical processes in aqueous solution.

Over the last decade, ZnO nanowires (NWs) have been used for a wide variety of sensing, electronic, and optoelectronic devices. For all these applications, their electrical properties (i.e. conductivity and mobility) should be controlled as much as possible. ZnO is intrinsically n-type owing to the high density of zinc interstitials and hydrogen and can intentionally be n-doped, for example, by aluminium. The doping of ZnO NWs has however been mainly performed by vapor phase deposition techniques and is still a major issue by solution deposition techniques. In the present work, ZnO NWs are doped with aluminium by using the low-cost, low-temperature, and easily implemented chemical bath deposition (CBD) technique. Aluminium nitrate is added to the standard precursors (i.e. zinc nitrate and HMTA [1]) in deionized water and the $[Al(NO_3)_3] / [Zn(NO_3)_2]$ ratio is varied from 0 to 10 %. It is shown by scanning and transmission electron microscopy (TEM) that this addition completely modifies the structural morphology of ZnO NWs [2]. The formation mechanisms are thoroughly investigated and supported by thermodynamic simulations yielding theoretical solubility plots and speciation diagrams. Their dependence on the pH of the solution through the addition of ammonia is further studied thoroughly [3]. The incorporation of aluminium is eventually investigated by energy dispersive x-ray spectroscopy using scanning TEM and scanning probe microscopy. Raman spectroscopy measurements show the occurrence of additional modes, revealing the thermally activated aluminium-doping of ZnO NWs from an annealing temperature of 200°C [2,3].



Figure 1: Schematic diagram of the physico-chemical processes (left). Raman spectra of ZnO NWs with varying $[Al(NO_3)_3] / [Zn(NO_3)_2]$ ratios from 0 to 7% (right).

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Structural properties and ordering studies in Zn_{1-x}Mg_xO alloys grown by MOVPE

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Keywords: ZnO nanowires, ZnO/ZnMgO core/shell_2, Order_3, Lateral QW_4

To study the influence of the growth parameters on optical and structural properties of ZnO based core/shell heterostructures, as well as the ordering in $Zn_{1-x}Mg_xO$ alloys (0 < x < x0.30), ZnO vertical nanowires have been first grown by metal-organic-vapor-phase-epitaxy on sapphire substrates. The use of N₂O and diethylzinc DEZn as oxygen and zinc precursors combined with high temperature (> 600 $^{\circ}$ C) led to the spontaneous growth of ZnO nanowires (NWs) on underlying three dimensional islands present at the bottom of each nanowire. Wurzite ZnO nanowires are grown free of extended defects, along the c axis, and exhibit mtype, non-polar facets. This configuration is favorable to band-gap engineering since Stark effect is not observed when heterostructures are grown on non-polar surfaces, thus leading to efficient optical band-gap recombination. Subsequently, we have grown a series of radial ZnO/ZnMgO core/shell structures with different shell thicknesses and Mg concentrations. For these experiments, we have used oxygen, (MCp)₂Mg and DEZn as oxygen, magnesium and zinc precursors. In addition, ZnMgO reference layers have been grown in the same run on both c-axis Al₂O₃ and ZnO substrates. Low temperature photoluminescence spectroscopy and Transmission Electron Microscopy equipped with EDX were used to estimate the Mg concentrations in the shells and layers, which can reach x=0.2. Micro-photoluminescence performed on single ZnO/ZnMgO quantum well exhibit excellent luminescence properties with a clear emission of the ZnO quantum well. Surprisingly, both electron diffraction on the core/shell nanowires (Fig.1) and high resolution X-ray diffraction on the reference ZnMgO layers (Fig.2) reveal the (001) reflection which is forbidden in a perfect disordered alloy. The appearance of such (001) diffraction suggests that Zn and Mg atoms distribution in the Zn_{1-} $_{\rm x}$ Mg_xO alloy is, at least partially, ordered. So, we have investigated the order-dependent versus Mg content in ZnMgO films.



Figure.1. electron diffraction pattern of $Zn_{1-x}Mg_xO(x=0.13)$ core/multi-shells



Figure.2. X-ray diffraction spectrums of $Zn_{1-x}Mg_xO$ layer (x=0.17).

Mass production of silicon nanowires

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Mots clés : Silicon nanowires, growth mechanism, doping, yield optimization

First produced by thin film technologies (CVD growth or etching), silicon nanowires (SiNWs) have shown great promises in nanoelectronics, sensors and energy storage. For the latter application, high quantities of SiNWs are required. We recently patented [1] a new technology of SiNW synthesis that allows for the preparation of large quantities of SiNWs in bulk in a small, simple reactor within a few hours at medium temperature (450°C). Our SiNWs are grown from metal nanoparticles deposited on an unreactive nanopowder of NaCl from an air-stable organosilane oil as Si source. After synthesis, the NaCl powder is dissolved in water to recover a dense mat of pure SiNWs. The process is currently scaled-up in the lab and for industrial applications in the start-up company ENWIRES: 200mg SiNWs in a 100mL steel reactor, grams in a 1L reactor.

NaCl particles play a critical role as a "solid solvent", keeping catalysts available to reactive gases and apart from each other during growth. Bulk grown SiNWs behave differently from SiH₄-fed CVDgrown SiNWs: long and very thin (10nm in diameter), they are strongly hydrophobic and show a low oxygen content even after exposure to air. We studied the sub-products to improve the yield of SiNW growth. It appears that several reactions compete for the Si source in the reactor, in which the organic part of the organosilane reagent plays a significant role. The organosilane reactivity offers new ways of tuning SiNW growth and physical properties.

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MOCVD GROWTH AND OPTICAL CHARACTERIZATION OF ZnTe/ZnMgTe CORE-SHELL NANOSTRUCTURES

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Keywords : ZnTe, nanowires, VLS, MOCVD

ZnTe is a direct band-gap semiconductor, 2.26 eV at room temperature. It is suitable for various optoelectronic devices such as pure-green light-emitting diodes, green laser diodes, and UV-green photodetectors. For realizing high-efficiency light-emitting devices based on ZnTe nanowires (NW), an appropriate coating shell material must be used to passivate the NW surface. It has been demonstrated that ternary $Zn_{(1-x)}Mg_xTe$ alloy is a possible candidate for the shell, since it has a wider band-gap energy than ZnTe. A magnesium concentration of 10% should improve the optical properties of the core-shell nanostructures.

In this context. ZnTe **NWs** and ZnMgTe/ZnTe core-shell nanostructures were synthesized by MOCVD at atmospheric pressure using a vapor-liquid-solid (VLS) gold-catalyzed method. The morphologies of the structures have been investigated as a function of growth temperature, growth time, gold droplet size and II/VI gases pressure ratio. Scanning electron microscopy (SEM) combined with EDS system were used to characterize the crystal structure and the chemical composition of the NWs. Photoluminescence (PL) measurements at 4K were carried out on both 2D ZnMgTe/ZnTe layers and 1D ZnMgTe/ZnTe core-shell nanostructures to study the effect of the passivation and the shell layer thickness.



Figure 1: SEM cross-section images from ZnTe nanowires grown on GaAs substrate for different time growth.

For 2D layers, PL results revealed that for thin ZnTe layer (250 nm), the near band edge (NBE) emission line is completely missing while for a thick ZnTe layer (2 μ m) the NBE luminescence can be observed. This could be explained by the fact that the free carriers are lost at the surface due to non-radiative recombinations, and that phenomenon dominates in the case of very thin structures. However, when coated with ZnMgTe shell, the thin ZnTe layers (250 nm) show an intense emission line at 2.36 eV, i.e. in the NBE emission region of ZnTe. Therefore, the ZnMgTe capping layer has efficiently passivated surface states at the origin of non-radiative recombination of photo-created carriers. Regarding to ZnTe NWs and ZnMgTe/ZnTe core/shell nanostructures, no luminescence have been detected, which may be explained by the diffusion of

gold in the ZnTe nanowire during the VLS growth process, leading to non-radiative deep level defects in the material.

Polarity-dependent selective area growth and physical properties of ZnO nanorods by chemical bath deposition

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Keywords: ZnO nanorods, chemical bath deposition, polarity, growth rate, selective area growth.

The polarity in ZnO is a consequence of the non-centrosymmetric wurtzite structure, resulting in a spontaneous polarization along the c-axis [1]. While it is known to affect the growth and properties of ZnO epitaxial films, its effects are mostly unknown in nanorods (NRs) [1]. In contrast to vapor phase deposition techniques, ZnO NRs grown by chemical bath deposition (CBD) can be of either O- or Znpolarity, opening the way for more deeply investigating these effects [2]. In this context, we thoroughly address the issue of the polarity-dependent growth and properties of ZnO NRs by CBD following the selective area growth approach [3]. To leave polarity as the only varying parameter, ZnO NRs are grown under identical conditions and during the same run of experiment on O- and Zn-polar ZnO single crystals patterned by electron beam lithography with the same pattern consisting of 15 different domains. Well-ordered O- and Zn-polar ZnO NW arrays with high structural uniformity are formed on all the domains. The comparison of their typical dimensions unambiguously reveals that Zn-polar ZnO NRs have significantly higher growth rates than O-polar ZnO NRs [3]. The distinct growth rates are explained in the framework of the surface reaction-/diffusive transport-limited elongation regime analysis, which yields a much larger surface reaction rate constant for Zn-polar ZnO NRs. The origin of the difference is discussed in the light of surface configurations and interactions in aqueous solution at the top polar c-faces of the ZnO NR. Additional electrical characterizations in different configurations using 4-point probe resistivity measurements are performed on single O- and Zn-polar ZnO NRs to study their physical properties.



Figure 1: FESEM images of ZnO NRs grown by CBD on patterned Zn- and O-polar ZnO single crystals for a hole size of 125 nm and five different hole periods. The top and bottom triangles correspond to the growth on O- and Zn-polar ZnO single crystals, respectively.

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DETERMINATION OPTIQUE DE LA POSITION DE BOITES QUANTIQUES SEMICONDUCTRICES INTEGREES DANS UN FIL PHOTONIQUE

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Mots clés : Source de photons uniques ; Boîte quantique semiconductrice ; Microphotoluminescence ; Imagerie de Fourier.

Le développement de sources de photons uniques, composants capables d'émettre à la demande des impulsions lumineuses contenant exactement un photon, fait l'objet d'efforts soutenus depuis une dizaine d'années, motivés par des perspectives d'application dans les domaines de la métrologie optique ou les communications quantiques. Dans ce contexte, notre équipe a introduit une approche originale, en intégrant une boîte quantique semi-conductrice au sein d'un guide d'onde en forme de nanofil [1,2]. En pratique, ces dispositifs sont réalisés en gravant une structure planaire contenant un

seul plan de boîtes quantiques auto-assemblées. La position latérale des émetteurs dans la section du guide d'onde est donc aléatoire. Or, cette position conditionne la brillance de la source, et surement ses propriétés spectrales fines.

Ce travail présente une méthode tout optique (et donc non destructive), permettant de déterminer la position radiale et azimutale de boîtes quantiques intégrées dans un fil photonique [3]. Nous exploitons ici deux modes guidés qui présentent des profils spatiaux très différents. La fraction d'émission spontanée dans chacun de ces modes dépend fortement de la position de la boîte quantique. En mesurant la structure du champ lointain émis par une seule boîte, il est alors possible de déterminer sa position.



Figure 1: Image MEB de l'antenne optique (gauche) et les champs lointains associés à deux boites quantiques (BQs) (droite).

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VERTICAL CDSE/ZNSE NANOWIRE-QUANTUM DOTS AND ENHANCED PHOTON EXTRACTION WITH A BOTTOM-UP PHOTONIC SHELL

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Mots clés : Nanowire, Quantum dot, photonic wire

Semiconductor nanowires (NWs) offer the possibility to grow high quality quantum dot (QD) heterostructures, and in particular CdSe QDs inserted in ZnSe NWs have demonstrated the ability to emit single photons up to room temperature. Taking advantage of the NW geometry it is possible to form photonic wires to extract more efficiently the photon from the QD. In this case the growth of vertically oriented NWs is compulsory, as well as the control of the optical dipole orientation through the QD aspect ratio.

We have grown vertically oriented CdSe/ZnSe NW-QDs (with typical diameter of 10 nm) by molecular beam epitaxy on a ZnSe(111)B buffer layer with a ZnMgSe passivating shell to increase the (otherwise weak) QD luminescence. We managed to obtain a low NW density (~ 1 NW/4 μ m²) so that single NW-QDs can be directly studied on the as-grown sample. Exciton, biexciton and charged exciton lines are clearly identified in single NW-QDs. Then we fabricate a photonic fiber-like structure around the NW-QDs by depositing a conformal Al₂O₃ oxide shell using atomic layer deposition [2]. Combining micro-photoluminescence and decay time measurements, we evidence an inhibition effect of the QD emission in the NW, indicating that the optical dipole is orthogonal to the NW axis. With an oxide shell of 110 nm thick we obtained a 4-fold increase in the collected photoluminescence from the QD. Numerical simulations suggest that the intensity collected in our NA=0.6 microscope objective can be increased up to a factor 7 with respect to the bare NW

case. We show that this improvement is due to an increase of the QD emission rate and a redirection of the emitted light [3]. Our ex-situ bottom-up fabrication technique allows a precise and reproducible fabrication on a large scale. Its improved extraction efficiency is compared to state of the art top-down devices.



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X-RAY FOR NITRIDE WIRES

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Mots clés : fils de nitrures, diffraction de rayons X, luminescence, composition

One-dimensional nitride heterostructures demonstrated novel optical and electronic properties making use of quantum confinement effects and strain engineering. The emergence of disruptive functionalities is now related to the growth and technology controls [1-5], but also to the development of advanced characterization techniques having high spatial resolution. To complement the information provided by usual grazing incidence diffraction techniques, focused X-ray beams provide innovative solutions to analyse quantitatively the morphology, defects, strain and composition of these materials. Recent breakthroughs obtained at the European synchrotron radiation facility (ESRF) on nitride wires and core-shell heterostructures grown by Metal Organic Vapour Phase Epitaxy will be presented.

The structure of single defects such as Inversion Domain Boundaries (IDB) inside ndoped GaN wires can be extracted from the *X-ray coherent diffraction imaging* with a monochromatic beam [6] with an unprecedented accuracy. The complex 3D IDB configuration inside a single wire is measured without any slicing in contrast to electron microscopy and the lattice displacements along/across the wire length is deduced from the analysis of the Bragg peak intensity by *phase retrieval* methods (with pm resolution), in full agreement with electronic structure *ab initio* calculations [7].

GaN/InGaN Multiple Quantum Well (MQW) core-shell heterostructure grown on mplane sidewalls of c-axis GaN wires [2-5] are analysed by X-ray excited optical luminescence (XEOL) and X-ray Fluorescence (XRF) with multimodal hard X-ray nanoprobe [8]. Blue light emission is measured in the spatiotemporal domain with 50 ps resolution to get the photoluminescence time decay (\approx 100 ps) and is related the electron confinement/local composition in the wire [8]. Optical luminescence is also related to Si incorporation in Npolar wires.

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DOPING PROFILES BY SCANNING SPREADING RESISTANCE MICROSCOPY ON CORE-SHELL p-n JUNCTION IN AIGaAs NANOWIRES

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Mots clés : SPM, SSRM, Nanowire, core-shell, GaAlAs, doping

Measurement methods to quantitatively measure the doping in semiconductor nanowires (NWs) are strongly requested in such one-dimensional nanostructures. Scanning spreading resistance microscopy (SSRM) based on atomic force microscopy, has emerged as a promising tool for a two-dimensional and high resolution carrier/doping profiling.

Vertical NWs consisting of of a p-type AlGaAs core and a n-type AlGaAs shell were grown on a silicon (111) oriented substrate for photovoltaic cell applications ^[1,2]. After setting the sample preparation process using silica dip-coating filling of the vertical NW field, contact-tip AFM and SSRM measurements were performed and we have obtained images as shown in the figure 1. The topographic image shows a surface roughness sufficiently low to be compatible with those needed for the AFM electrical contact mode. We can observe the hexagonal shape of the emerging AlGaAs NWs. The SSRM image shows a clear contrast in between the NWs and the silica, pointing out the conductive properties of the doped NWs. From the measured SSRM resistance value into the NW core, we could estimate the p-type doping in the 10¹⁶ cm⁻³ range which is two orders lower than the targeted doping. The 10 nm thickness of the n-type shell is too low to be resolved by SSRM. An electrical model of the core-shell p-n junction will be discussed in order to interpret these results.



Figure 1: (a) Topographic image of the surface after the sample preparation, b) Image of the resistance measured by SSRM, c) SSRM resistance measured along the line drawn in blue on b).

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Cartographie de la polarisation d'une couche d'amorce de nanofils de ZnO par les techniques de la microscopie en champ proche

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Mots clés : Nanofils ZnO, Microscopie à Force Atomique, Piezoresponse Force Microscopy.

La mesure statistique de la polarité des couches d'amorce et des nanofils de ZnO crûs par dépôt en bain chimique à partir de ces couches est de première importance pour leur intégration dans de futurs composants. La polarité influence en effet grandement les mécanismes de nucléation et de croissance, les propriétés physiques ainsi que les performances des composants [1]. Cependant, la mesure très locale de la polarité possède également un intérêt certain. Un des moyens de mesurer cette polarité à l'échelle locale, avec une résolution spatiale nanométrique, est d'utiliser les techniques issues de la microscopie à force atomique (AFM), notamment celles qui tirent profit de l'effet piézoélectrique inverse pour dresser une cartographie de l'activité piézoélectrique des couches minces et des cristaux. Nous présentons ici les résultats d'une cartographie de ce type, obtenue par Piezoresponse Force Microscopy (PFM), sur des échantillons de différentes compositions [2]. Nous montrons qu'il est possible d'obtenir la polarisation individuelle de chaque grain composant la couche d'amorce, et d'obtenir des informations sur la répartition des polarités de l'échantillon même si la proportion de l'une d'elles est très faible, grâce à la résolution spatiale de ce type de techniques. L'analyse d'image permet d'obtenir très rapidement une statistique locale des deux polarités opposées et est corrélée à des mesures statistiques effectuées par diffraction anomale de rayons X au seuil du zinc employant le rayonnement synchrotron [2].



Figure 1: Topographie de la couche d'amorce obtenue par AFM (gauche). Cartographie de la direction de polarisation (zones sombres: polarité O, zones claires: polarité Zn) (droite).

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Elaboration et caractérisation de nanofils Ge_xSn_{1-x} par CVD via le mécanisme VLS

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Mots clés : Nanofils, GeSn, CVD-VLS, croissance, caractérisations physico-chimiques.

Depuis les débuts de l'électronique, l'augmentation des performances des composants suit de manière prodigieuse la conjoncture de Moore. Cependant, depuis quelques années, la tendance à la miniaturisation arrive à ses fins et les recherches se tournent de plus en plus vers l'intégration 3D et/ou les dispositifs à basse consommation. C'est dans ce contexte « more than Moore » que l'alliage GeSn s'inscrit, prédisant un gap plus faible, une mobilité des porteurs plus importante et une masse effective h^+ plus faible que le silicium ou le germanium ainsi qu'une transition du gap indirect vers un gap direct pour des concentrations d'étain supérieurs à ~8% [1]. Cet alliage du groupe IV se montre alors particulièrement intéressant pour beaucoup d'applications, autant photonique (détecteurs IR, LEDs ...) qu'électronique (TFET [2], p-MOSFET). Cependant la faible solubilité de l'étain dans le germanium (< 1%) ainsi que la grande différence de paramètre de maille (≈14%) rendent la croissance de ce matériau difficile et mènent à des défauts structuraux lors de la croissance de couches 2D. Une des solutions permettant de s'affranchir de ces défauts est la croissance directe de nanofils par le dépôt chimique en phase vapeur (CVD) via le mécanisme « vapor-liquid-solid » (VLS). En plus, les nanofils offrent une relaxation des contraintes ainsi qu'une bonne qualité cristalline. Nous présentons dans ce travail l'élaboration et la caractérisation de nanofils GeSn. Ces nanofils ont été obtenus par CVD-VLS en utilisant l'or comme catalyseur (colloïdes d'or de 100 nm), le GeH₄ et le SnCl₄ comme précurseurs. La morphologie et l'incorporation de l'étain dans les nanofils sont étudiés par MEB, Raman, Nano-Auger, HRTEM et STEM EDX en fonction du rapport de pression partielle des flux P_{SpCl4}/P_{GeH4} et de la température de croissance.

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Yellow MOVPE grown InGaN/GaN nanowire light-emitting diodes

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Key words: InGaN/GaN Nanowires, LEDs, green and yellow electroluminescence.

Core-shell InGaN/GaN multi quantum well nanowires (NWs) grown by metal-organic vapor phase epitaxy (MOVPE) can be optically active in a spectral range from the near UV to the green with applications to light emission, photodetection and photovoltaic conversion [1,2,3]. However, MOVPE grown InGaN present limitations at the lower energy part of the visible spectrum because the growth of defect-free structures with high In content is very challenging.

In this contribution, we report on the optoelectronic properties of MOVPE grown core-shell InGaN/GaN NW LEDs emitting in the 500-650 nm range. The NW arrays and single wire devices are characterized by correlative photo- and electro-luminescence spectroscopy. The optical properties are correlated with the structure and composition and are discussed in terms of charge carrier transport in the core/shell system.



Figure 1: Electroluminescence (EL) spectra acquired from an active NW LED for (a) 15 mA and (b) 30 mA injection current and EL signal as seen by a CCD camera

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GaN/InAlN multiple quantum well tubes

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Mots clés : Nitrides, GaN wires, core-shell MQW, tubes, MOVPE, UV light

Nanotubes are nowadays well-known nanostructures, but exhibit no or modest light emission. Indeed, the surface states and defects favor the non-radiative recombinaison that quench light emission. To overcome this problem, we have develop a new type of nanotubes with walls only composed of multiple quantum wells (MQWs), acting as the active region for light emission. The quantum wells composed of multiple nanometer-sized layers are well known for their remarkable light emission properties due to quantum confinement effects and are daily used in LEDs or lasers.

To successfully produce such MQW-tubes, GaN wires with core-shell GaN/InAlN MQW heterostructure grown by metal-organic vapor-phase epitaxy are simply annealed under H_2/NH_3 gas mixture [1]. This selective annealing only etches the inner GaN wire leading to tubes composed of MOW.

MQWs. We demonstrated by ToF-SIMS measurements that the tube walls was actually composed of the GaN/InAlN heterostructure proving that the annealing did not degrade the quantum wells. Such smart MQW-tubes exhibit excellent light emission in the UV range (around 330 nm) until room temperature. The simplicity of the method to fabricate thin nitride tubes with embedded MQWs opens routes for the development of novel tube-based devices ranging from emitters to chemical and biological sensors.



Figure 1: Nitride tubes with GaN/InAlN wells chemical signature in the tube wall observed by ToF-SIMS and optical features with UV emission by PL at 5K.

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DSA-based process for InAs horizontal nanowires in sub-10nm FET

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Keywords: PS-b-PMMA, directed self assembly (DSA), InAs nanowires

ABSTRACT

To extend their downscaling while keeping their efficiency, transistors evolve towards new architectures and new channel materials. III-V CMOS technology has gathered a lot of researcher's efforts in order to access the high electron mobility of III-V compounds, such as InAs or InGaAs.

In this study, we realized horizontal InAs nanowires (NW) for sub-10nm node NW transistors. We used a PS-b-PMMA 30 nm-period block copolymer (BCP) as a mask for the etching of an InAs epitaxial layer.

A 25 nm thick InAs epitaxial layer has been grown by MOCVD on a GaSb buffer layer on (001)-Si substrate. Then a PS-b-PMMA BCP has been deposited onto InAs surface. Under optimized deposition conditions, BCP self-aligned between confinement pattern¹. PMMA is then etch selectively to the PS with a pulsed H_2/N_2 plasma². The remaining PS will act as an etching mask. Next, the InAs is etched under BCl₃/SiCl₄/Ar plasma at high temperature³.

We obtained a high density of 20 nm InAs fins. To pursue, the GaSb etch has to be developed in order to obtain suspended InAs nanowires between the Au pads. We will then be able to develop the gate-all-around process to obtain InAs n-channel multi-NW (Gate All Around) GAA transistors.

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Green emission from In-rich core-shell InGaN/GaN MQWs along GaN nanowires

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Keywords: Nanowires, green emission, flexible LED

A lot of attention has been focused on the $\pm c$ -axis GaN nanowires encircled by core-shell InGaN/GaN multiple quantum wells (MQWs) for the fabrication of efficient light emitting diodes (LEDs). The green emission still remains a challenge for this core-shell geometry due to the usual lower In incorporation in *m*-plane wire sidewalls with respect to *c*-plane. In this work, a green emission of such self-assembled MQWs system is achieved along with the demonstration of a flexible LED [1]. A complete investigation has been performed combining structural and optical analysis to address the open issues for green emission.

Self-assembled catalyst-free microwires have been grown by metalorganic vapor phase epitaxy with silane addition on sapphire *in situ* capped with SiN_x mask. This was followed by core-shell MQW growth [2] performed with decreasing QW growth temperature (750-620°C) to tune the In-content. The core-shell morphology is preserved even for the lowest temperature. An emission is observed in the green region (500-550 nm) by photo- and cathodoluminescence only for the wires with QWs grown at 650°C. This optimized temperature corresponds to a balance between In-content and defect formation. As revealed by transmission electron microscopy (TEM) and atomic probe topography (APT), the In content is estimated to be (23±2) %, thus giving rise to green emission.

Following the success of achieving green emission with self-assembled wires, a further attempt can be made to fabricate green LEDs with selective area growth (SAG) of InGaN/GaN NWs. The growth of organized wires can help in homogenous emission which will contribute to the better efficiency of the LED. Thus, the advantages of using selective area growth of nanowires over self-assembled wires can be further discussed to achieve the improved flexible devices in terms of homogenous emission and efficiency.

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Influence des paramètres de synthèse sur la structure des nanofils de ZnO pour la fabrication de nanogénérateurs piézoélectriques

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Mots clés : Nanofils, ZnO, piézoélectricité, nanogénérateur, microsource d'énergie.

Actuellement, la recherche scientifique se focalise sur la miniaturisation et le développement de systèmes autonomes en énergie. De ce fait, des récupérateurs d'énergie directement intégrés sur des batteries, tels que les nanogénérateurs piézoélectriques (NGs) fabriqués à partir de nanofils (NFs) d'oxyde de zinc (ZnO), sont apparus ces dernières années comme une solution à fort potentiel. ceci essentiellement grâce à une croissance des NFs par synthèse aqueuse, une méthode peu coûteuse et industrialisable à grande échelle [1]. Les paramètres de synthèse doivent alors être choisis afin de limiter les défauts dans le matériau, responsables d'une importante diminution des performances du dispositif [2]. L'objectif de cette communication est de comparer deux méthodes de synthèse par voie aqueuse et de voir leur influence sur la quantité de défauts dans les nanofils de ZnO, ainsi que la corrélation avec l'énergie récupérée par un NG. La méthode de croissance, notée A, est présentée en détail dans [3]. La méthode de croissance B a pour principale différence l'ajout d'une plus grande concentration d'ammoniaque [4], ainsi que le chauffage qui se fait par bain d'huile, et la croissance qui s'effectue sur une couche d'ensemencement de ZnO. Des mesures par photoluminescence (PL) (Fig. 1a.) effectuées sur des NFs de même longueur ont permis d'identifier une quantité moindre de défauts par la méthode A. Le rapport d'intensité des pics UV/visible, représentatif de la quantité de défauts [5], vaut 2.10⁻² pour la méthode A, contre 3.10⁻³ pour la B. Parmi les hypothèses avancées pour tenter d'expliquer ce résultat : la vitesse de croissance beaucoup plus rapide (25-50 nm/min pour B contre 3 nm/min pour A), pourrait impacter la cristallisation et la structure des nanofils. Le pH du milieu, très basique pour la méthode B (>11,5), pourrait également dégrader les NFs en croissance [6] . Afin de vérifier l'influence des méthodes de croissance sur les propriétés du ZnO, des NGs ont été fabriqués [3] (Fig. 1b.) et caractérisés électriquement à faible force (3N) sur le même banc de test. Les résultats présentés Fig. 1c montrent qu'actuellement les NGs obtenus avec la méthode A donnent une puissance maximale 1,5 fois plus grande que ceux obtenus selon la méthode B.



Figure 1: a. Mesures par PL pour les 2 méthodes **b.** Architecture d'un NG **c.** Puissance maximale moyenne pour une série de 3 NGs avec la méthode A et 5 NGs avec la méthode B

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Development and characterization of flexible/non-flexible energy transducers based on ZnO nanowires

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The present work focuses on fabrication and characterization of piezoelectric ZnO nanowires (NWs) based energy transducers - Vertical integrated nanogenerator (VING) - using flexible stainless-steel foil of different thickness and non-flexible Silicon substrates. The NWs are grown using solution growth method with dimensions 200 ± 50 nm in diameter and 3.5 ± 0.3 µm in length confirmed by Scanning Electron Microscopy (SEM). These NWs are then integrated into devices by coating PMMA as dielectric and top layer insulation. The output of these devices are analysed by applying tensile and/or compressive loading (stress) based on the flexibility of the device using specific characterization set-ups. The resultant output of these devices is 170mV for ~6mm bending-amplitude (flexible) and 90 mV for 1.5N compressive force (non-flexible).



Figure 1: Vertical integrated nanogenerator (VING) structure Figure 2: SEM cross-sectional view



Figure 3: Characterization setups for flexible and non-flexible devices

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Capteurs piézoélectriques capacitifs souples à base de fils de GaN : principes et réalisations

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Des fils GaN crûs par épitaxie en phase vapeur à base d'organométalliques (MOVPE) sont utilisés pour la fabrication de capteurs piézoélectriques souples dans une structure capacitive. Ces fils majoritairement de polarité N, (orientation (–c)) par rapport au substrat de saphir) possèdent une forme légèrement conique à section hexagonale et une longueur comprise entre 10 et 700 μ m [1]. La morphologie des fils peut être contrôlée par les conditions de croissances, à savoir l'addition de silane, la température, la pression ou le flux de précurseurs et bien sûr le temps.

Des capteurs piézoélectriques souples sont réalisés en intégrant verticalement les fils de GaN dans une couche diélectrique souple de PDMS contactée de part et d'autre par des électrodes métalliques formant ainsi une structure capacitive. Cette architecture permet de conserver l'orientation relative issue de la croissance et de s'affranchir des problématiques liées à l'orientation aléatoire des fils qu'on retrouve dans les dispositifs à fils horizontaux [2].

L'étude de l'influence des paramètres géométriques des fils sur le rendement des capteurs montre que l'utilisation de fils longs (>100 μ m) permet d'améliorer le rendement des capteurs et leur sensibilité étant donné qu'ils sont plus sensibles à la variation de la force comparés aux fils courts (<60 μ m). Ces résultats, issu de simulations en éléments finis, ont été confirmés expérimentalement par la caractérisation électrique de plusieurs capteurs fabriqués selon un procédé simple et facilement reproductible [3].



Figure 1. (a) Image MEB de la couche diélectrique contenant les fils, avec en inset une image du dispositive. (b) et (c) Signaux électriques fournis par un dispositif contenant des fils de 190 et 70 µm respectivement en excitation manuelle. (d). Signaux électriques fournis pour une excitation de 1000 cycles.

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Random and periodic arrays of GaAs/InGaAs core-shell nanowires for photovoltaic applications

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Mots clés : GaAs nanowires, self-catalyzed growth, core-shell nanowires, photovoltaics.

We present self-catalyzed growth experiments for the fabrication of GaAs-based nanowire arrays for photovoltaic applications on Si(111) substrates, covered either with native oxide or pre-patterned with arrays of nano-apertures on SiO₂. In order to remove any uncertainties related to the varying native oxide characteristics from one wafer to the other, we have developed a controlled chemical oxidation process to replace the native oxide on Si(111) substrates with a reproducible chemical oxide [1]. A high yield (>90%) of vertical GaAs nanowires is achieved with excellent uniformity on chemical oxide-covered substrate. We show that the crystalline quality is significantly improved over that of GaAs nanowires grown on native oxide-covered substrate, and pure zinc blende crystal structure can be achieved with minimal defects. The chemical oxide can be used as a template for producing in parallel different combinations of nanowire densities and sizes on the same wafer, or as a surface treatment to improve the yield and selectivity of vertical nanowires in the nano-apertures of a periodically-patterned oxide mask. Finally, we assess the material quality, Indium incorporation and optical properties of GaAs/InGaAs core-shell nanowire arrays grown using the chemical oxide method, and we present results from preliminary nanowire *p-i-n* diode devices.



Figure 1: (*Left*) Array of GaAs nanowires grown on a chemical oxide. (*Right*) Cross-section of a core-shell GaAs/InGaAs nanowire observed in a high resolution TEM experiment.

Référence : [1] S. L. Tan et al., *Nanotechnology* **2017**, 28, 255602-13.

ZnO NANOWIRE BASED MICROFLUIDIC SYSTEM FOR PHOTOCATALYTIC WATER PURIFICATION

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Mots clés : ZnO nanowires ; Microfluidics ; Water purification.

In recent years, the use of semiconductor (SC)-based photocatalysts to degrade organic compounds in contaminated air or water; has been extensively studied due to their low-cost and environmental-friendly nature. With high surface/volume ratio, the nanostructured SC shows enhanced photocatalytic efficiency in drinking water and wastewater treatment [1, 2]. In this work, we used ZnO nanowire (NW) integrated microfluidic system as a micro-reactor for water purification. The photocatalytic efficiency has been enhanced for threes dyes (methylene blue-MB, methyl orange-MO & acid red 14-AR14) as well as for VOCs-polluted water purification. ZnO NW samples have been prepared using two-step hydrothermal method [3]. Fig.1 (a-f) show the photodegradation effect of three dyes: after ~3h UV irradiation, the degradation rate reaches 86%, 49% and 93% for MB, MO & AR14 respectively. However, by using the micro-reactor with integrated ZnO NWs (Fig.1(A)), the same degradation rate was reached after only 6-7 min photocatalysis (Fig.1(g-i)). In order to confirm the photocataltic efficiency of the ZnO-based microfluidics system, an even smaller micro-reactor has been realized with *in-situ* grown ZnO nanostructures (Fig.1(B)). The same initial concentration dyepolluted water needs only one-pass (few seconds) to reach quasi total degradation. This microfluidic system showed also high photocatalysis efficiency for the VOCs-polluted water purification.



Figure 1: Adsorption spectra of MB (a, b), MO (c, d) and AR14 (e, f) 10 mM solutions in the presence of the ZnO NWs under exposure to UV. (g-i) Photodegradation performance of microfluidic system. (A & B) Schematic illustration and photography of two types of used micro-reactors devices. **References:**

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TRANSITION-METAL DOPED ZnO NANOWIRES FOR PHOTOCATALYTIC WATER PURIFICATION

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Mots clés : ZnO nanowires ; Transition-metal doping ; Photocatalytic activity.

In this research work, ZnO nanowires doped with Fe³⁺, Ag⁺, Cr³⁺, and Co²⁺ ions were synthesized using a facile and inexpensive hydrothermal method [1]. The transition-metal ions successfully substituted Zn^{2+} in the ZnO crystal lattice without changing the morphology and crystalline structure of ZnO nanowires, confirmed by SEM and DRX measurements. The atomic percentages were 1% and 3% for each doping element, in the doped ZnO nanowires. The photocatalytic results showed that Fe-doped, Ag-doped, Cr-doped, and Co-doped ZnO nanowires all exhibited higher photocatalytic activities than undoped ZnO nanowires. Among the doped ZnO nanowires, Fe-doped ZnO is considered a more efficient photocatalyst material. Furthermore, iron is an environmental-friendly material. The photocatalytic performance was investigated by methyl orange (MO) degradation with a Mercure-Xenon combined lamp (UV-Visible). According to our previous work [2], also confirmed by the literature, MO is one of the most difficult dyes to degrade by photocatalysis, compare to the other dyes (ex. MB and AR14). The enhancement of the photocatalytic performance originates from the doped metal ions, which enhance the light absorption ability and inhibit the recombination of photo-generated electronhole pairs as well. The effect of the doping element concentration also investigated. The obtained results showed that the 3% doping exhibited a better photocatalytic performance than 1% doping.



Figure 1: (a) Schematic illustration of the photocatalytic performance enhancement of transition-metal doped ZnO nanowires. (b) Photodegradation performance for 10 mM MO polluted water of the ZnO NWs doped @ 1% by Co, Fe, Cr, Ag, and undoped ZnO NWs, respectively.

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GAS SENSING PERFORMANCE ENHANCEMENT OF TRANSITION-METAL DOPED ZnO NANOWIRES

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Keywords: ZnO nanowires; Transition-metal doping; Gas sensing

Due to human activities, the presence of toxic gases in the atmosphere continues to increase. Gas sensing and air purification become permanent challenge for the humanity. On the other side, ZnO is a very promising material due to its excellent and versatile intrinsic properties, making it suitable for wide range applications such as, transparent conductors, optoelectronic, photocatalyst, and gas sensors devices [1]. In our previous work, we demonstrated the high sensitivity of ZnO nanostructure for ethanol, acetone, and ammonia detection at low temperatures [2]. The aim of this work is to study the enhancement of the gas sensing performance of doped ZnO nanowires including classical nanowire array (NWs) and nanowires/nanofibers (NWs/NFs) homohierarchical nanostructures, both synthesized via low cost hydrothermal method. Both two type nanostructures were doped with four transition metals (Ag, Cr, Co and Fe) in 1% and 3%, via hydrothermal process. Ethanol (EtOH) and Acetone (Act) have been used for gas sensing test, monitored both by UV-Vis spectrophotometry on the ZnO sample surface and by FTIR spectroscopy in the reactor media.

The results showed an improvement of the gas sensing sensitivity for the ZnO hierarchical nanostructure owing to its larger specific surface area. Moreover, the transition-metal doped ZnO nanostructures showed an enhancement of the gas sensing efficiency. The best performances for the acetone sensing were obtained with Co-doped ZnO nanostructures, the pollutant removal rate increased from ~34% to 76% for Co- doped ZnO NWs and to 78% for Co-doped ZnO NWs/NFs, as shown in Figure 1d; while for the ethanol sensing, the best results were obtained with Ag- and Fe-doped ZnO NWs: X increased from 28% to79% and 88%, respectively.



Figure 1: (a) & (b) Ethanol and acetone sensing performance study for transition-metal doped ZnO NWs by UV-Vis absorption spectrometry; (c) Acetone sensing performance for undoped and 3% Co-doped ZnO NWs/NFs; (d) Acetone sensing performance for undoped, 1% Co-doped and 3% Co-doped ZnO NWs.

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INVESTIGATING THE LONG-TERM STABILITY OF SI AND SIC NANOWIRES UNDER PHYSIOLOGICAL CONDITIONS

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Keywords: Silicon Carbide, Silicon, Nanowire, Chemical stability, Sensor, NWFET, DNA

The fast and direct detection of small quantities of biological and chemical species is of key importance for numerous biomedical applications. Extensive research has been conducted on nanoelectronic devices that can perform such detection with high sensitivity using silicon nanowires and nanostructures. However, it was recently demonstrated that Si material suffers a lack of long-term stability in physiological environments at nanometer scale [1,2], and is hence not suited for in situ sensing of biological molecules. Here, from systematic HRTEM investigations, we present a comparative study of bare Si and SiC nanowires undergoing degradation under physiological conditions. The results presented are an important steps toward the realization of core-shell Si-SiC NWFETs for the detection of biomolecules in liquid media. We show that SiC NWs exhibit higher chemical stability than Si NWs under physiological conditions. Indeed, after 28 days, Si NWs have suffered a mass loss of 76% on average whereas SiC NWs were consumed by only 25% (Figure 1).



Figure 1. Monitoring of normalized mass of Si and SiC nanowires derivated from the measurement of individual diameters using HR-TEM, and standard error

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MERCREDI 15 NOVEMBRE

SESSION 8 : CROISSANCE 2

OBSERVATION IN-SITU DANS UN MICROSCOPE ELECTRONIQUE A TRANSMISSION DE LA CROISSANCE PAR JETS MOLECULAIRES DE NANOSTRUCTURES III-V

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Mots clés: microscopie électronique en transmission, croissance in-situ, Epitaxie par Jets Moléculaires

Le projet Nanomax permet d'observer la croissance de nanostructures jusqu'à l'échelle atomique dans un microscope électronique en transmission. Il est prévu d'étudier en temps réel la croissance de nombreuses nanostructures : nanofils de semiconducteur III-V ou de silicium/germanium mais également des nanotubes de carbone. La croissance est réalisée in-situ dans un microscope électronique en transmission Titan ETEM équipé d'un correcteur d'aberration géométrique sur l'image. Le microscope ETEM a été modifié afin de pouvoir disposer d'une qualité de vide suffisante pour permettre la croissance en épitaxie par jets moléculaires (EJM). Par ailleurs, la vitesse de pompage dans la chambre objet du microscope est assez importante pour réaliser des croissances par CVD et UHV-CVD afin de permettre la croissance de nanotubes de carbone ou de nanofils/nanostructures d'éléments IV. Nous avons mis au point pour ce microscope des microcellules à effusion pour éléments III et éléments V permettant de réaliser la croissance in-situ de nanofils de semiconducteur III-V (en particulier GaAs, InAs et GaSb). Les sources sont très collimatées afin d'éviter la contamination de la chambre du microscope, elles émettent sans perturber le fonctionnement du microscope. Nous montrons qu'il est possible d'étudier la croissance par EJM de nanofils de semiconducteur in-situ dans le microscope à une résolution atomique.

Les premières études nous ont permis de mettre en évidence l'influence de l'angle de contact du catalyseur sur la phase cristalline des nanofils. L'observation de la croissance à l'échelle atomique en temps réel permet d'accéder directement à la statistique de nucléation des marches et à leur vitesse de déplacement en fonction des conditions de croissance. En modifiant le rapport des flux III/V, on observe en direct le changement de volume du catalyseur gallium et la modification de l'angle de mouillage de la goutte, ce qui entraîne un changement de la phase cristalline du nanofils (hexagonale vs cubique). Le projet Nanomax est développé dans le cadre de l'Equipex « TEMPOS ».

Homogeneous and high-quality InGaN nanowires with different indium compositions

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Mots clés : Nanowires, Indium Gallium Nitride, Hydride Vapor Phase Epitaxy.

The growth of high-quality InGaN nanowires across the entire composition range has recently been achieved by Hydride Vapor Phase Epitaxy (HVPE). The remaining technological challenges concern the achievement of high efficient optical devices. In this work, we report on the HVPE synthesis and properties of InGaN nanowires on silicon and GaN template using GaCl and InCl₃ as III-element precursors. The influence of temperature and input partial pressures of different gaseous species on InGaN nanowires morphology and indium incorporation was investigated. The structural and optical properties of the InGaN nanowires are addressed by Transmission Electron Microscopy (TEM) and Photoluminescence (PL) investigations. Morphological studies using Scanning Electron Microscopy (SEM) confirm an homogenous growth in the entire substrate. The indium incorporation, length and diameter of InGaN nanowires grown on GaN template increase by decreasing III_{Ga}/III_{In} ratios. On silicon, Local Energy Dispersive X-rays spectroscopy (EDX) measurements reveal an homogenous In incorporation along the entire length of a single wire. Particularly, indium-rich InGaN nanowires exhibiting a pure wurtzite phase along their entire length are obtained. Only some defects in InGaN nanowires has been observed by High Resolution Transmission Electron Microscopy (HRTEM) whatever the indium composition. These results constitute the state-of-the-art of the crystalline phase for the entire indium composition. This appears very promising for the integration of high-indium nanowires in optical components.



Bright-fiel HRTEM images and SAED patterns showing the crystaline quality of InGaN nanowires synthesized at different temperatures. Defect-free cristal obtained for x >70 %

Defect-free Bi_{1-x}Sb_x Nanowires on Si by MBE

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Keywords: BiSb, Nanowires, Thermoelectrics, Quantum Computer, Topological

1D nano structures of Bismuth Antimonide $(Bi_{1-x}Sb_x)$ alloys, especially nanowires, are promising for quantum computing, thermoelectrics and spintronics.¹ Varying the Sb composition (x), $Bi_{1-x}Sb_x$ is suggested to behave as: a semi-metal (x<0.07), an indirect bandgap semiconductor (0.07<x<0.09), a direct bandgap semiconductor (0.09<x<0.15), an indirect bandgap semiconductor (0.15<x<0.22) and again semi-metal (x>0.22).¹ Thus, a precise control of this parameter hold promises for future quantum devices. For instance, if x=0.03, 3D Dirac cones should be observed in the structure and could be used to host Majorana zero mode when coupled with a superconducting contact.² If 0.08<x<0.24 the material behaves as a 3D-topological insulator, and for x>0.23, the high electron mobility and strong spin-orbit interactions make it an interesting candidate for spintronics.^{1,3}

Hence, excellent quality of 1D nanostructure with control of the Sb composition is necessary in order to understand and engineer the material. This study is the initial step on addressing these existing hurdles and on exploring opportunities. Epitaxial $Bi_{1-x}Sb_x$ nanowires with controlled Sb concentrations are integrated on Si(001) and Si(111) substrates for the first time.

The process starts with the removal of native oxide from unpatterned Si(001) and Si(111) wafers with the help of hydrofluoric acid (HF 5%). The degassing of substrates at 200°C follows next. Then, self-catalyzed growth of $Bi_{1-x}Sb_x$ nanowires (varying x) occurs on these substrates in a solid source molecular beam epitaxy (MBE) system. Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) characterizations are carried out for morphology, composition and crystallographic studies. Electrical characterizations will be performed to extract intrinsic properties (electron mobilities, resistivity etc.).

SEM characterizations confirm high density of $Bi_{1-x}Sb_x$ nanowires with diameters around 30 nm and lengths up to 15 μ m. As expected, the composition has a significant impact on these nanostructures including density, morphology, and crystallography. Finally, the electrical characterizations will be presented.

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ZnO NANOWIRE FACETS TRANSFORMATION INDUCED BY Ga DOPING

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Mots clés : ZnO nanowires, MOCVD growth, doping, surfactant, photoluminescence

ZnO nanowires typically grow along (0001) C-axis and exhibit (10-10) M-facets with hexagonal morphology. This ability of non-centrosymmetric wurzite semiconductors such as GaN or ZnO to show spontaneous 1D growth without any catalyst process is due to several phenomena, among them, the anisotropy of polar and non-polar surface energies. However, previous works show that growth direction, morphology and facets orientation can be tuned by adding a dopant in the source material (in CVD processes), or metal ions in the solution (e.g. hydrothermal growth).

In this work, the effect of gallium doping on the MOCVD growth mechanisms of ZnO nanowires is presented. Ga-doped and undoped ZnO nanowires were realized following spontaneous (catalyst-free) or gold-assisted (so-called vapor-liquid-solid VLS) processes. ZnO/ZnO:Ga core-shell structures and reference layers were also synthesized. By using scanning electron and transmission electron microscopies (SEM, TEM), a strong influence is observed on all kinds of ZnO structures, as illustrated in the figure below. Gallium atoms on the surface modify the growth along C-axis with a drastic change in the lateral facets orientation. TEM analysis shows an evolution of the roughness of the M-plane nanowire facets with, progressively, the appearance of (201), (101) and (001) surfaces. All these planes traduce a rotation of the initial M-plane surface towards the <001> direction :

(100) M plane => (201) (angle 15°) => (101) (28°) => (001) C (90°).

In addition, photoluminescence spectra clearly show an increase of the donor bound exciton emission at 3.359 eV, assigned to Ga incorporated impurities. The mechanisms at the origin of such transformation are discussed and compared with the "usual" surfactant effects.



Figure 1: SEM ant TEM images of Ga-doped ZnO nanostructures

SESSION 9 : DISPOSITIFS PIEZOELECTRIQUES

PIEZO-ELECTRIC III-N NANOWIRES FOR ENERGY HARVESTING

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Mots clés: III-N Nanowires, Piezoelectric properties, Piezogenerators, Energy harvesting

In order to address the worldwide challenge dealing with the development of autonomous microdevices for sensing, monitoring and nomad electronics, new competitive integratable electrical energy sources are required. These sources must generate sufficient power to supply the micro-devices without increasing their size and weight. Recently, III-Nitride nanowires (NWs) have emerged as excellent candidates to fabricate novel ultra-compact and efficient piezoelectric generators. Thanks to their superior mechanical properties, higher sensitivity to applied force and higher piezoelectric response over conventional 2D films and bulk materials, the NWs have the potential to fundamentally improve the generator performances.

Based on a systematic multi-scale analysis, going from single NW properties to macroscopic device fabrication and characterization, we establish for GaN NWs the relationship between the material properties and the piezo-generation [1-3]. The piezo-conversion of individual MBE-grown p-doped GaN NWs in a dense array is assessed by atomic force microscopy equipped with Resiscope module yielding an average output voltage of 228 ± 120 mV and a maximum value of 350 mV generated per NW. In the case of p-doped GaN NWs, the piezo-generation is achieved when a positive piezo-potential is created inside the nanostructures, i.e. when the NWs are subjected to a compressive deformation.

Thank to this fine understanding of the piezo-conversion mechanisms, we propose an efficient piezo-generator design operating under compressive strain. The devices integrate NW arrays of several square millimeters in size and deliver a maximum power density of 12.7 mW/cm³ [4]. This value settles the new state of the art for piezo-generators based on GaN NWs and more generally on nitride NWs. These results offer promising prospects for the use of GaN NWs for high-efficiency ultra-compact energy harvesters, since the generated power density is already interesting for real world applications such as remote wireless transceivers.

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OPTIMISATION GEOMETRIQUE DES NANOGENERATEURS A BASE DE NANOFILS D'OXYDE DE ZINC

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Mots clés : Nanofils piézoélectriques, Nano-générateurs, Méthode des Eléments Finis

La récupération d'énergie mécanique utilisant des nano-générateurs piézoélectriques (PENGs) à base de nanofils d'oxyde de zinc (ZnO) a suscité depuis 2006 un intérêt croissant pour le développement de microsystèmes autonomes. Parmi diverses méthodes de croissance, la synthèse hydrothermale est la plus souvent utilisée, car elle est simple et peu coûteuse.¹ L'optimisation de la qualité du matériau piézoélectrique ainsi que de sa morphologie² et de sa densité surfacique³, est cruciale pour l'amélioration des performances des dispositifs PENGs. Ce travail s'intéresse à l'effet de la forme du nanofil (NW) sur le potentiel généré sous l'effet d'une compression dans l'axe du NW. Dans ce but, nous avons effectué des simulations par éléments finis, à l'aide du logiciel COMSOL Multiphysics. Ainsi, la répartition du potentiel piézoélectrique généré dans les NWs de ZnO a été calculée en statique, lorsqu'une pression de 100 kPa est appliquée au sommet du NW, dans la direction de sa hauteur. En terme de potentiel piézoélectrique, les nanofils cylindriques fournissent un potentiel bien supérieur (25 mV) à celui généré par leurs homologues de forme conique (0,6 mV) (Figure 1). Pour valider expérimentalement ces résultats de simulation, nous avons cherché à adapter les conditions de croissance pour obtenir précisément la forme prédéfinie du NW. Pour cela, nous avons exploité l'effet de la température de croissance sur la morphologie des NWs, en effectuant des synthèses à différentes températures dans la gamme 70-100 °C (par pas de 10°C), dans les conditions décrites dans¹. Comme le montre la Figure 2, des NWs à section hexagonale peuvent être obtenus à basse température (~70 °C), tandis qu'ils présentent une morphologie plus effilée ou conique pour une température de croissance proche de 100 °C. Cette différence de morphologie peut s'expliquer par des effets thermodynamiques liés à l'énergie de surface des différentes faces cristallines du ZnO.⁴ La caractérisation fonctionnelle des PENGs obtenus avec les différentes morphologies permettra de confirmer l'effet de la morphologie des NWs sur les performances du dispositif.



sur le diamètre supérieur du NW.



Figure 1: Potentiel piézoélectrique généré Figure 2: Images MEB à différentes températures: (a) 70 °C pour diffèrents ratios du diamètre inférieur et (b) 100 °C. L'image à droite correspond à la distribution du potentiel electrique obtenu par COMSOL.

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ZnO NANOWIRE BASED PIEZOELECTRIC NANOGENERATOR

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Keywords: ZnO nanowires ; Energy harvesting ; Nanogenerator.

Harvesting energy from the environment is a potential approach to build the self-powered microsystems. In this work, we demonstrated a prototype of ZnO nanowire (NW) based piezoelectric nanogenerator (NG) aiming at aeronautical application. When stimulated by vibrational and compressive mechanic motion, energy harvested by NG (by converting the mechanic motion to piezoelectricity) can supply different kinds of self-powered sensors.

Easy and low coast hydrothermal method [1] was used to grow the ZnO NWs on gilded silicon substrate. The NWs owning an aspect ratio ~25 were obtained for a growth time of 4h. Figure 1 shows the NG device fabrication process and the final device photography encapsulated by PDMS. Figure 2 shows the experimental set-up for energy harvesting and the output voltage of our NG (with effective surface of 0.7 cm²) solicited under compressive mode, which increased from 1.7 V to 5.6 V while the applied force raised from 2 N to 6 N with shaking frequency of 9 Hz. Those results are agreeing with our previous numerical simulation on ZnO NW array based NG [2].









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High-Resolution Fingerprint Sensing with Vertical Piezoelectric Nanowire MATrices: main achievements of the PiezoMAT project

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Keywords: ZnO nanowire, pressure sensor, microelectronics

Ended in June 2017, the 44 months FP7 European project PiezoMAT brought together 8 partners from academia, R&D centres along with small and medium enterprises as well as large enterprises from across the EU [1]. The project proposes proof of concept of a new technology of very high-resolution fingerprint sensors based on a matrix of interconnected piezoelectric ZnO nanowires (NWs) able to reach resolution higher than 1000 dpi. Three different sensing architectures have been fabricated with the charge collections at the bottom of single nanowires or with top-bottom contacts. We developed a direct write lithography method by using EBID for contacting vertical ZnO facets with electrically isolated lines with sub-micron size [2]. The second main result is the first experimental demonstration of bottom-bottom contacted 'bending mode' force sensor with a resolution of 5000dpi [3]. We also demonstrated the feasibility of the third architecture [4], with the fabrication of a demonstrator embedding a silicon chip with 250 pixels (1000dpi), and its associated electronics for signal collection and post-processing (fig.1).



Figure 1: tactile mapping by wire stamp and set-up device for force sensitivity demonstration of the chip

Valuable experience and know-how was gained in several areas, such as optimization of seed layers processing, localized growth of well-oriented ZnO nanowires on silicon substrates, mathematical modelling of complex charge generation [5], and development of multi-functional UV-crosslinkable encapsulating polymers [6].

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