SAND2017-0927C

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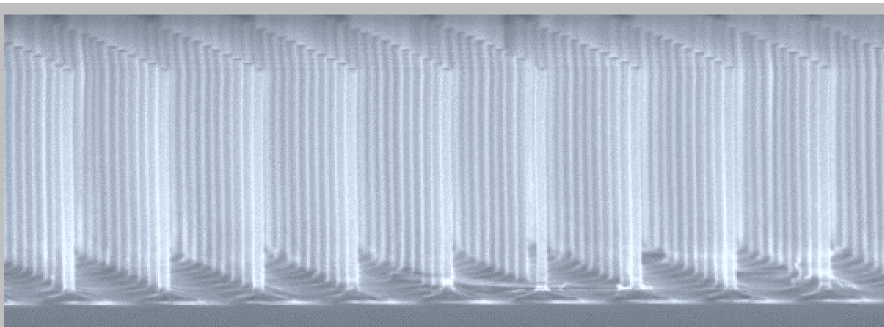

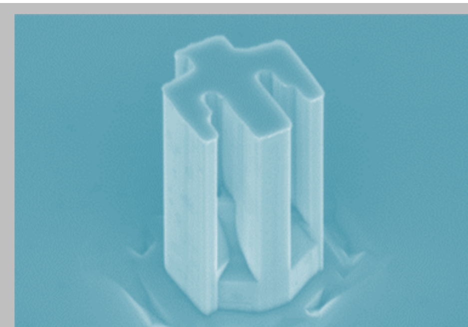

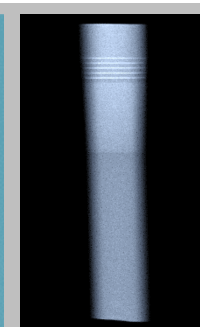

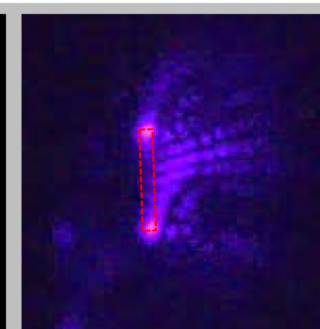

Top-down fabricated III-nitride nanowire photonics

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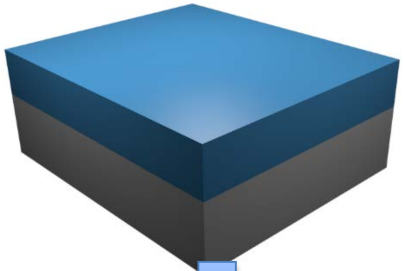
Motivation: Top-down approach for nanostructures

Motivation: Advantages of *top-down approach* for fabrication, for both *material synthesis and integration*

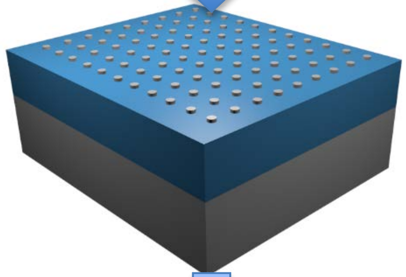
Steps | **Advantages**

Conventional GaN film



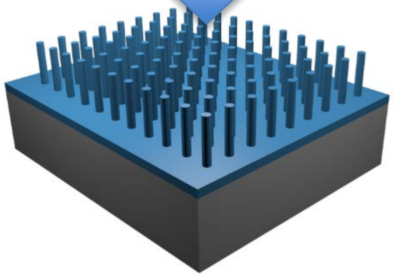
- Standard/optimized growth conditions (not limited by 1D growth window) give control over:
  - Dopant, impurity incorporation
  - Axial heterostructuring
- Can be characterized by standard methods

Lithographic technique (e.g. e-beam, nanoimprint, interferometric)



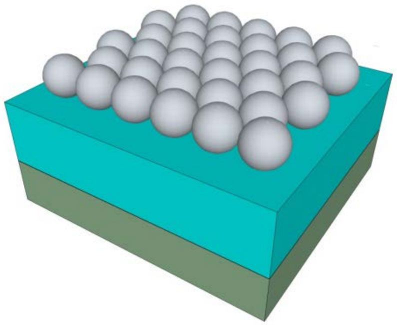
- Ordered and periodic arrays
- Independent control over height, width and pitch
- Cross-sectional shape control

Anisotropic etch



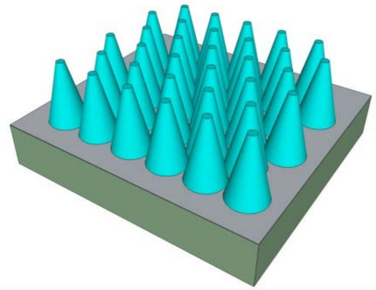
- Potentially less tapered, higher aspect-ratio structures possible for highly anisotropic, crystallographically selective etches

2-Step hybrid "dry + wet" top-down ordered nanowire fabrication process

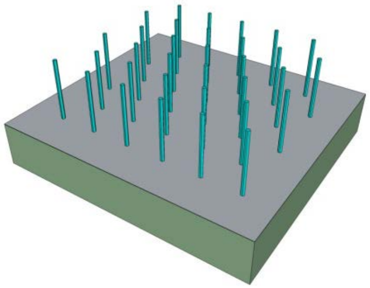


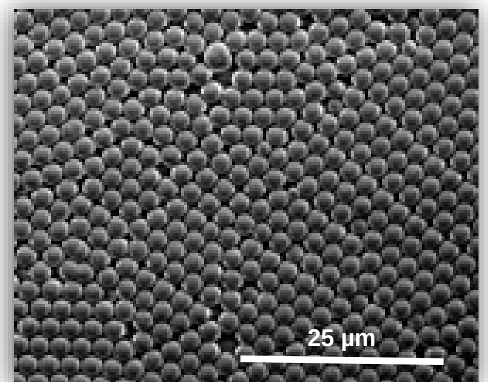
(0001) GaN on sapphire

ICP etch

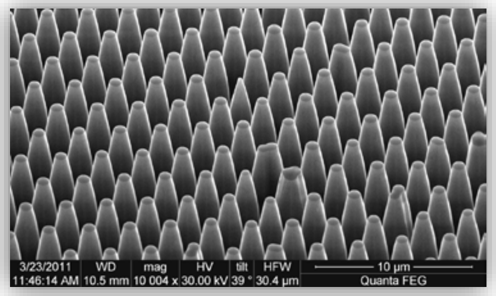


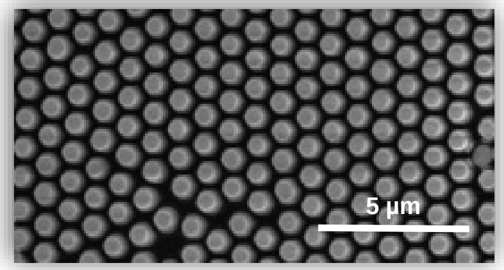
Selective KOH-based wet etch (AZ-400K developer)





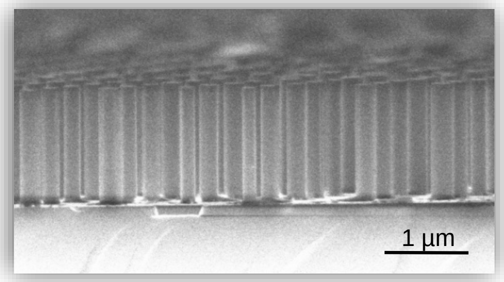
Q. Li, J. J. Figiel, G. T. Wang, Appl. Phys. Lett., **94**, 231105 (2009).

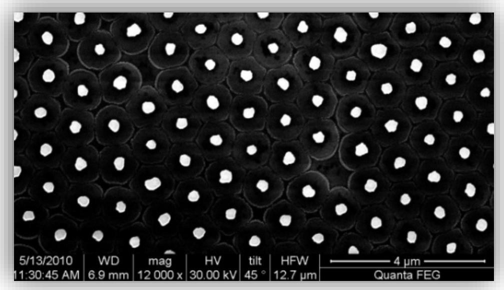




Plasma etch causes sidewall damage

Tapered; no well-defined facets





Wet etch: straight sidewalls, removes sidewall damage

Fabricated top-down GaN nanostructures

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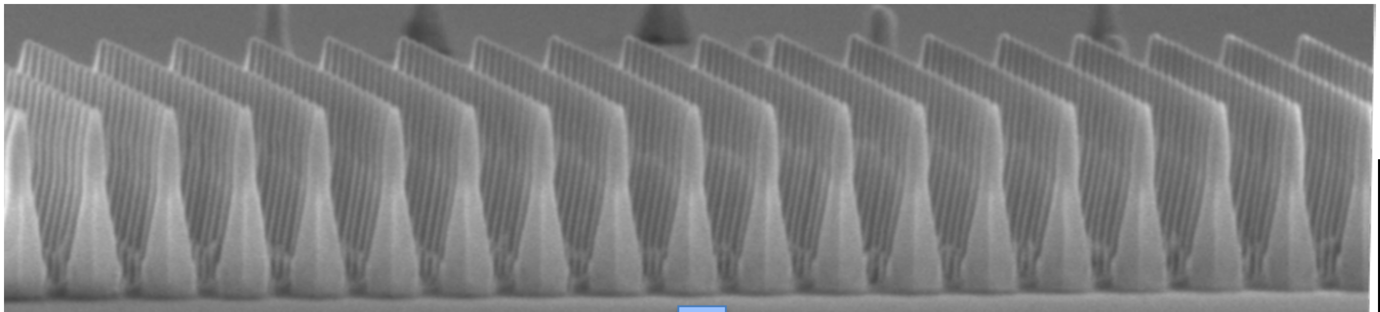
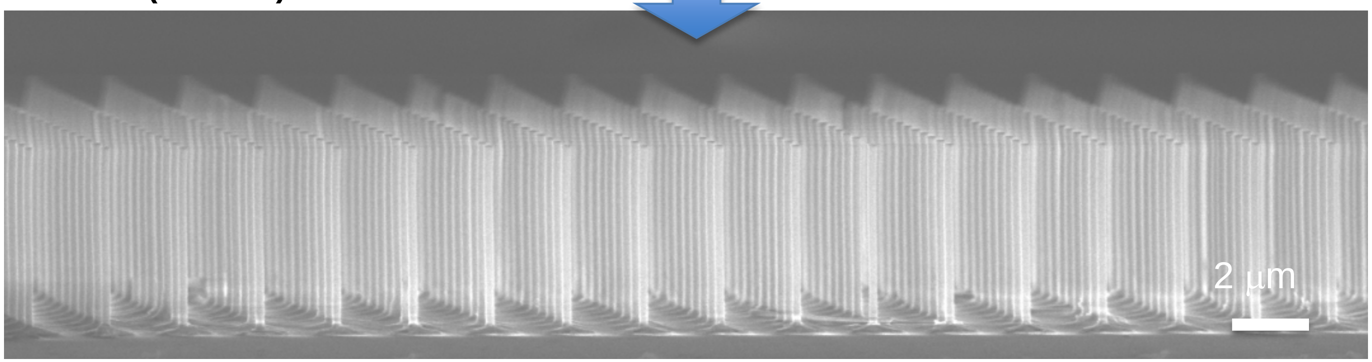
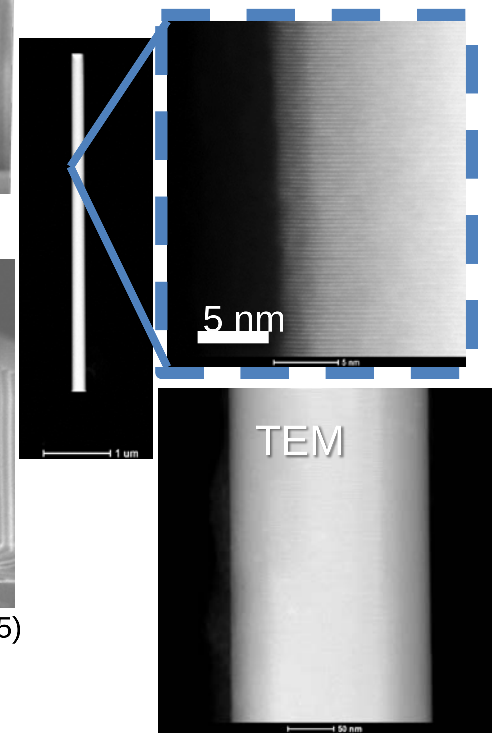
Dry etch (Cl_2 based ICP)



Wet etch (AZ400K)



TEM shows smooth vertical sidewalls



Diameter: 180 nm, Height: 4.5 µm, Pitch: 2 µm (AR: 25)

See: Q. Li et al., Opt. Exp., **20**, 17873 (2012)

Q. Li et al., Opt. Exp., **19**, 25528 (2011) etc.…

What is the wet-etch mechanism from *rough tapered to smooth vertical sidewalls*?

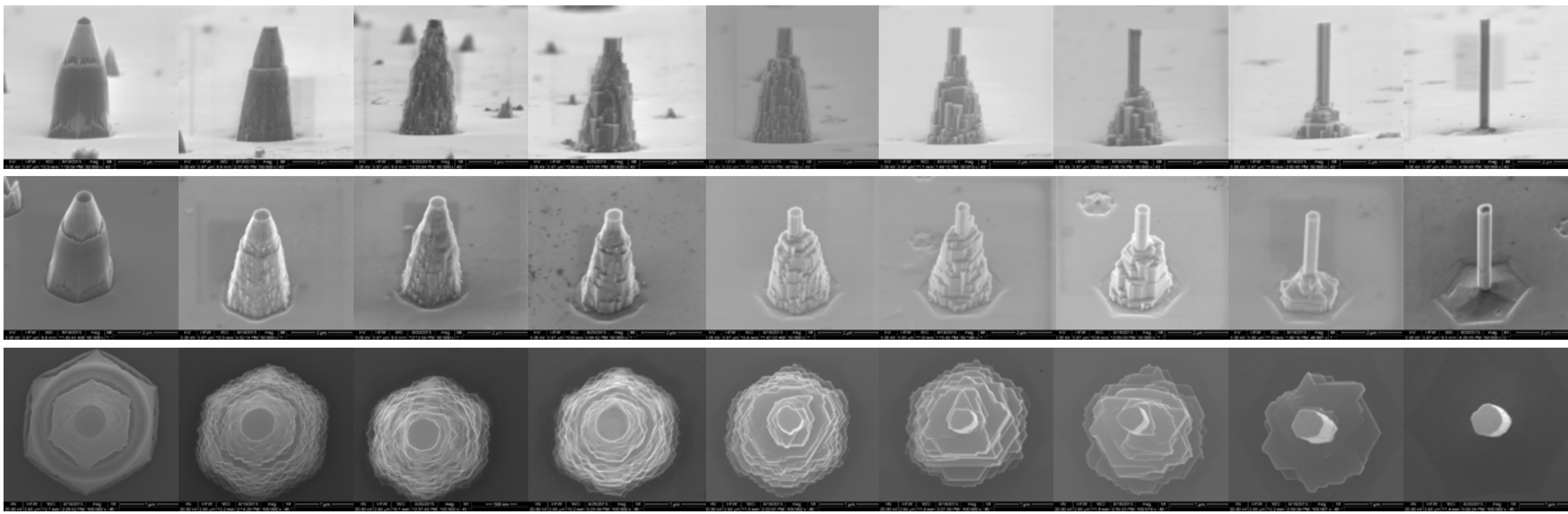
Wet etch evolution for nanowires

As ICP etched | 1 min | 2 min | 5 min | 10 min | 15 min | 20 min | 30 min | 52 min

Side view

45°

Top view



AZ400K, 65^{o}C

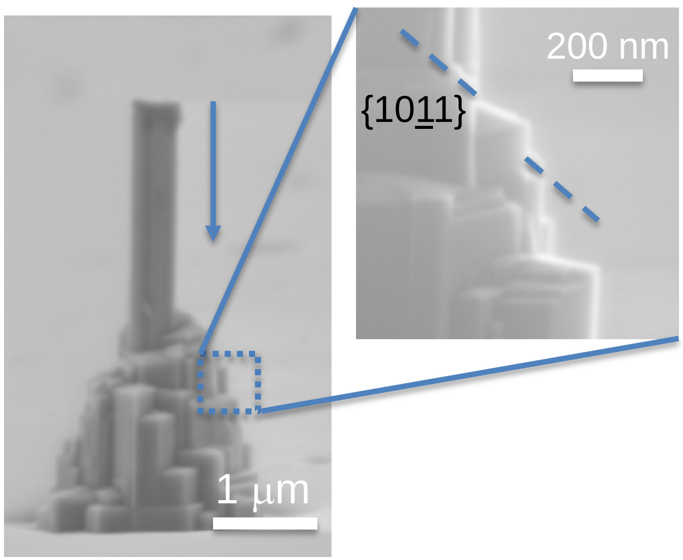
Wet etch proceeds "vertically" rather than horizontally

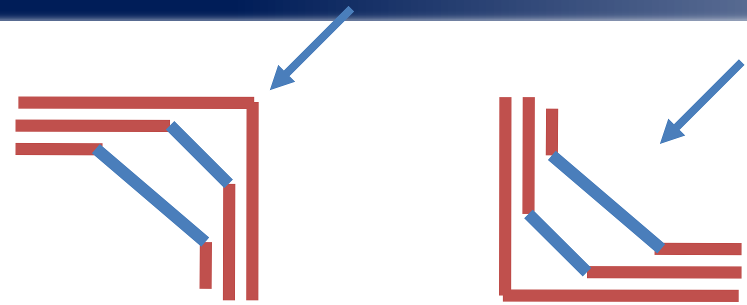
Mechanism/Basis for evolution of facets during etching

What is the mechanism from *tapered to smooth vertical sidewalls* through faceting?

Slow etching: c-plane, m-plane

Fast etching: Semipolar (10$\bar{1}$1)





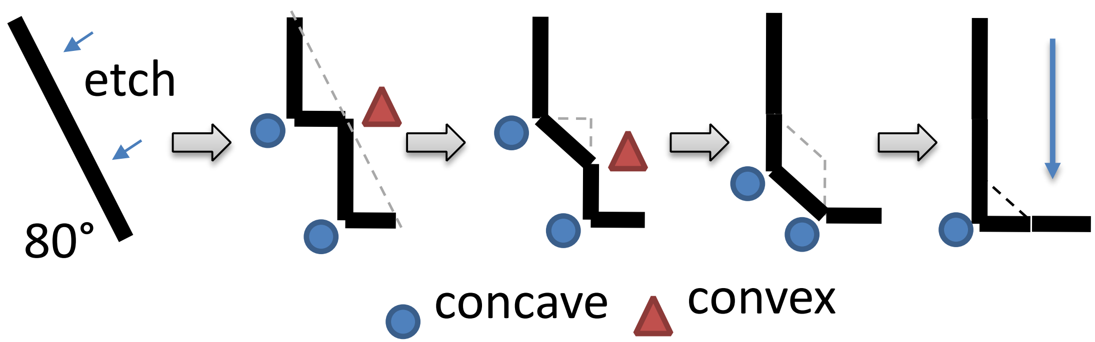
Convex

Fast etch facet persists

Concave

Slow etch facets persists

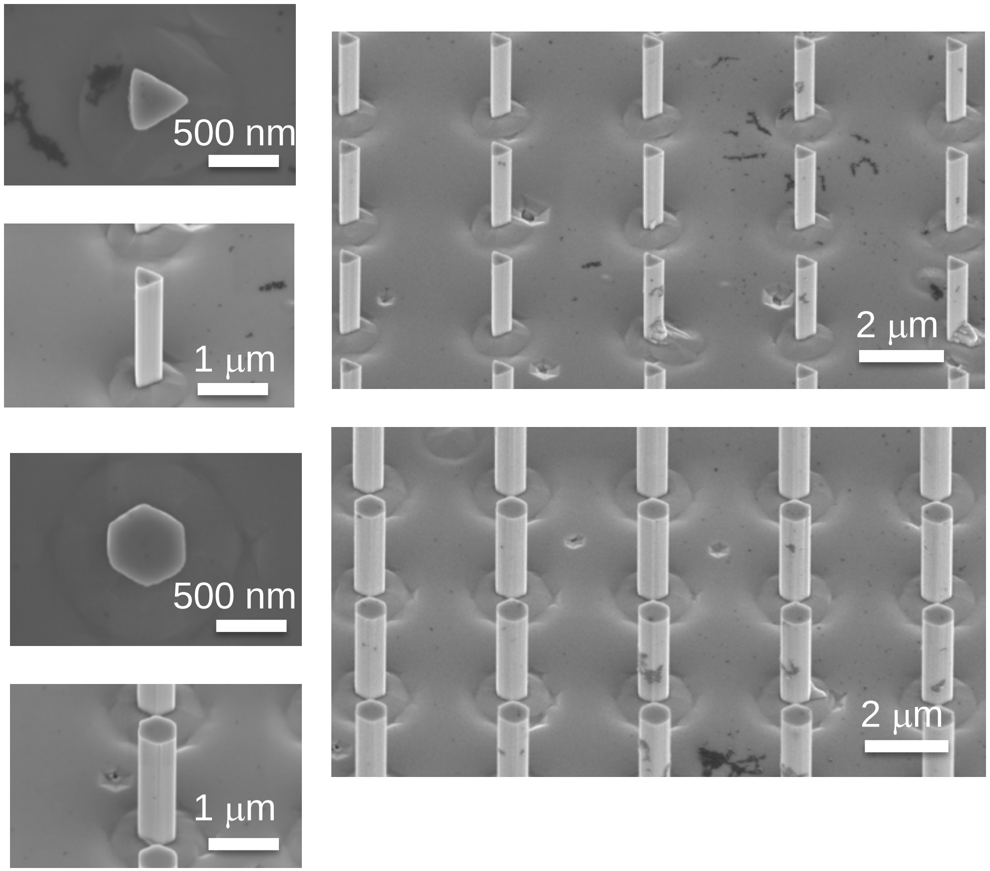
The facet etch sequence:

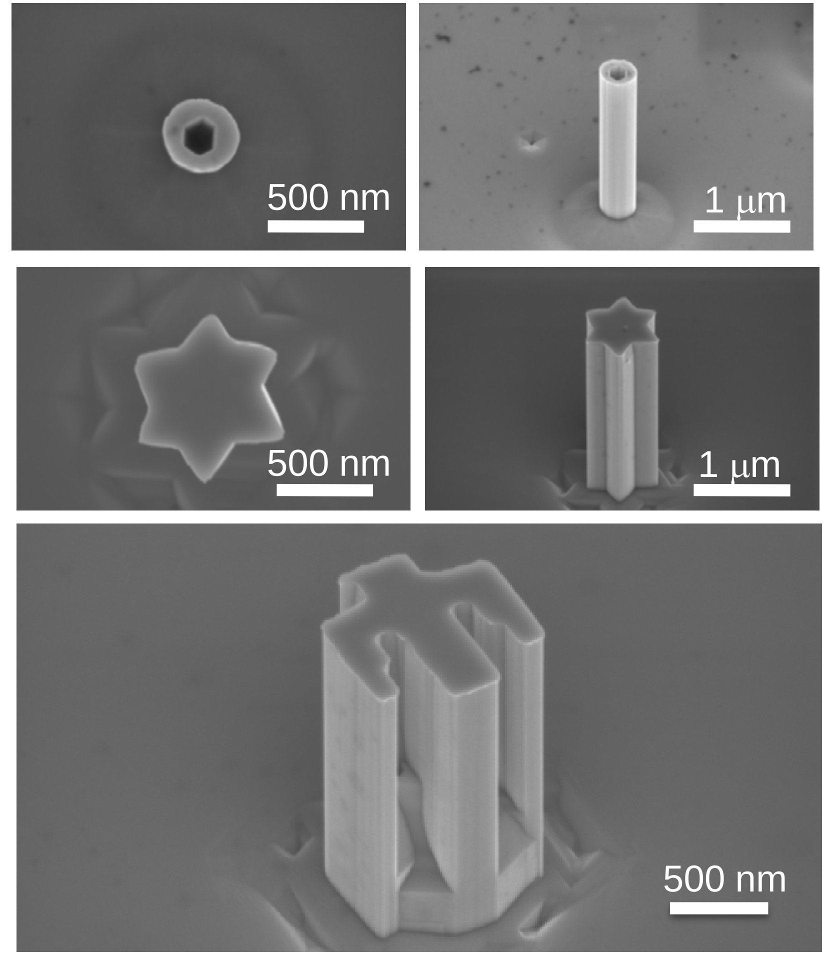


The appearance (disappearance) of fast (slow) etching facets in concave (convex) geometries is the basis of the etch mechanism

Controlled and complex cross-sections

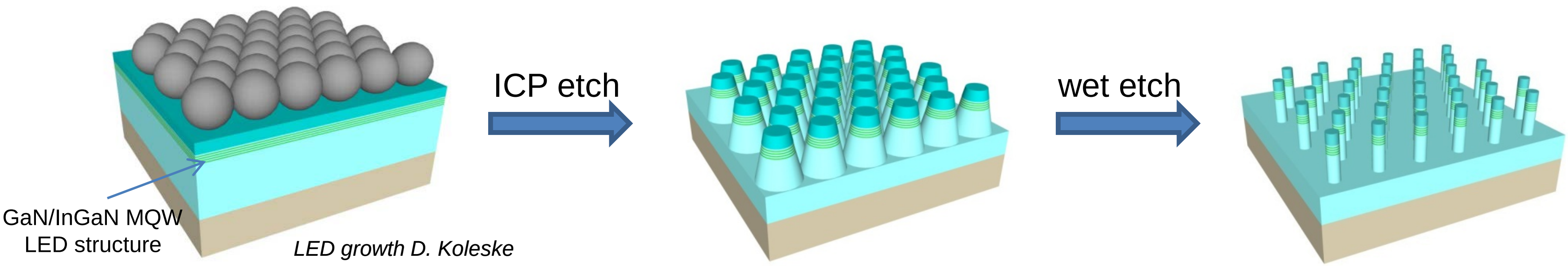
Can we expand the use to more *complex geometries* ?

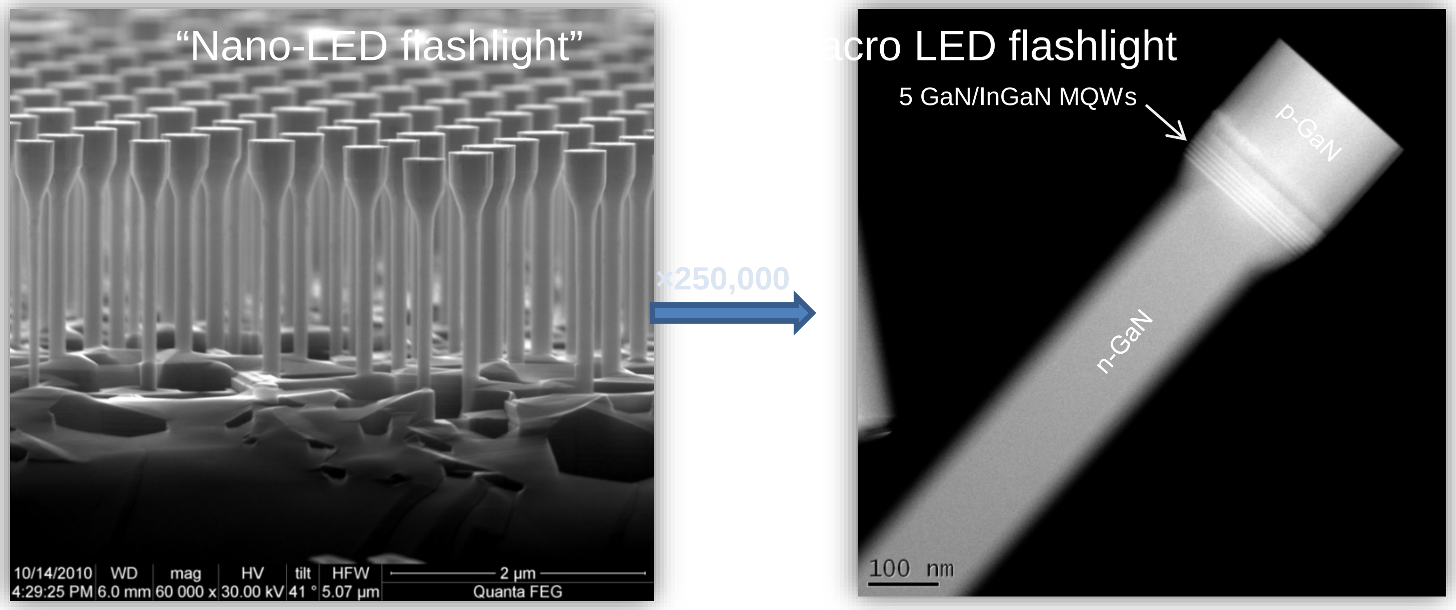




Understanding of the etch evolution enables design of a wide range of smooth faceted vertical nanostructures

Etching of Axial Heterostructures - GaN/InGaN nanowire LEDs



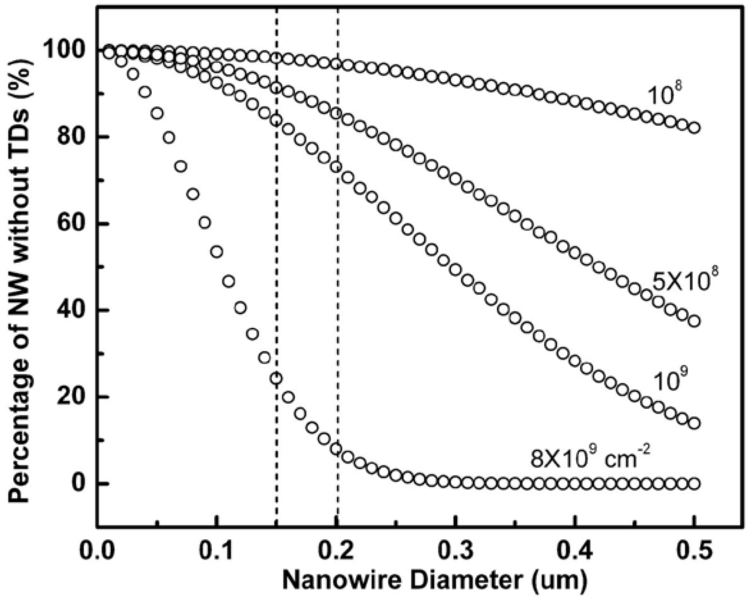


Q. Li et al., *Optics Express* **19**, 25528 (2011)

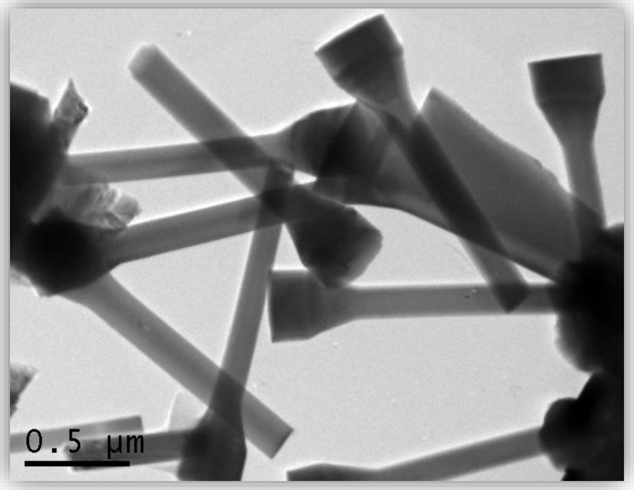
Wet etch affected by doping (composition)

Top-down nanowires - threading dislocations

Monte Carlo simulation



Bright-field TEM

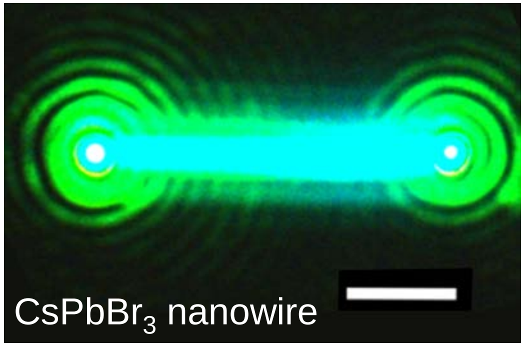


Nanowires etched from ~5e8 cm^{-2} planar LED

- Etched nanowires inherit the dislocation density of the parent film
- However, as the diameter approaches zero, the *fraction* of nanowires with one or more dislocations also approaches zero! [# TDs per rod ~ (TDD)×($A_{cross\text{-}section}$)]
- ~94% of nanowires ~150 nm in diameter from TDD~5e8 cm^{-2} film dislocation free!
- *Thus, nanowire LEDs can function as arrays of largely dislocation-free individual lighting elements*

Nanowire Lasers

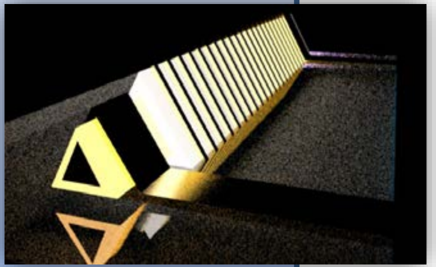
- Nanowire forms a freestanding, low loss optical cavity
- Optically pumped nanowire lasers reported:
  - ZnO, CdS, GaAs, GaSb, GaP, GaN, etc.
- Most commonly Fabry-Perot type lasing from end facets
- Promising nanophotonic elements - compact and low power due to small mode volume



S. W. Eaton, *PNAS* **113**, 1193 (2016)

Need to understand & control optical properties

- **Mode control** – single vs. multi-mode behavior
- **Polarization control** – linear vs. elliptical
- **Wavelength tuning**
- **Beam shaping**
- **Beam steering**



1) S. Arafin, X. Liu, Z. Mi, "Review of recent progress of III-nitride nanowire lasers," *J. Nanophotonics* **7,** 074599-1 (2013)
2) C. Couteau et al., "Nanowire Lasers," *Nanophotonics* **4**, 90 (2015)

Typical GaN Nanowire Lasing: Multimode Operation

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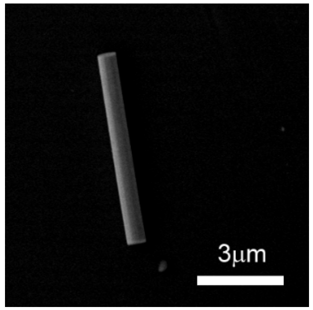
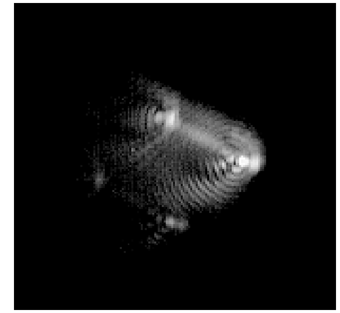
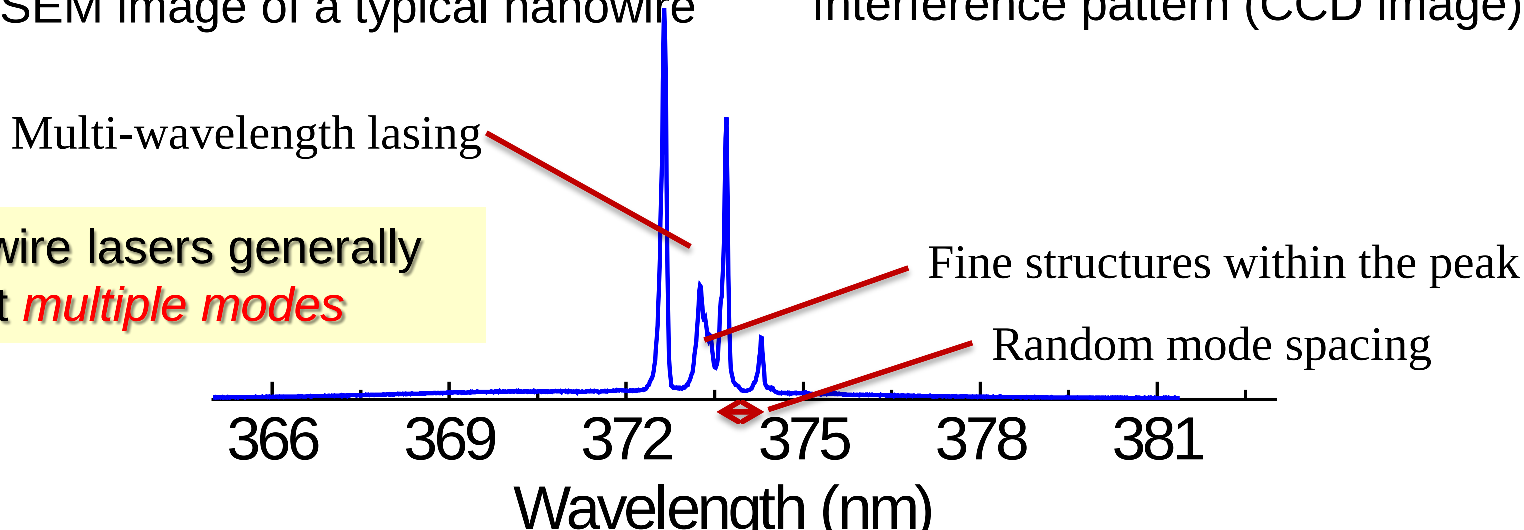


SEM image of a typical nanowire



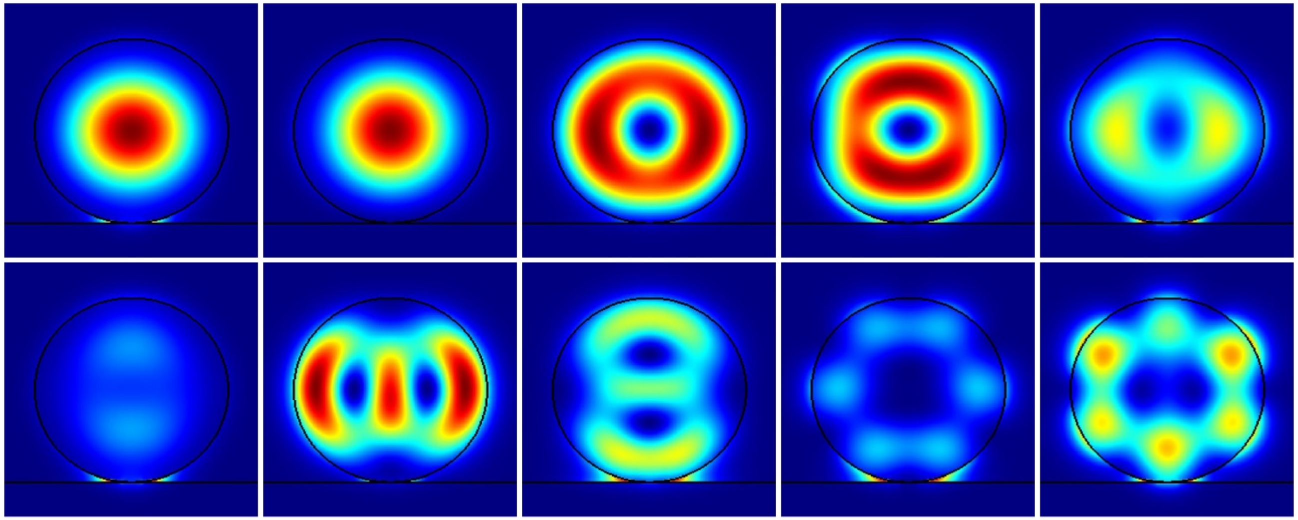
Interference pattern (CCD image)

Nanowire lasers generally exhibit *multiple modes*



- *Single mode* behavior desired for highest resolution and beam quality; may also reduce lasing threshold due to reduced mode competition

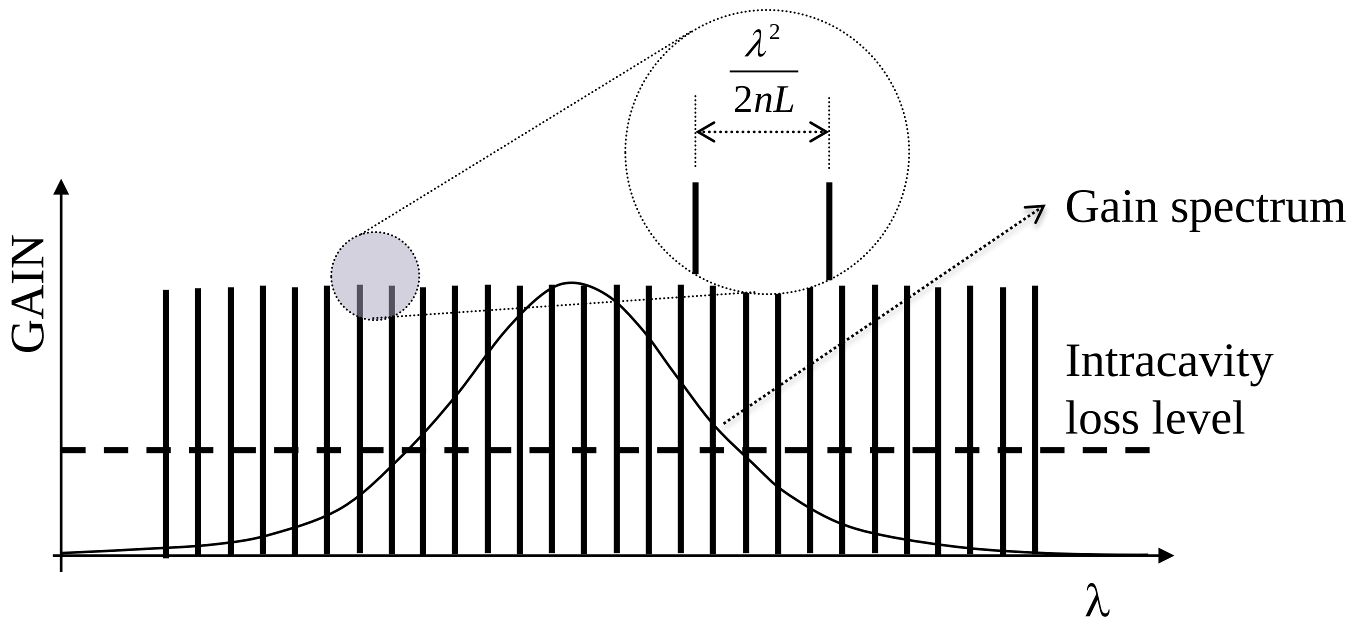
Why Multimode Lasing Occurs



Calculated transverse modes supported by a 300 nm diameter GaN nanowire.

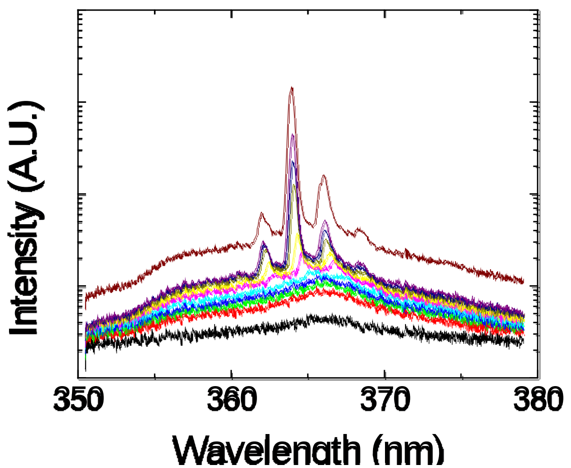
- ✓ Multiple transverse mode waveguide;
- ✓ Single-mode cutoff diameter ~120 nm;
- ✓ Lasing in multiple transverse mode operation
- ✓ Polarization is mode-dependent

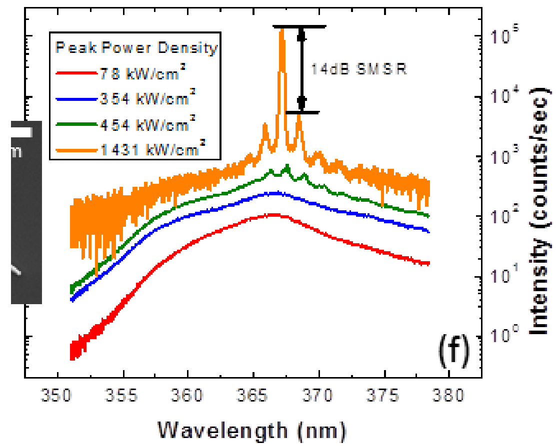
Why Multimode Lasing Occurs

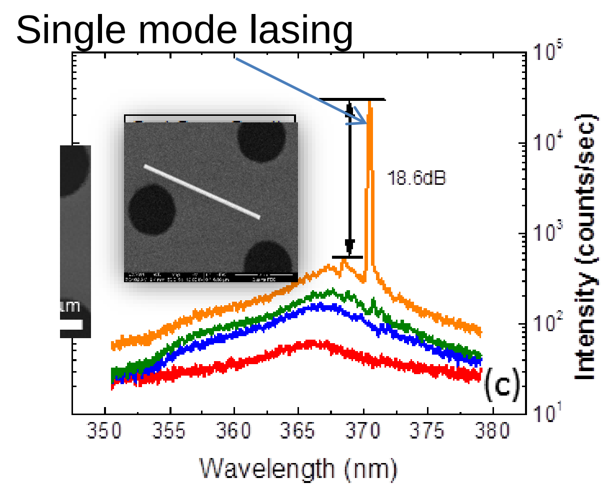


- ✓ Gain bandwidth ~7 nm;
- ✓ Longitudinal mode spacing ~1 nm;
- ✓ Several modes located in the gain spectrum;
- ✓ Multiple longitudinal mode oscillation

Method 1: Single-mode NW laser via geometry control







~500 nm x **4.7µm (short)** | **145 nm** x 7.2 µm **(skinny)** | **~130nm x 4.7µm (short & skinny)**

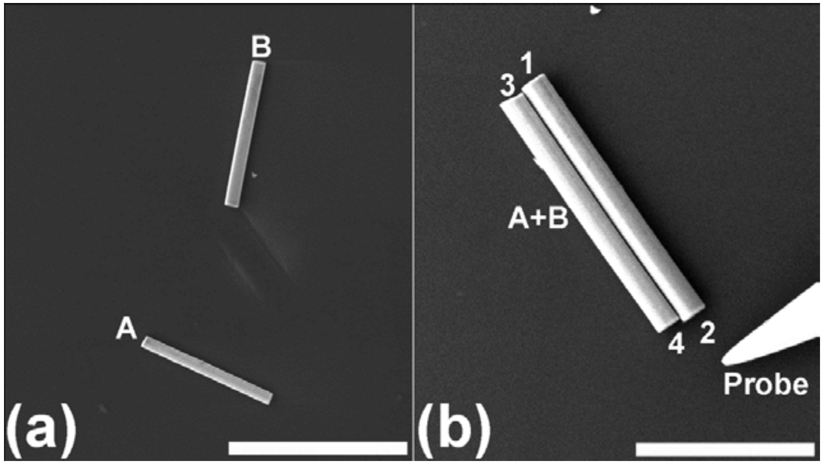
NW diameter x length

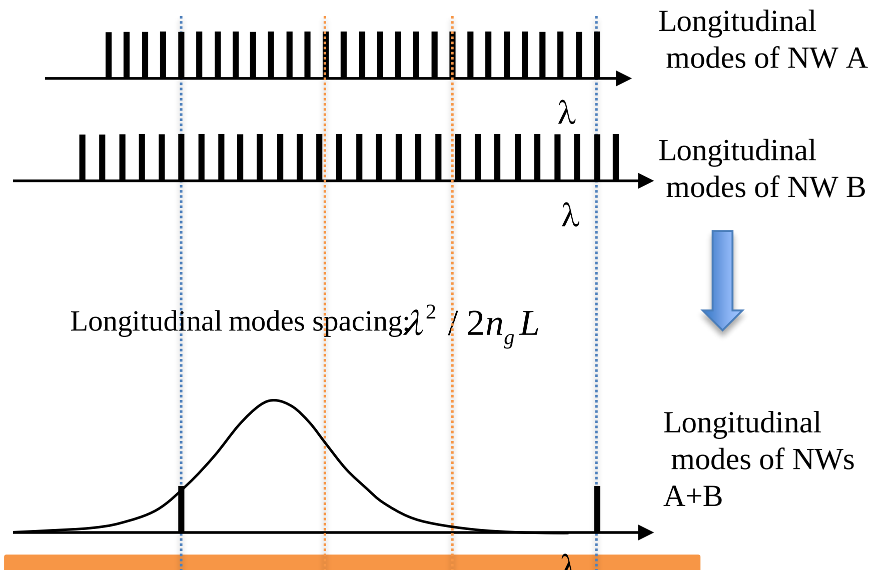
Single-mode: Narrow-Linewidth (<0.1 nm), 18.6 dB Side Mode Suppression Ratio

- Reduction of diameter (<~130 nm): reduction of transverse modes
- Reduction of length: (<~6 µm length): reduction of longitudinal modes
- **Reduced dimensionality leads to single mode lasing**

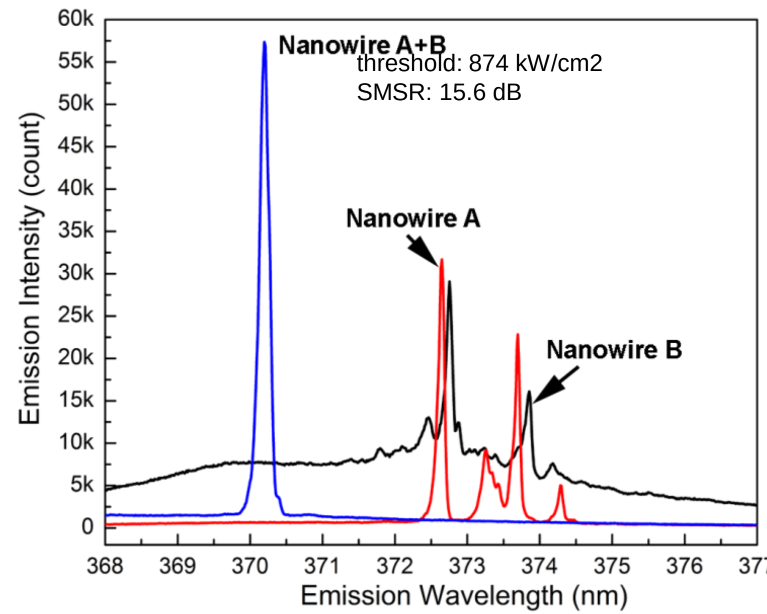
Q. Li et al., "Single-Mode GaN Nanowire Lasers, "*Opt. Exp.* ***20*** *17873 (2012)*

Method 2: Single-mode lasing via coupled nanowire cavities





Longitudinal mode spacing greatly enhanced!

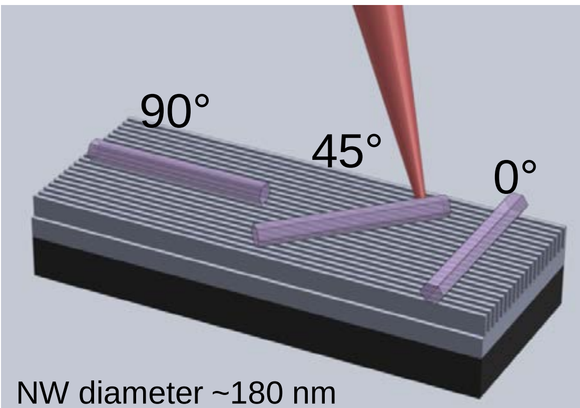


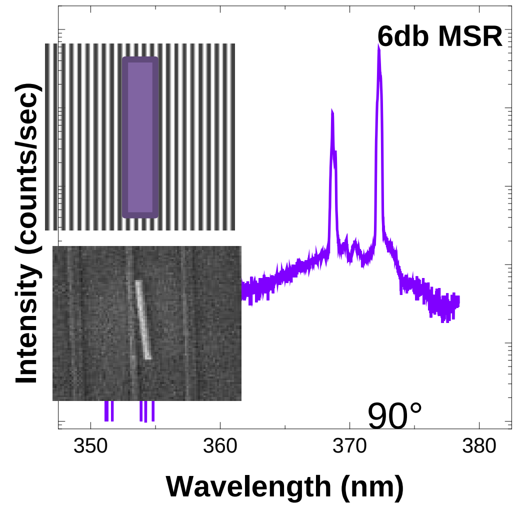
- Individual large NWs shows multiple modes.
- Coupled nanowires show single mode!
- Vernier effect – only resonant modes survive

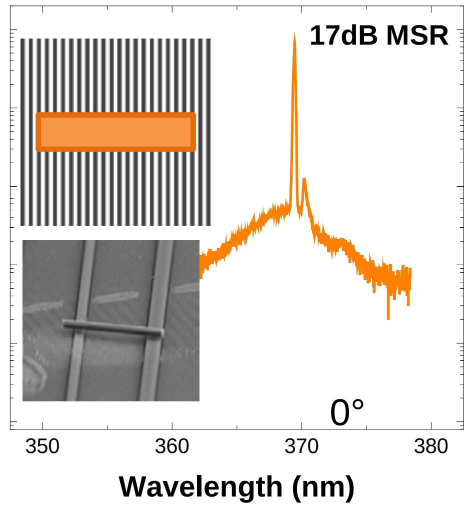
H. Xu et al., "Single-mode lasing of GaN nanowire-pairs," *Appl. Phys. Lett.* **101** 113106 (2012)

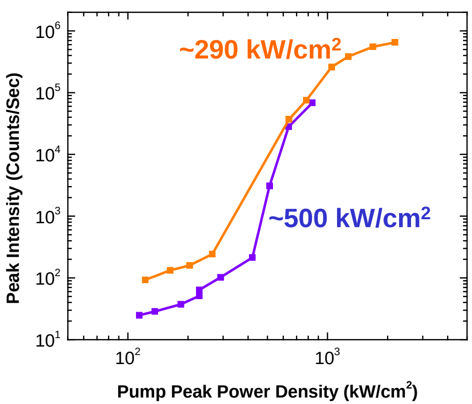
Method 3: Distributed Feedback (DFB) Nanowire Laser

- Standard method for mode control in semiconductor lasers
- Here, single nanowires are coupled to grating substrate to achieve DFB
- Stop-band position and width tuning via nanomanipulation of the angular alignment







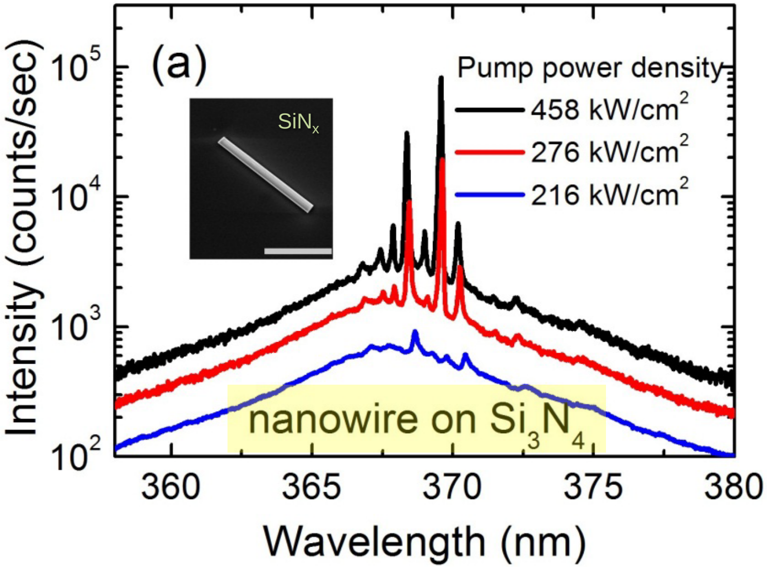


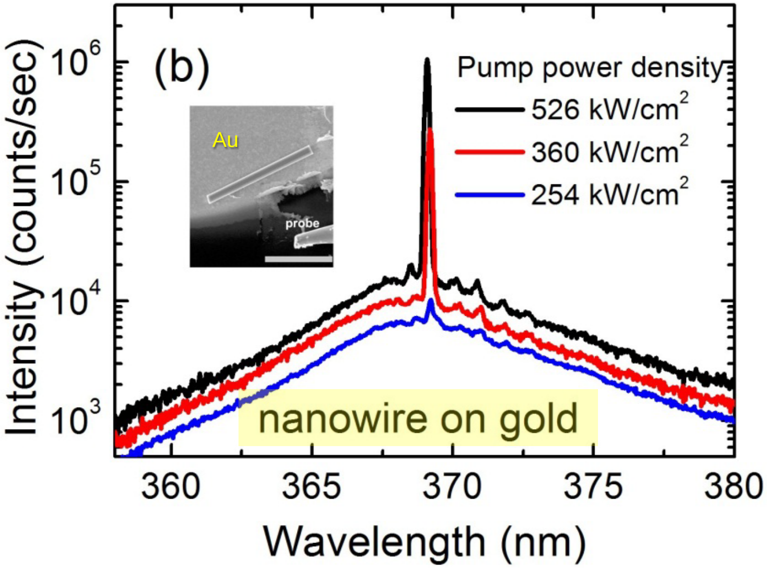
- At the designed alignment <u>single-mode lasing was achieved</u> with a 17dB mode suppression ratio.
- Also observed reduction in the lasing threshold

J.B. Wright, S. Campione, S. Liu, J.A. Martinez, H Xu, T.S. Luk, Q. Li, G.T. Wang, B.S. Swartzentruber, L.F. Lester, and I. Brener, *APL* **104** (4), 041107 (2014).

Method 4: Metal substrate induced single-mode nanowire lasing

diameter~350 nm, length ~5.3 µm

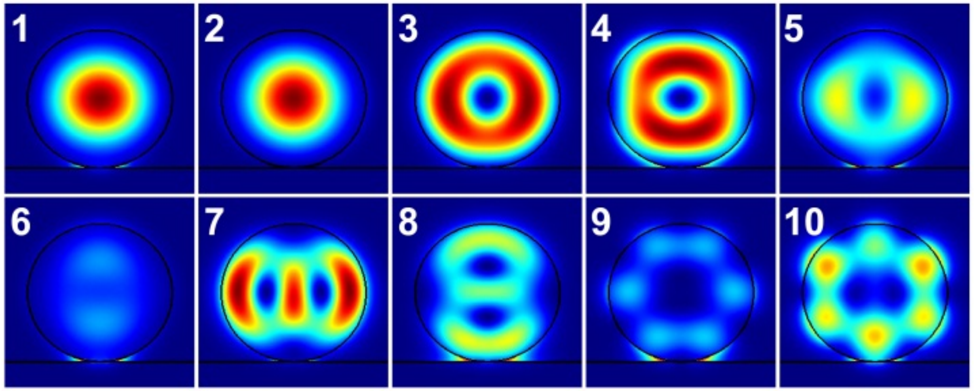




- NWs on Si_3N_4 show multi-mode lasing
- **Same NWs moved onto gold-coated spot show single-mode lasing**
- Increase in lasing threshold of only ~13%

H. Xu et al., "Gold substrate-induced single-mode lasing of GaN nanowires, "*Appl. Phys. Lett.* **101** 221114 (2012)

Gold-substrate induced single-mode lasing



H. Xu et al., *Appl. Phys. Lett.* **101** 221114 (2012)

Transverse modes supported by a 300nm diameter nanowire on gold substrate.

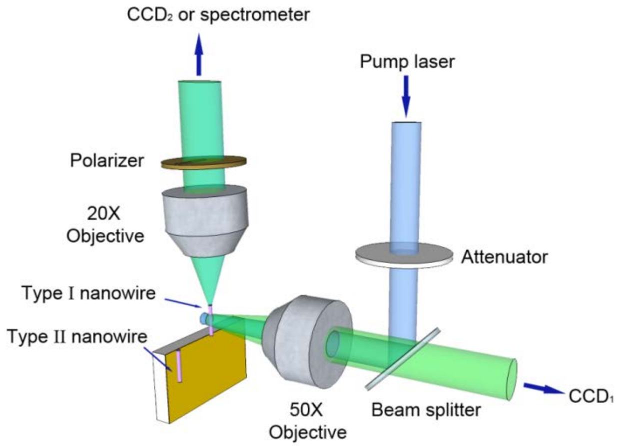
Only surviving mode

	Mode 1	Mode 2	Mode 3	Mode 4	Mode 5
Loss (dB/cm)	8151	1730	4806	7109	16041
	Mode 6	Mode 7	Mode 8	Mode 9	Mode 10
Loss (dB/cm)	34858	7551	22205	29175	28706

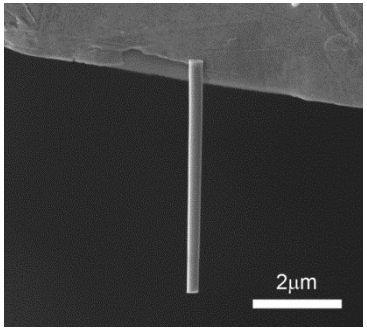
Propagation loss of different transverse modes.

Metal substrate generates a mode-dependent propagation loss

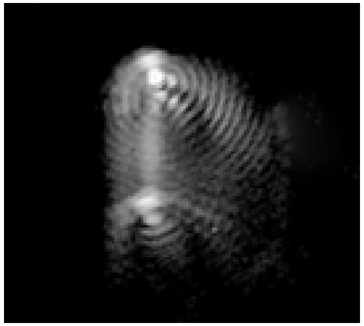
Lasing Polarization Properties of Freestanding GaN NWs



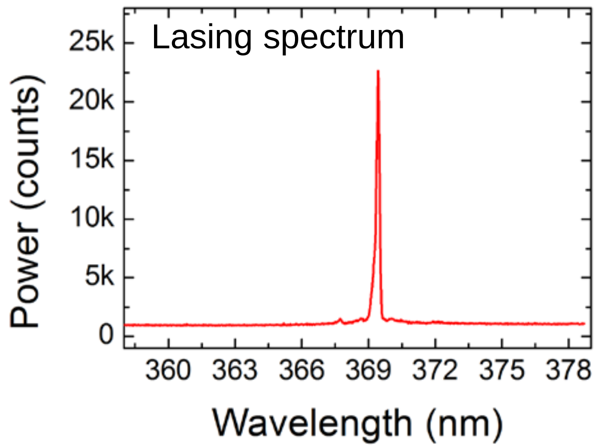
End-facet optical characterization setup

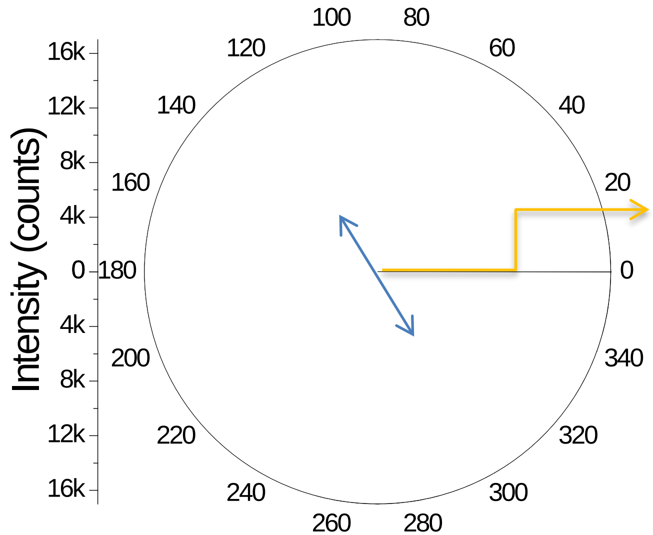


SEM image of a nanowire hanging off substrate



CCD image of lasing nanowire hanging off substrate





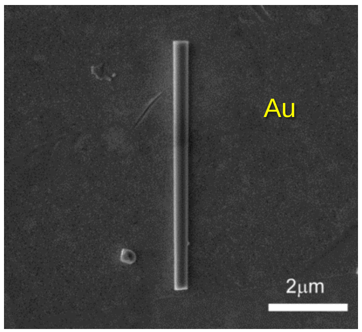
Indicative of elliptical polarization

Polarization angle random wrt surface

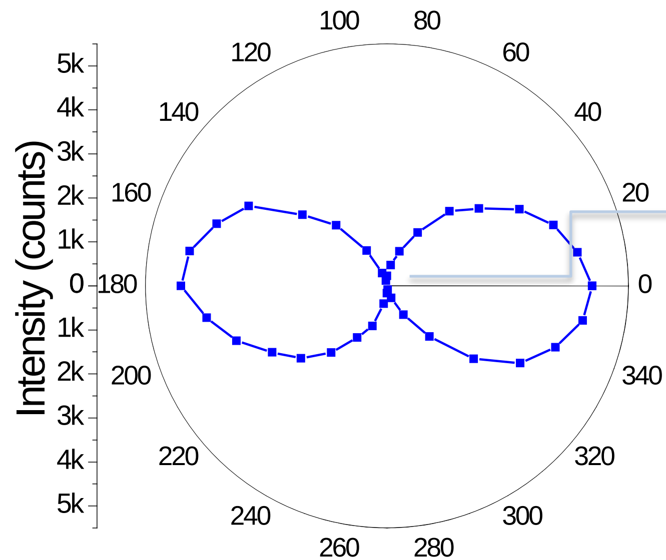
Intensity vs. the rotation of the polarizer

Polarization Control: Nanowire on Gold Substrate

Polarization control (linear polarization) desired for many applications

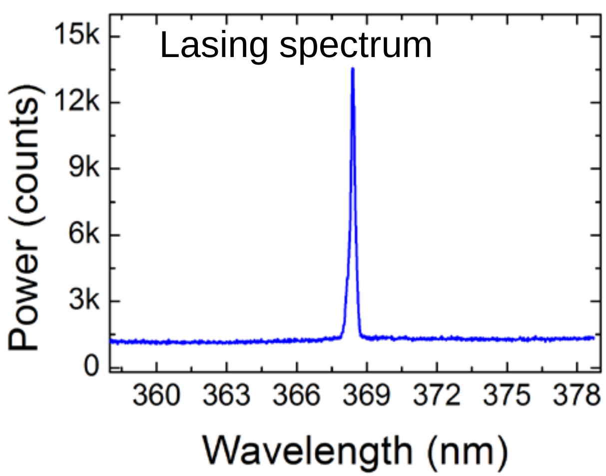


nanowire on **gold**



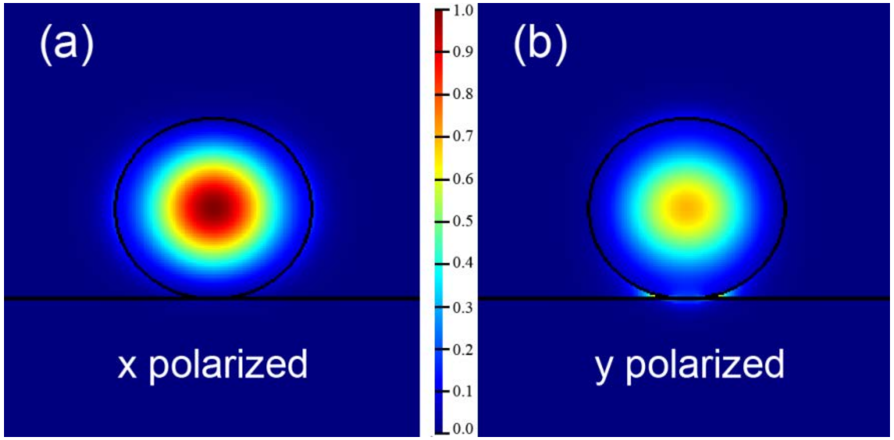
Indicative of linear polarization

Intensity vs. the rotation of the polarizer



- ✓ Single-wavelength lasing
- ✓ Linear polarization
- ✓ Large cross-polarization suppression ratio (CPSR): 21:1
- ✓ 0º polarization angle

Why Does Linear Polarization Happen on Gold?



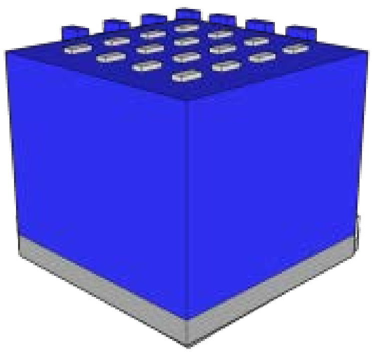
Fundamental modes supported by the nanowire-gold geometry

- ✓ Differently polarized modes experience different cavity losses
- ✓ Loss for x-polarized mode: 0.36 dB/mm
- ✓ Loss for y-polarized mode: 2.11 dB/mm

A nanowire on gold substrate experiences a larger cavity loss for the perpendicularly polarized mode, leading to ***linear polarization*** with a fixed polarization parallel to the substrate

H. Xu et al., "Polarization control in GaN nanowire lasers", *Optics Express*, **22**, 19198 **(2014)**.

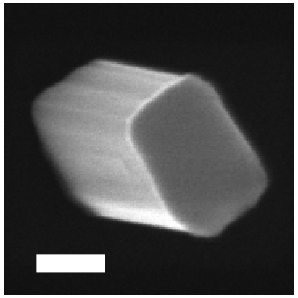
Intrinsic polarization control from rectangular nanowire lasers



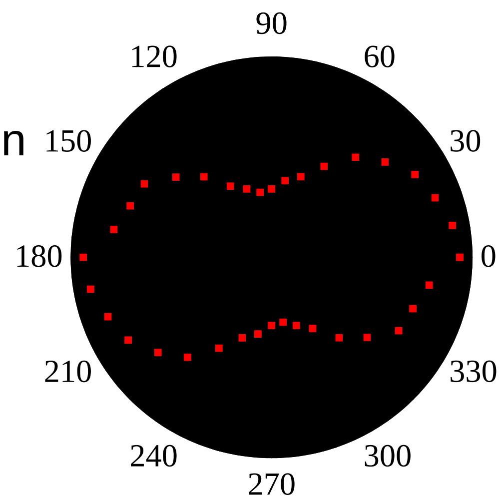
- Top-down approach enables *cross-sectional shape control* of NWs
- Previous work: change from elliptical to linearly polarized emission when nanowire placed on gold substrate H. Xu et al., *Optics Express*, ***22***, 19198 **(2014)**.
- Rectangular GaN nanowire lasers show *intrinsic linearly polarized emission*, obviating the need for manipulation by external environments

Li, Changyi, et al. "Intrinsic polarization control in rectangular GaN nanowire lasers." *Nanoscale* **8**, 5682 (2016).

Less rectangular

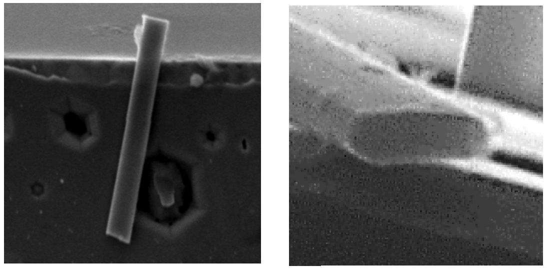


Y = 300nm
X = 450nm

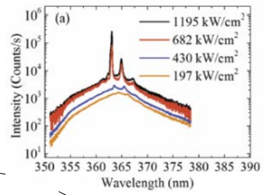


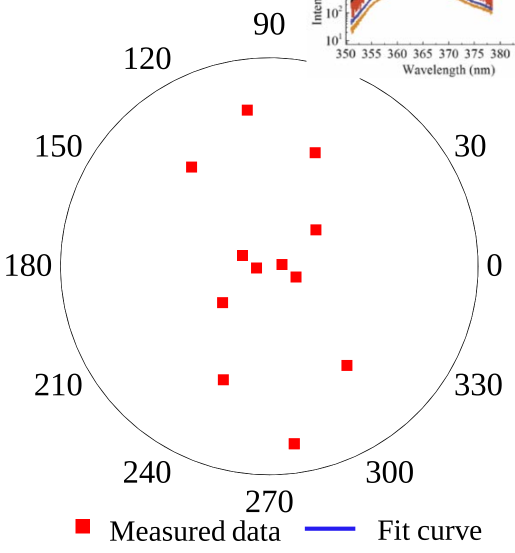
Elliptical polarization

More rectangular



Y = 120nm
X = 450nm



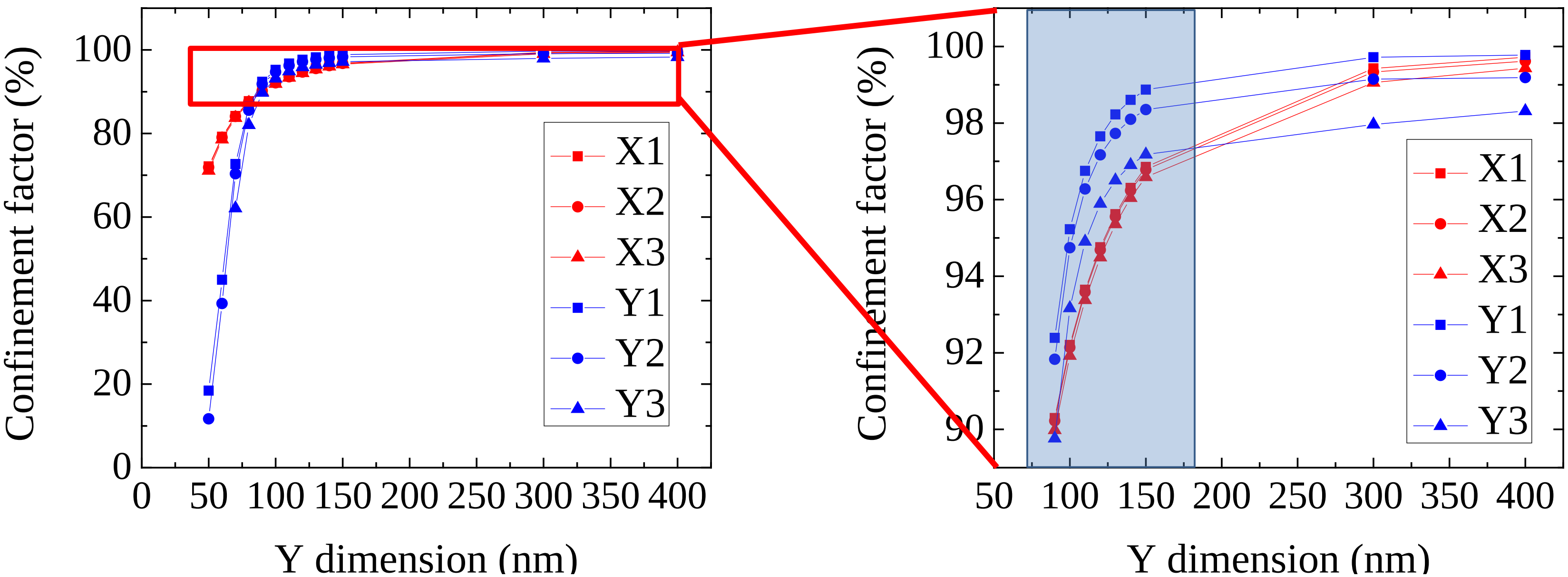


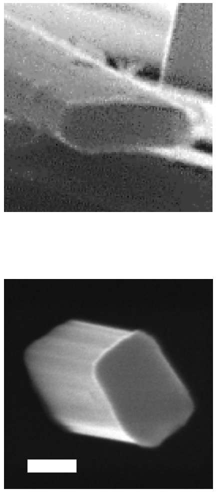
Linear polarization

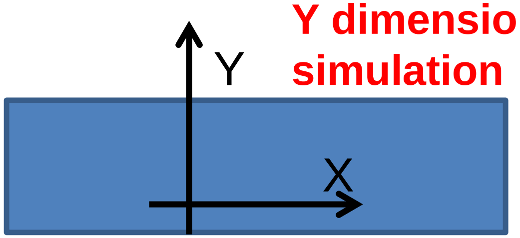
Mode simulation of rectangular cross-sectional GaN nanowire lasers

Sandia National Laboratories

Calculated confinement factors of transverse modes







Y dimension: varied during simulation

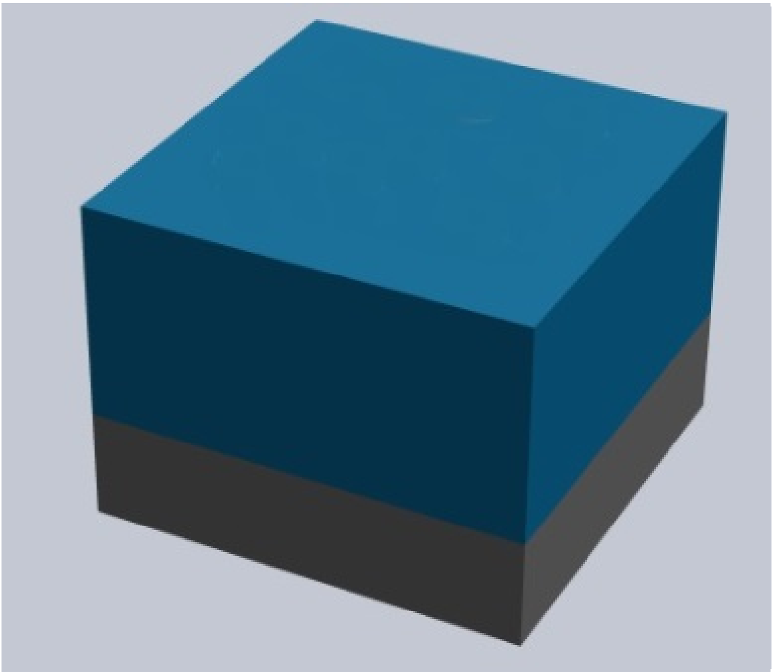
X dimension: fixed at 450 nm

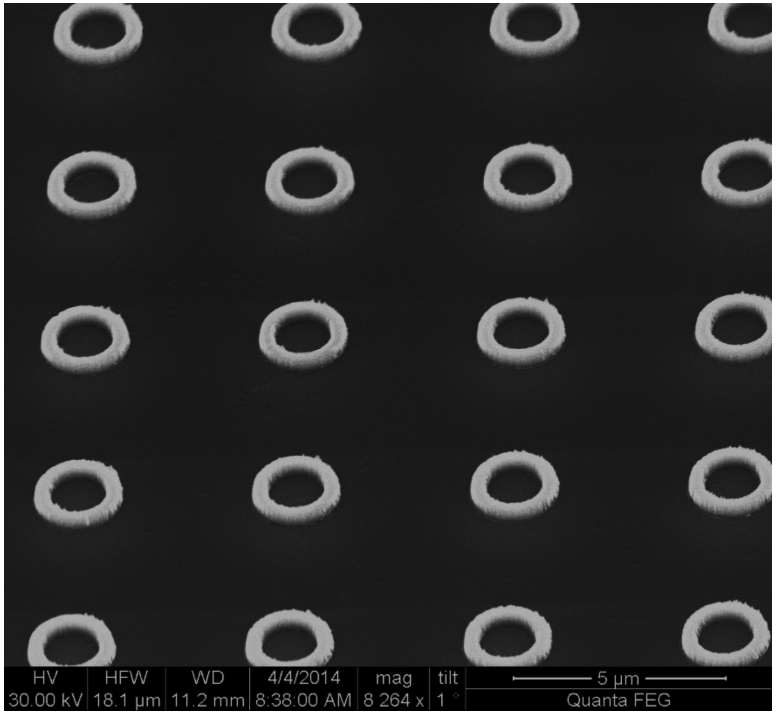
- Below Y-dimensions ~ 200 nm, confinement factors for Y-polarized modes dominate over X-polarized modes, leading to linearly polarized emission

Li, Changyi, et al. "Intrinsic polarization control in rectangular GaN nanowire lasers." *Nanoscale 8*, 5682 (2016).

Cross-sectional shape control - GaN nanotubes

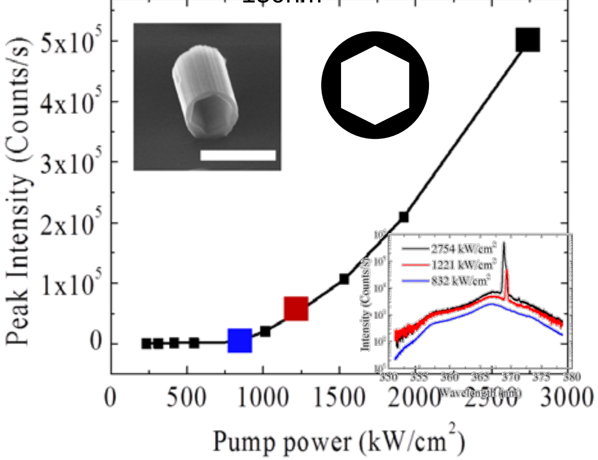
EBL Patterning and PMMA Lift-off
ICP dry etch
AZ400K wet etch



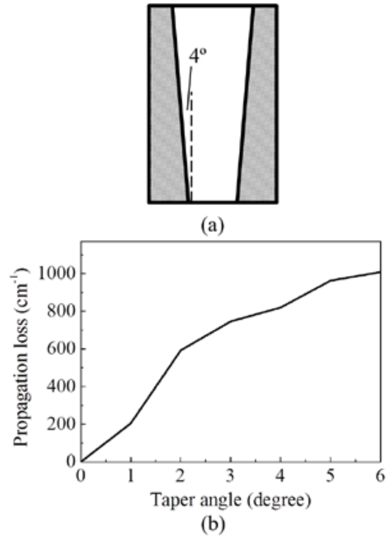


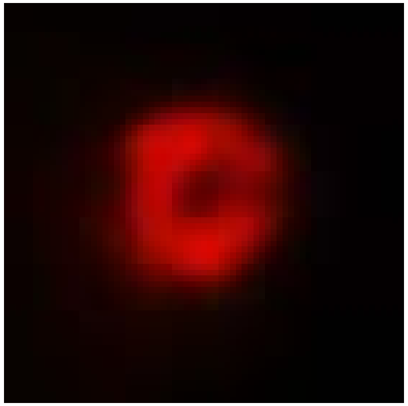
Annular emission from GaN nanotube lasers

- Length: 4µm
- Outer Diameter: 1.3µm
- Thickness: 150nm

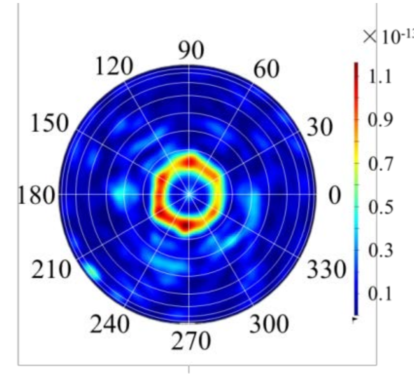


Threshold pump density = 1055 kW/cm²





CCD image far field lasing emission from nanotube



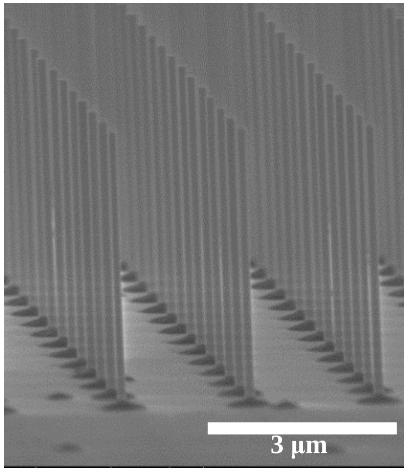
Simulated far field pattern of nanotube

- Higher threshold of nanotube vs. nanowire may be due in part to inner taper
- Hollow nanotube geometry potentially useful for optofluidic/biosensing applications (lases in silicone oil but with higher threshold)
- Observed annular shaped emission agrees with predicted emission and shows *use of cross-sectional shape control for beam shaping* in nanowire lasers

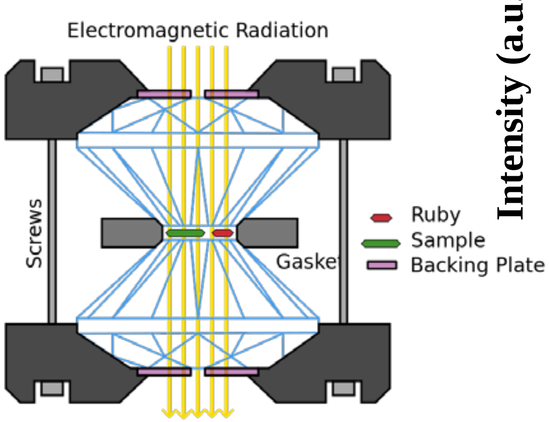
C. Li et al., "Annular-Shaped Emission from Gallium Nitride Nanotube Lasers", *ACS Photonics*, **2**, 1025 **(2015)**.

Continuous & dynamic wavelength tuning in GaN NW lasers via hydrostatic pressure

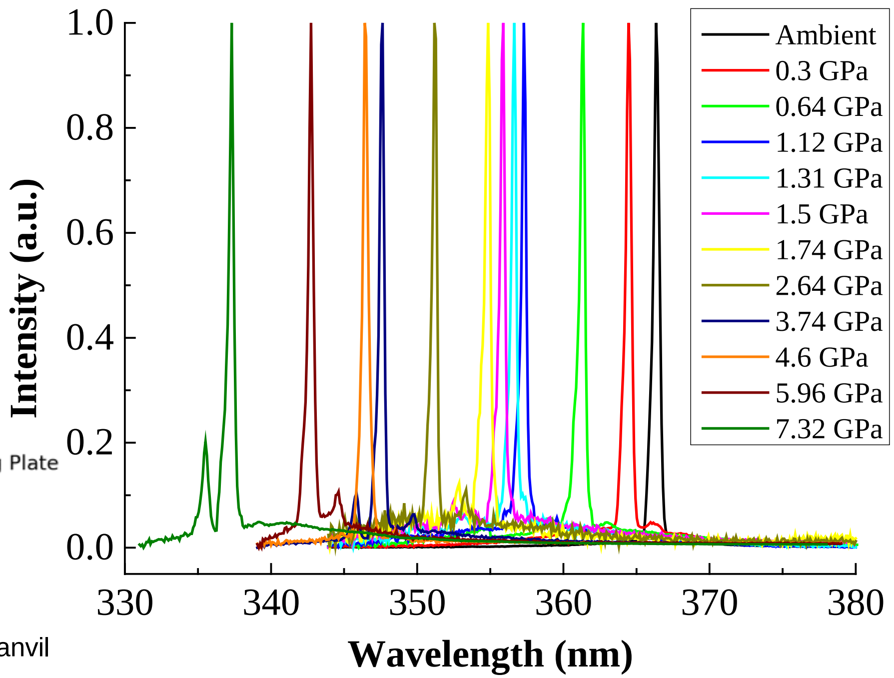
NWs patterned using e-beam lithography:
Diameter ~140 nm
Length ~ 4µm



Diamond Anvil Cell: Hydrostatic pressure medium (silicone oil) transmits pressure isotropically



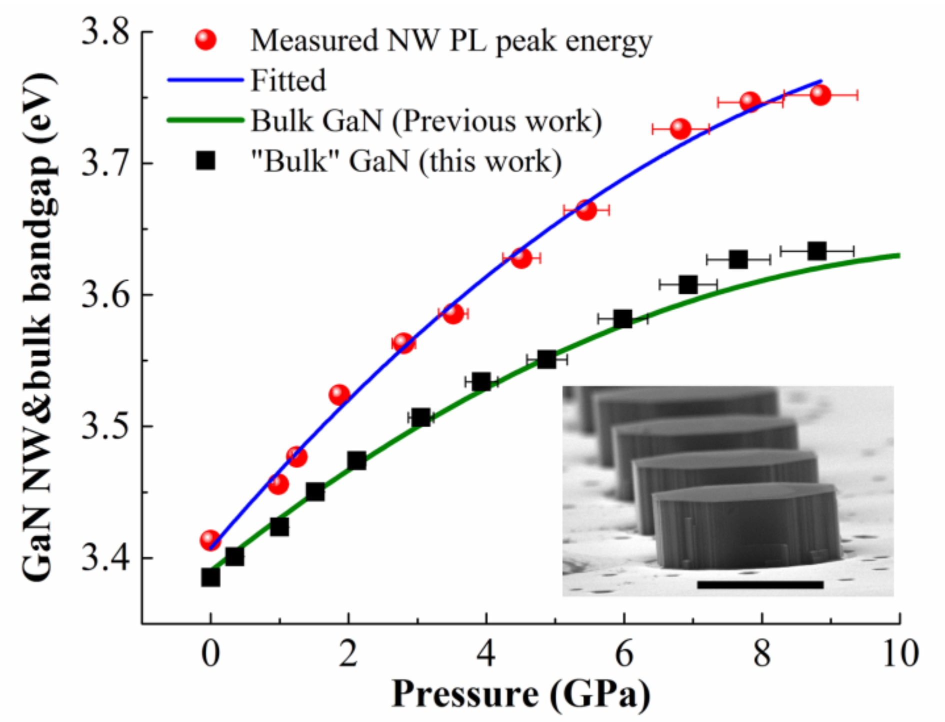
http://en.wikipedia.org/wiki/Diamond_anvil_cell



- Applied pressure induces bandgap increase
- Continuous and reversible tuning ~30 nm lasing tuning demonstrated over ~7.3 GPa (previous works were wavelength selection)
- Well defined fine tuning (<0.1nm) possible with current setup

S. Liu, C. Li, J. J. Figiel, S. R. Brueck, I. Brener, G. T. Wang, *Nanoscale*, **7**, 9581 **(2015)**.

Continuous & dynamic bandgap tuning in GaN NW lasers via hydrostatic pressure

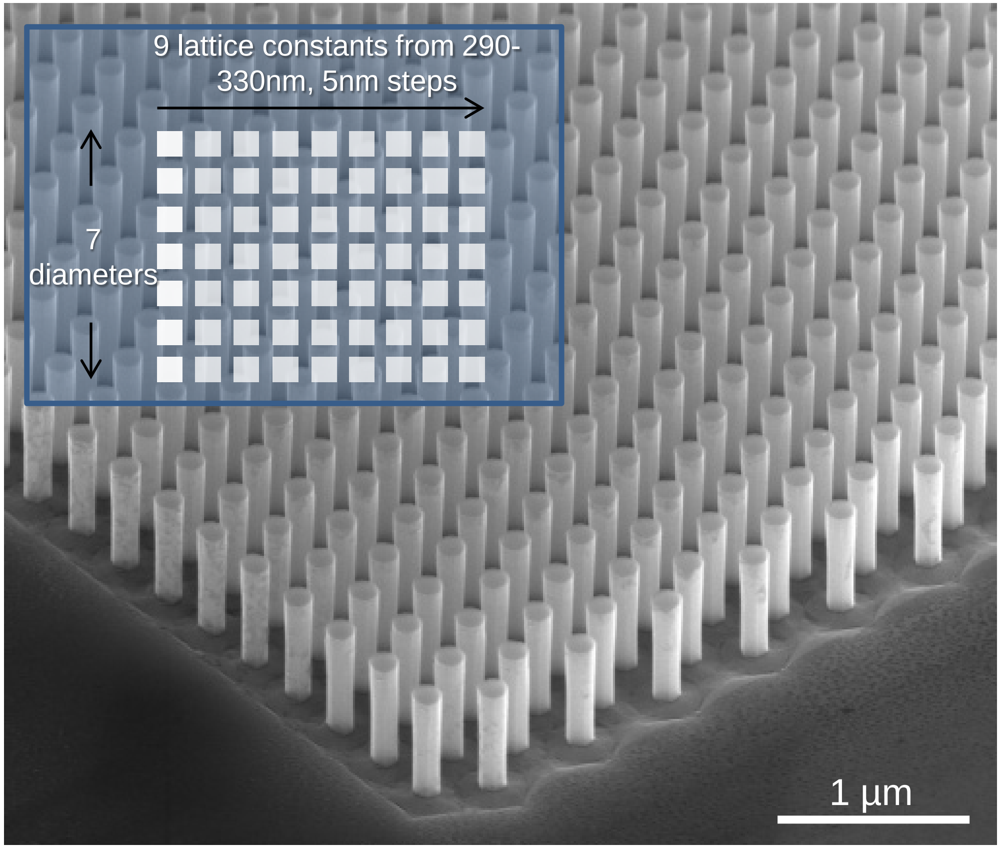


- Pressure coefficient ~40% higher for GaN NWs vs GaN micropillars or bulk GaN, allows for larger tuning range.

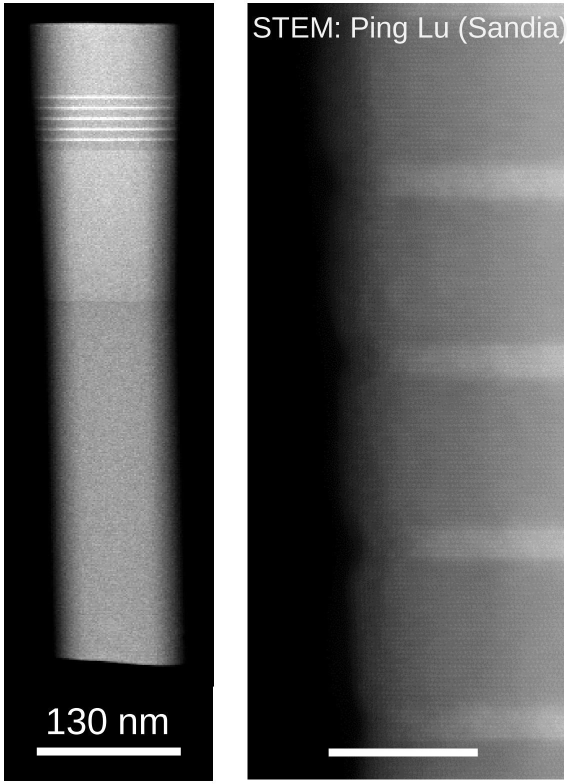
S. Liu, C. Li, J. J. Figiel, S. R. Brueck, I. Brener, G. T. Wang, *Nanoscale*, ***7***, 9581 **(2015)**.

Wavelength tuning in III-N Nanowire Photonic Crystal Lasers

Motivation: Achieve single-mode, tunable lasing on same chip. Applications in optical information processing, biology, solid state lighting, displays, etc.



Nanowire PCs fabricated by top-down method using e-beam lithographic mask

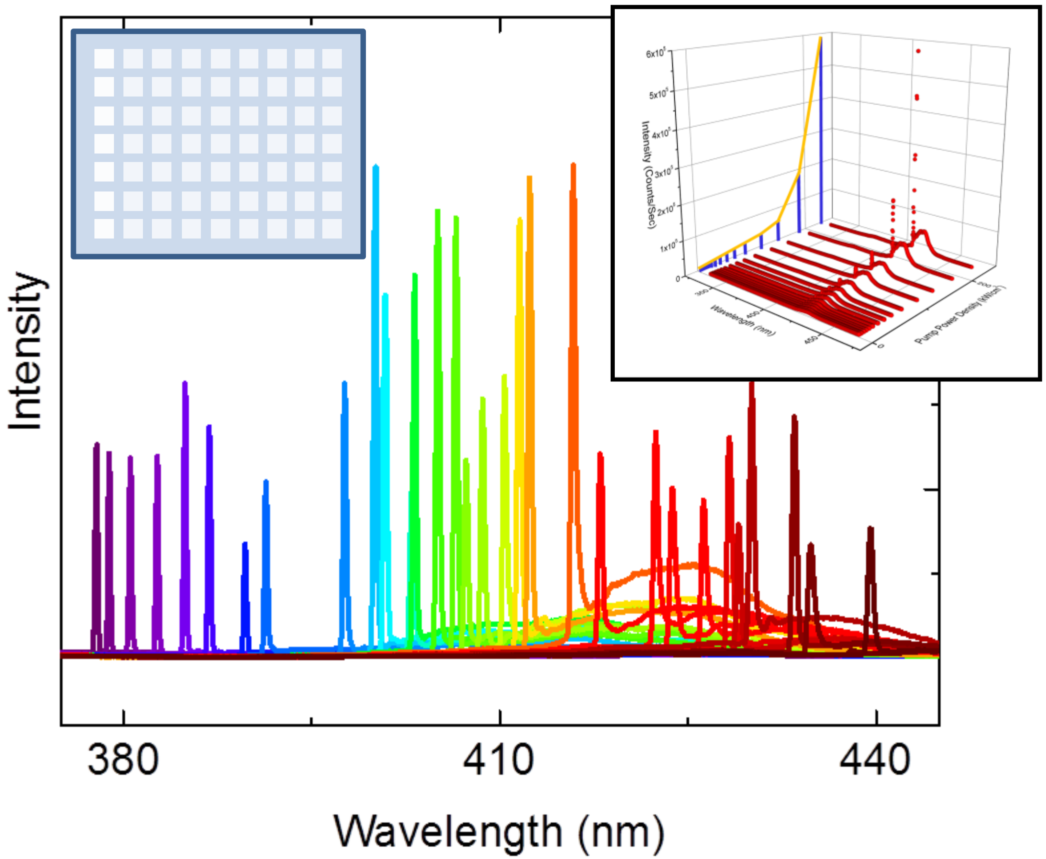


NW STEM images: 5x MQW InGaN emission centered at 420nm, $In_{0.02}GaN$ underlayer

Wavelength tuning in III-N Nanowire Photonic Crystal Lasers

Broad gain width of InGaN MQWs with PC design allows for tunable single mode lasing over large range on same chip

61 color nanowire laser array



- High-yield >95% (2 of the PCLs were accidentally removed during sample handling.)
- Spectral Coverage from **380-440nm on same chip**
- Emission wavelength increases with the diameter and the lattice constant
- Thresholds are reasonable compared to other optically pumped III-N nanowire devices. (<500kW/cm^2 for all devices)

J.B. Wright et al., "Multi-Colour Nanowire Photonic Crystal Laser Pixels," *Scientific Reports* 3, Article number: 2982 (2013)

George T. Wang

Top-down fabrication of AlGaN nanowires

After ICP etch only

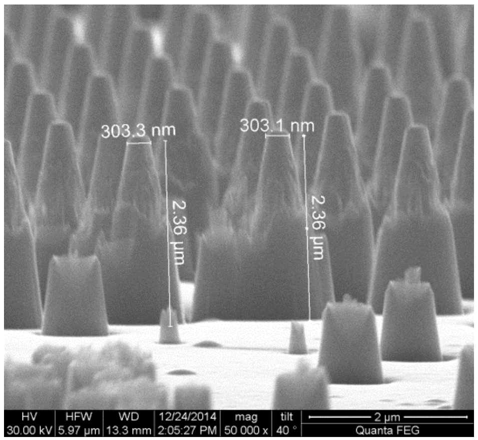
After AZ400K 65°C wet etch

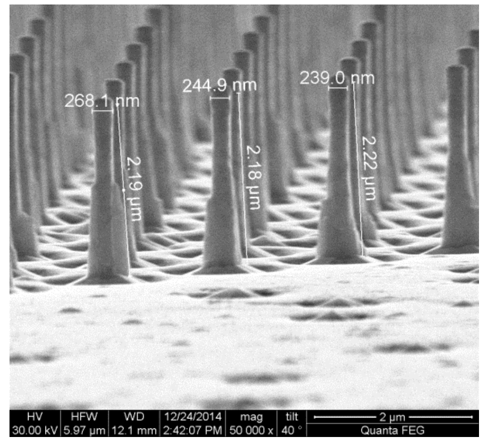
240-500nm diameter

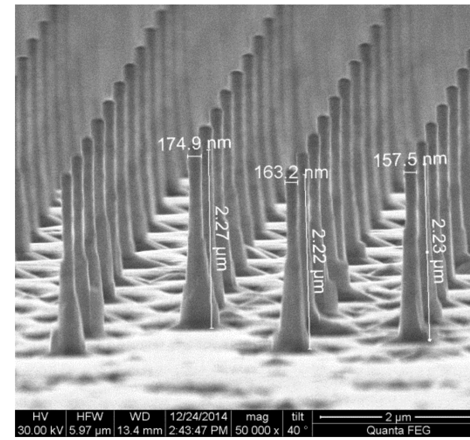
150-250 nm diameter

30% AlGaN

4.0 µm $Al_{0.31}Ga_{0.69}N$ on 1.6 µm AlN on *c*-sapp

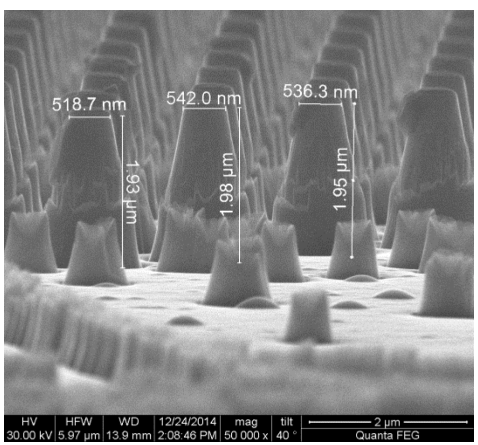


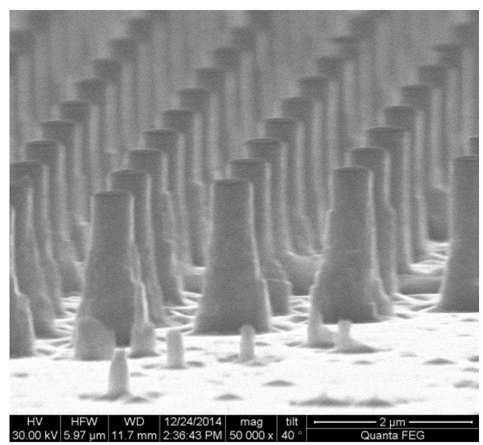


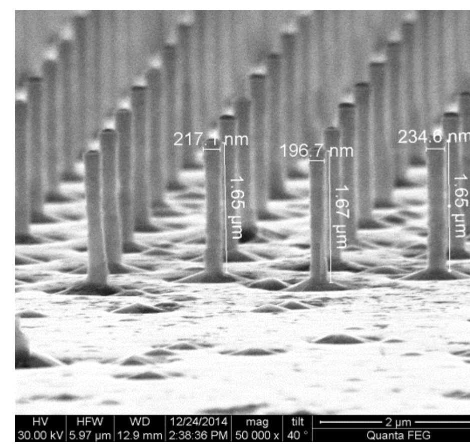


70% AlGaN

3.0 µm $Al_{0.71}Ga_{0.29}N$ on 1.5 µm AlN on *c*-sapp

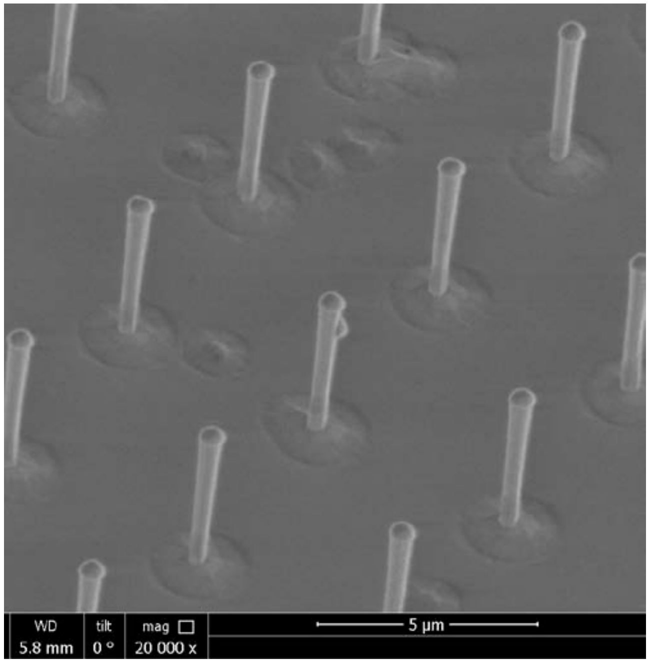


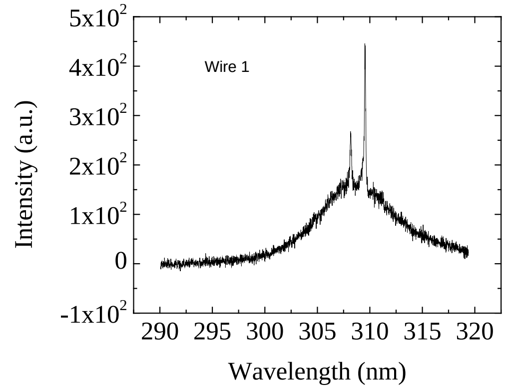


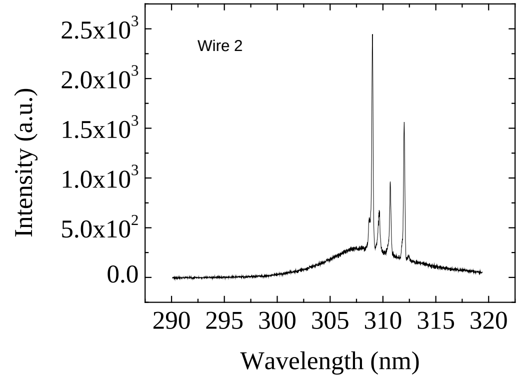


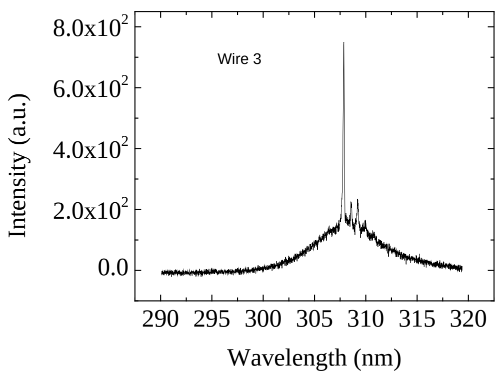
AlGaN samples grown by Andrew Allerman

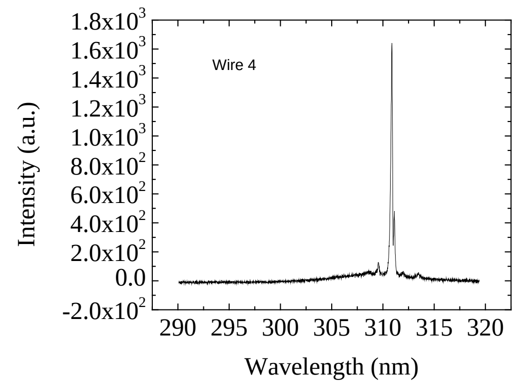
Top-down AlGaN nanowires - lasing achieved











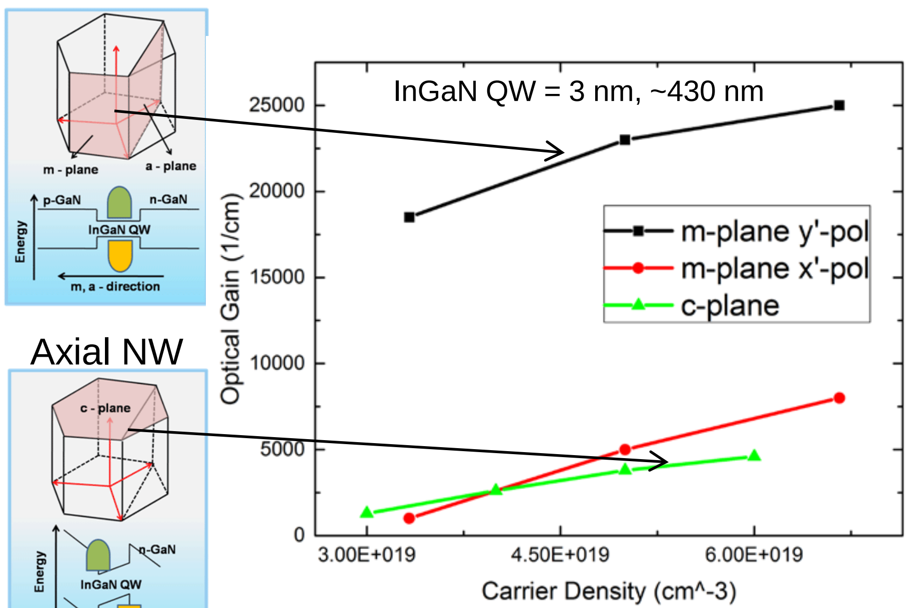
Nanowire Length: 4.0 – 4.1 um
Diameter: Top ~420-440 nm, bottom: 350-380 nm

- Single top-down $Al_{0.31}Ga_{0.69}N$ nanowires show multimode lasing ~308-312 nm
- Estimated threshold ~200 kW/cm^2 (similar to top-down GaN nanowires)
- *Nanowires burn-out after only ~3-6 seconds* (less resilient than GaN nanowires!?)

Optical gain for axial vs radial InGaN/GaN nanowire designs

Optical Gain - Crystallographic Dependence

Radial NW

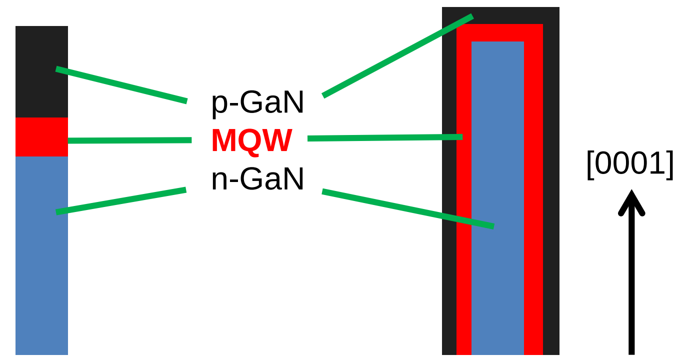


Crystallographic figures from: http://csel.snu.ac.kr/research/LED.php

Much higher optical gain for m-plane QWs (benefits radial NW design)

Optical Gain – Axial vs. Radial Geometry

L = 6 µm, R = 250nm, Thickness of MQW = 20nm



Axial structure

$$V_{active} = 0.003925\ \mu m^3$$

Radial structure

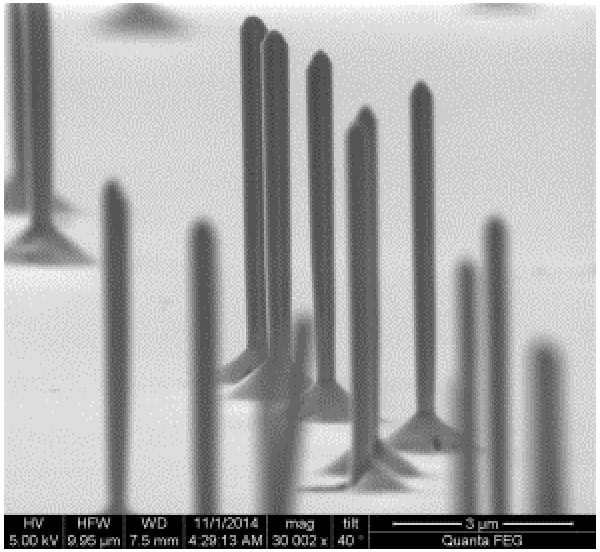
$$V_{active} = 0.1884\ \mu m^3$$

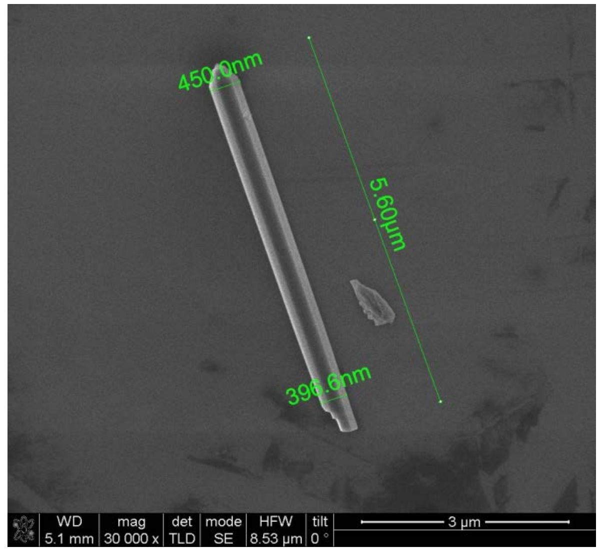
Radial: 48 times larger gain region compared to axial

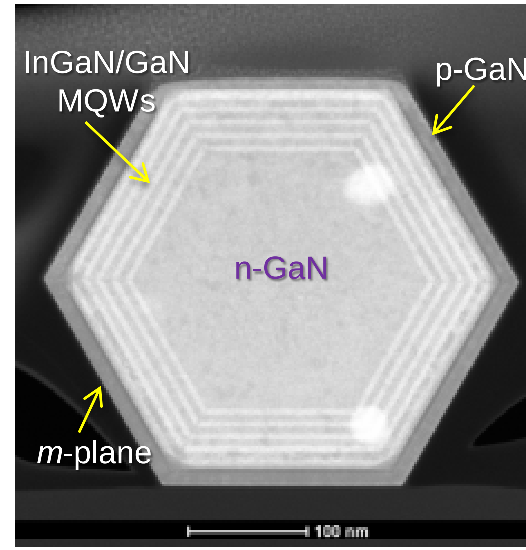
S.-H. Park and D. Ahn, "Optical polarization characteristics of m-plane InGaN/GaN quantum well structures and comparison with experiment," *Appl. Phys. Lett.*, **103**, *pp. -, (2013)*

J. Piprek, R. K. Sink, M. A. Hansen, J. E. Bowers, and S. P. DenBaars, "Simulation and optimization of 420-nm InGaN/GaN laser diodes," 2000, pp. 28-39.

Synthesis of core-shell p-i-n InGaN/GaN core-shell NW lasers

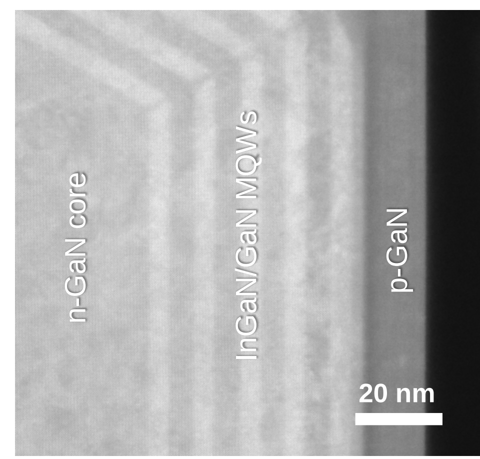






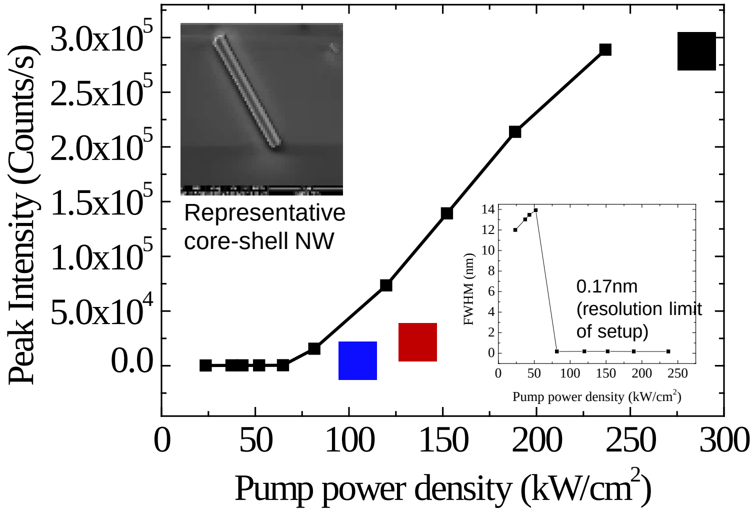
Core-shell radial p-i-n InGaN/GaN MQW nanowires following MOCVD regrowth on top-down fabricated n-type GaN nanowires

- Regrowth on top-down n-GaN nanowire results in taper. Growth of semipolar planes also results in non-flat tip, which may lower mirror reflectivity
- Growth conditions, layer thicknesses, varied over several runs, then nanowires tested by optical pumping for lasing

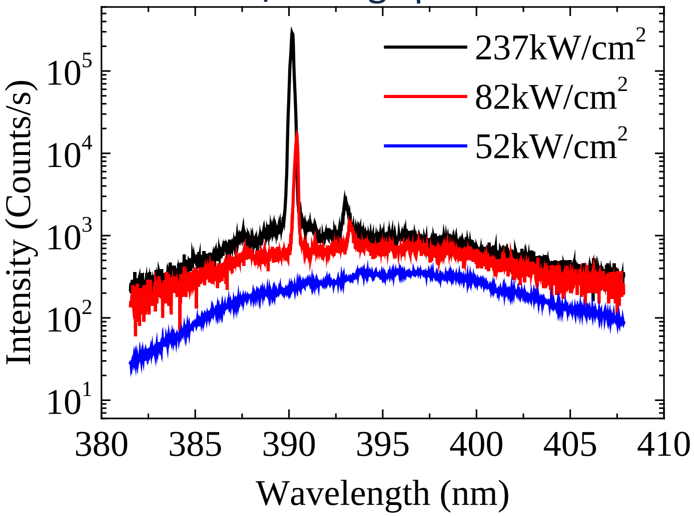


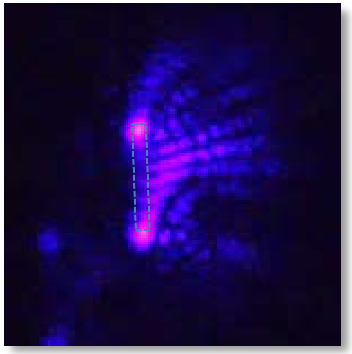
STEM by Ping Lu

Optically pumped lasing from radial nonpolar p-i-n InGaN/GaN MQW nanowires



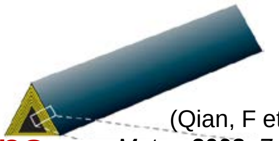
PL/Lasing spectra





CCD image of lasing NW

- Threshold pump density = 75 kW/cm^2
- Significantly lower (~10x) than thresholds for semipolar InGaN/GaN core-shell nanowires (~700-2000 kW/cm^2) despite much shorter lengtns



(Qian, F et al. C. M. *Nat. Mater.* **2008**, *7* (9), 701–706.)

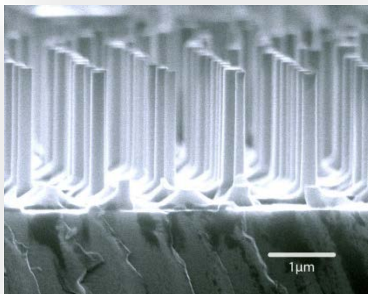
- First non-polar GaN/InGaN MQW core-shell single NW laser
- Demonstrates feasibility of p-i-n core-shell architecture for future electrically injected single nanowire lasers

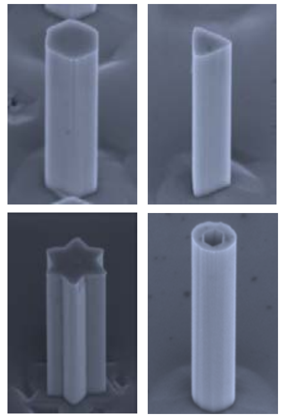
C. Li et al., submitted, *Nano Letters*

Summary

Precision top-down fabrication

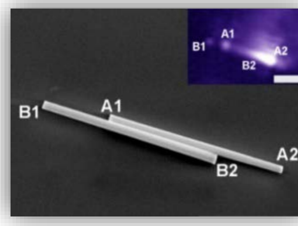
Controlled geometries and material properties

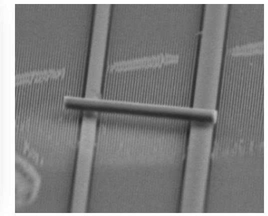


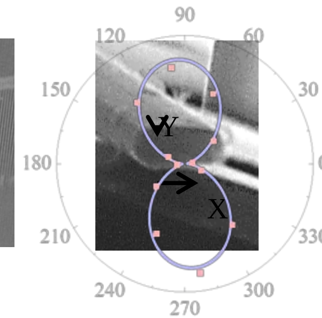


Q. Li et al., *Optics Express* **19**, 25528 (2011)

Mode & polarization control in GaN nanowire lasers



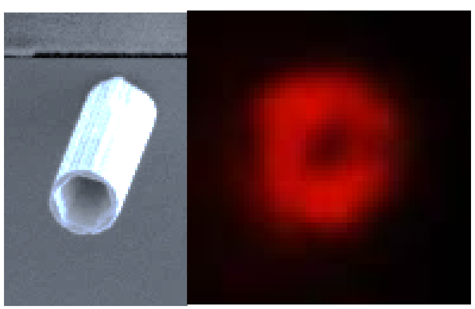




Q. Li et al., *Optics Express* **20** 17874 (2012)
H. Xu et al., *Appl. Phys. Lett.* **101** 113106 (2012)
H. Xu et al., *Appl. Phys. Lett.* **101** 221114 (2012)
J.B. Wright et al., *Appl. Phys. Lett.*, 104, 041107 (2014)
Li, Changyi, et al. *Nanoscale* ***8***, 5682 (2016).

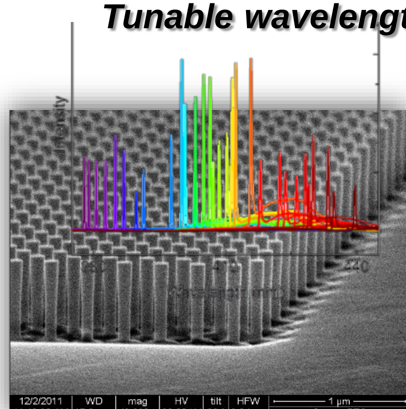
Beam shape control

nanotube lasers

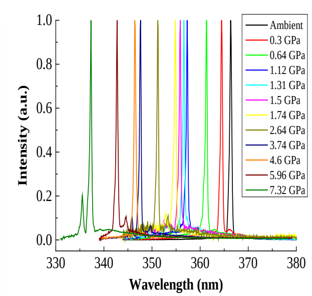


C. Li et al., *ACS Photonics*, **2**, 1025 **(2015)**.

Tunable wavelength nanowire lasers

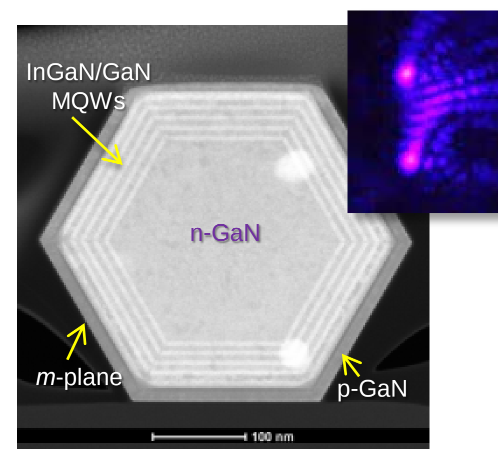


J.B. Wright et al., *Sci. Reports* 3, Art no. 2982 (2013) doi:10.1038/srep02982

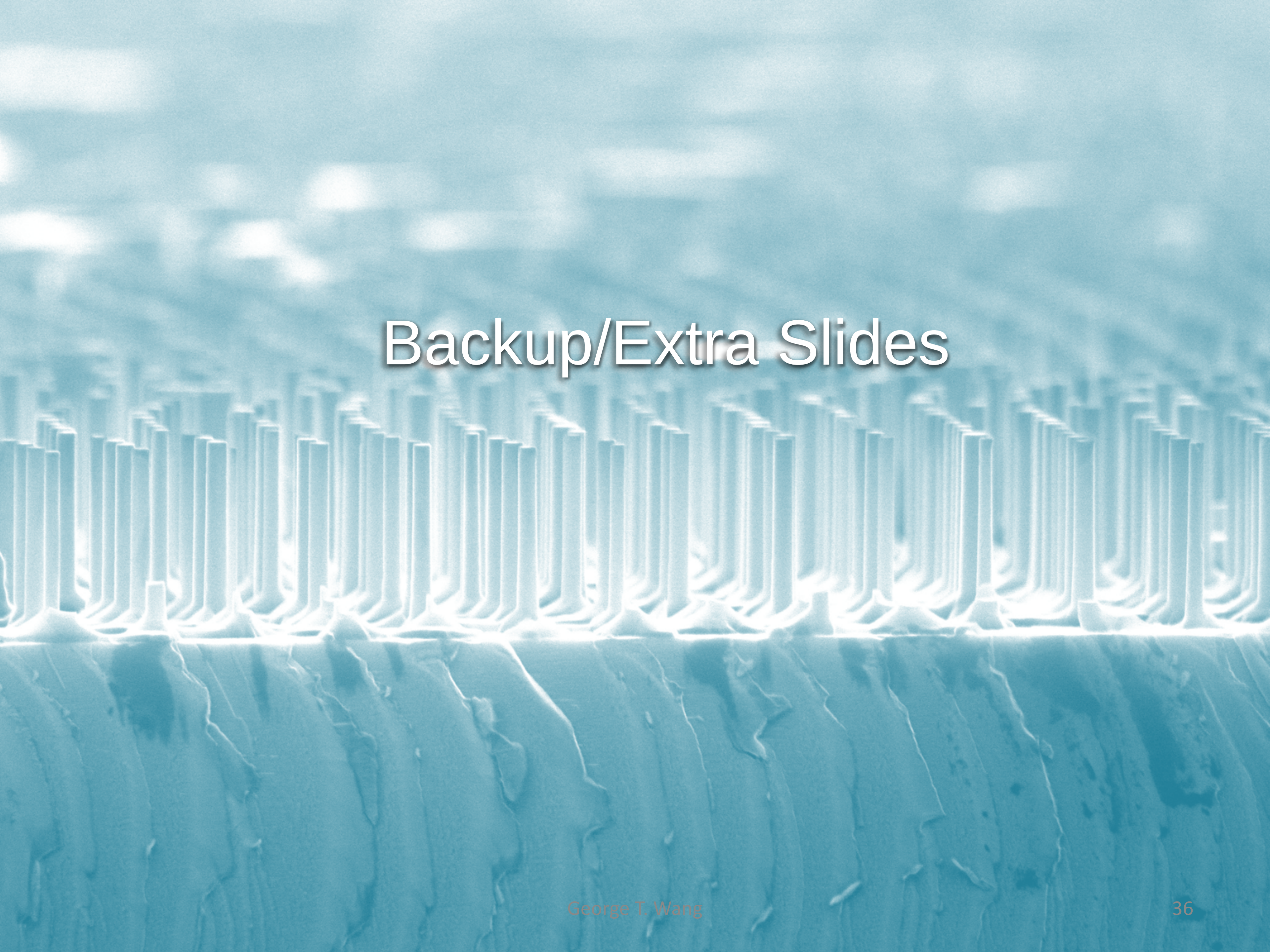


S. Liu, C. Li, J. J. Figiel, S. R. Brueck, I. Brener, G. T. Wang, *Nanoscale*, *7*, 9581 **(2015)**.

Nonpolar core-shell nanowire lasers



C. Li et al., *Nano Lett*, submitted



Backup/Extra Slides

Acknowledgments

Qiming Li, Jeremy Wright, Huiwen Xu, Changyi Li (Ph. D student), Sheng Liu, Igal Brener, Willie Luk, Weng Chow, Steven Brueck (U. of New Mexico), Daniel Feezell (U. of New Mexico), Ganesh Subramania – Nanowire lasers

Ping Lu – Scanning TEM/EDS

Benjamin Leung and Miao-Chan Tsai – GaN and AlGaN nanowire fabrication

Daniel D. Koleske, Jeffrey Figiel - GaN/InGaN MOCVD growth

Andrew Allerman, Mary Crawford – AlGaN growth

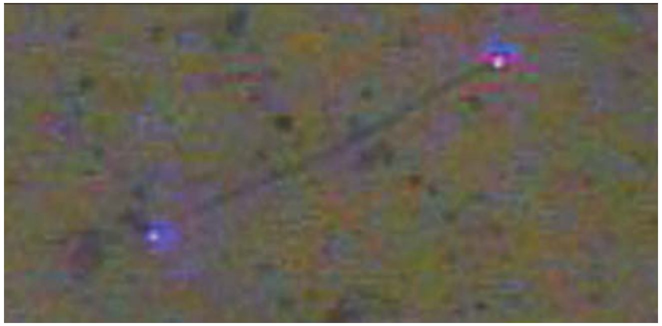
This work was performed, in part, at the Center for Integrated Nanotechnologies (CINT), a U.S. Department of Energy, Office of Basic Energy Sciences user facility.

Funding Acknowledgment: DOE Basic Energy Sciences (BES) DMSE, Sandia's Solid-State-Lighting Science Energy Frontier Research Center (DOE BES), and Sandia's LDRD program

Contact: e-mail: gtwang@sandia.gov http://www.sandia.gov/~gtwang

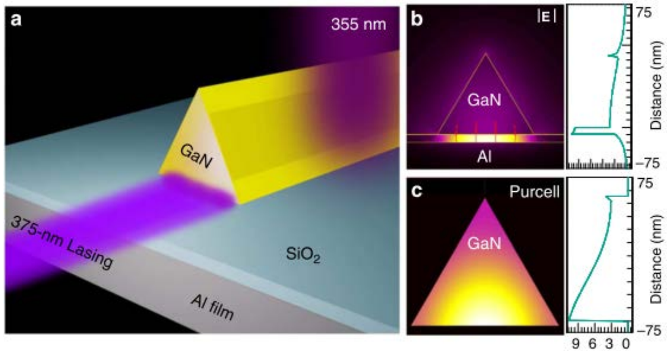
Types of III-nitride nanowire lasers

Fabry-Perot cavity



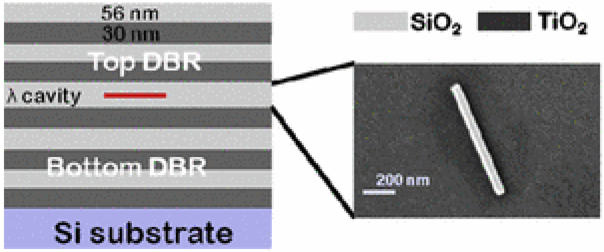
Johnson, Justin C., et al. "Single gallium nitride nanowire lasers." *Nature materials* 1.2 (2002): 106-110.

Surface plasmon nanowire laser



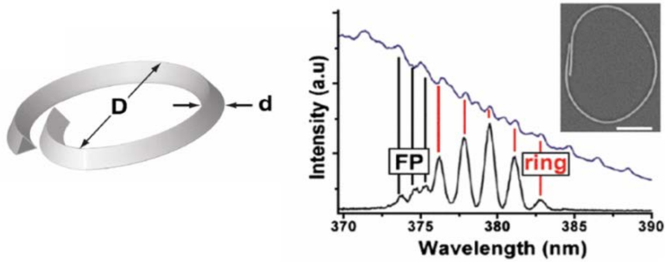
Zhang, Qing, et al. "A room temperature low-threshold ultraviolet plasmonic nanolaser." *Nature communications* 5 (2014).

Nanowire polariton laser



Das, Ayan, et al. "Room temperature ultralow threshold GaN nanowire polariton laser." *Physical review letters* 107.6 (2011): 066405.

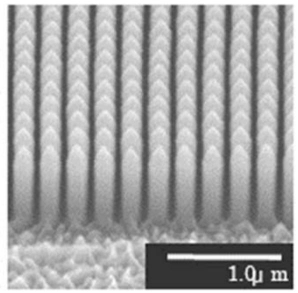
Nanowire ring laser

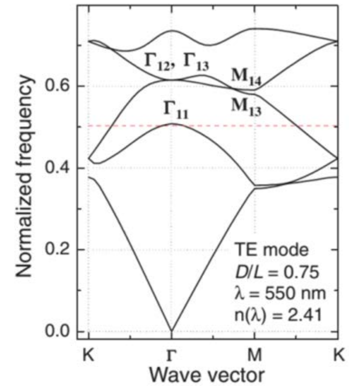


Pauzauskie, Peter J., Donald J. Sirbuly, and Peidong Yang. "Semiconductor nanowire ring resonator laser." *Physical review letters* 96.14 (2006): 143903.

Types of III-nitride nanowire lasers

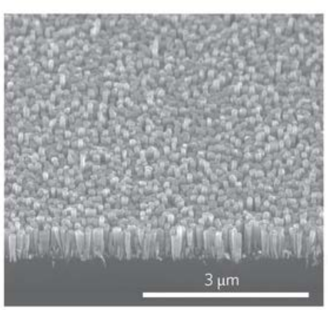
Photonic crystal nanowire laser

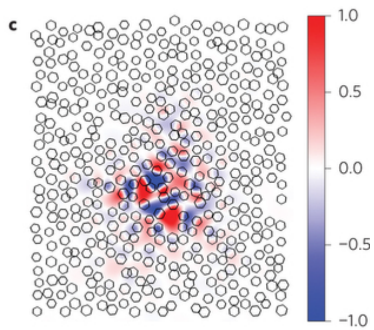


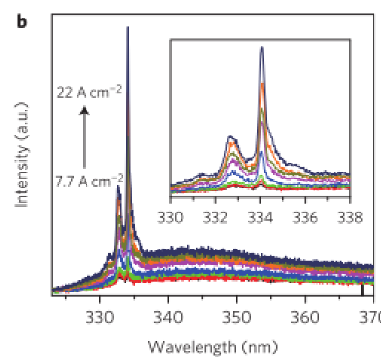


Kouno, Tetsuya, et al. "Two-dimensional light confinement in periodic InGaN/GaN nanocolumn arrays and optically pumped blue stimulated emission." *Optics express* 17.22 (2009): 20440-20447.

Nanowire random laser





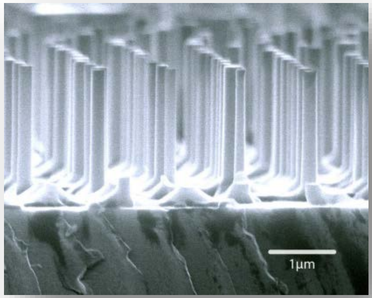


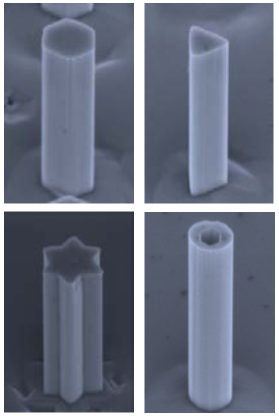
Li, K. H., et al. "Ultralow-threshold electrically injected AlGaN nanowire ultraviolet lasers on Si operating at low temperature." *Nature nanotechnology*10.2 (2015): 140-144.

Summary

Precision top-down fabrication

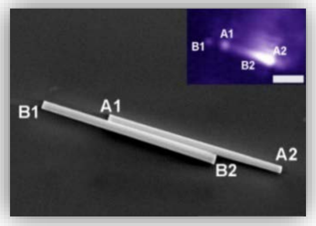
Controlled geometries and material properties

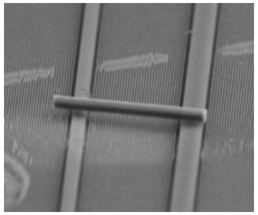


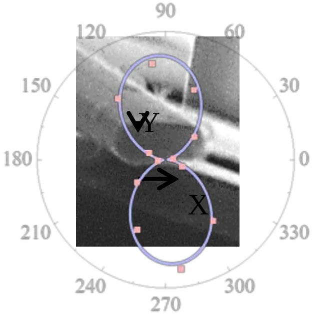


Q. Li et al., *Optics Express* **19**, 25528 (2011)

Mode & polarization control in GaN nanowire lasers

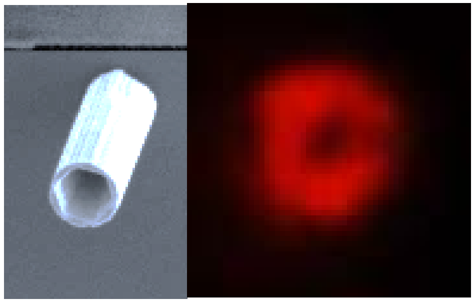






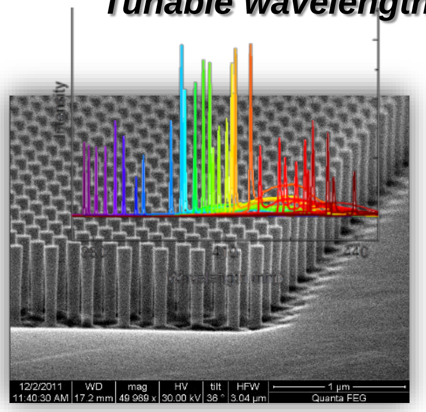
Q. Li et al., *Optics Express* **20** 17874 (2012)
H. Xu et al., *Appl. Phys. Lett.* **101** 113106 (2012)
H. Xu et al., *Appl. Phys. Lett.* **101** 221114 (2012)
J.B. Wright et al., *Appl. Phys. Lett.*, 104, 041107 (2014)
Li, Changyi, et al. *Nanoscale* ***8***, 5682 (2016).

Beam shape control nanotube lasers

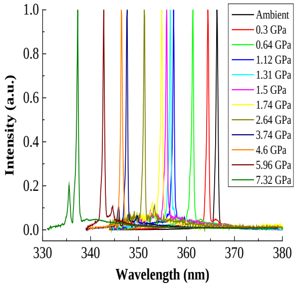


C. Li et al., *ACS Photonics*, **2**, 1025 **(2015)**.

Tunable wavelength nanowire lasers

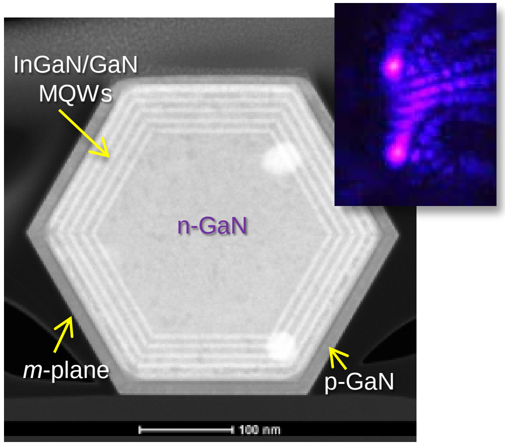


J.B. Wright et al., *Sci. Reports* 3, Art no. 2982 (2013) doi:10.1038/srep02982



S. Liu, C. Li, J. J. Figiel, S. R. Brueck, I. Brener, G. T. Wang, *Nanoscale*, *7*, 9581 **(2015)**.

Nonpolar core-shell nanowire lasers



C. Li et al., *Nano Lett*, **2**, submitted

Straight GaN nanowires with controllable geometries

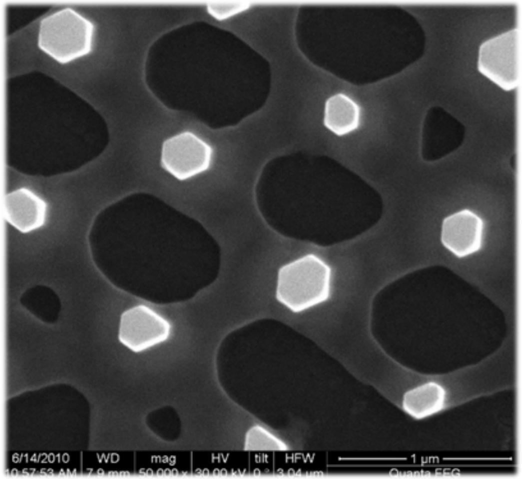
Wet etch rate negligible for top (Ga-polar) *c*-face & fast for semipolar facets, leads to vertical NWs with **straight (low taper) & smooth** facets

Superior and independent control of:

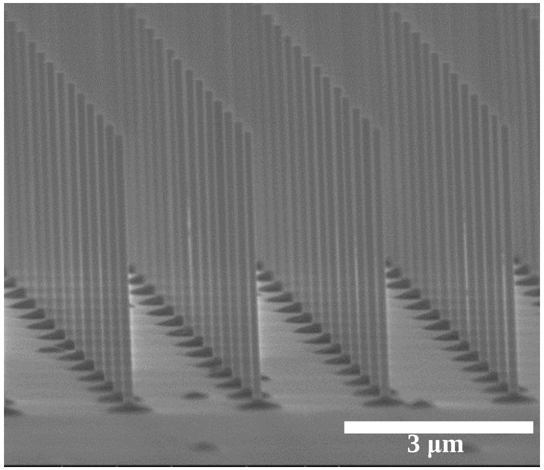
Height (dry etch depth)

Diameter (wet etch time)

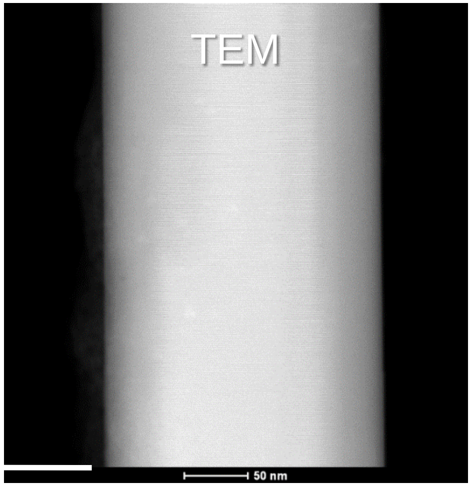
Pitch/arrangement (defined by masking template)



Following GaN regrowth



Mask using e-beam lithography



Smooth sidewall created by wet etch

TEM: Ping Lu, SNL

See: Q. Li et al., *Optics Express* **19**, 25528 (2011)

George T. Wang

Summary

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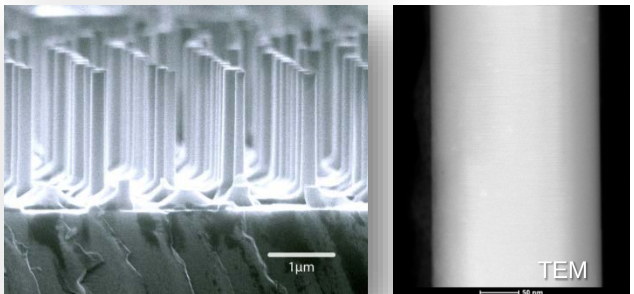
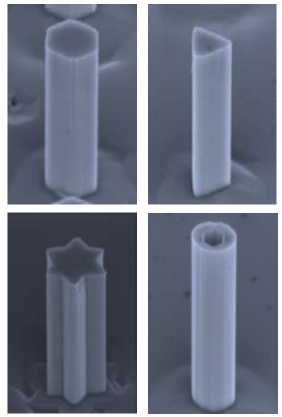
Controlled geometries and material properties





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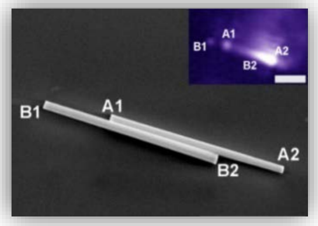
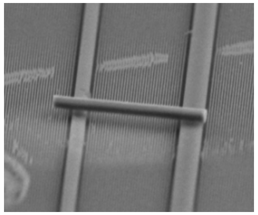
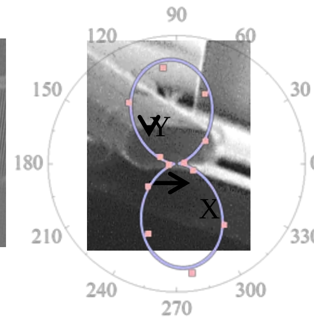


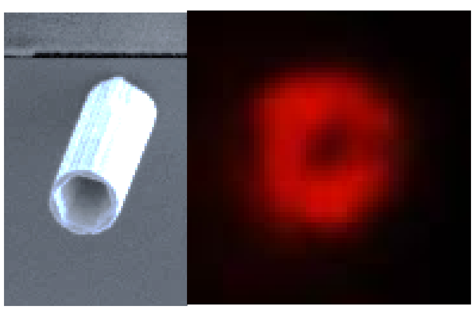




Q. Li et al., *Optics Express* **20** 17874 (2012)
H. Xu et al., *Appl. Phys. Lett.* **101** 113106 (2012)
H. Xu et al., *Appl. Phys. Lett.* **101** 221114 (2012)
J.B. Wright et al., *Appl. Phys. Lett.*, 104, 041107 (2014)
Li, Changyi, et al. *Nanoscale* ***8***, 5682 (2016).

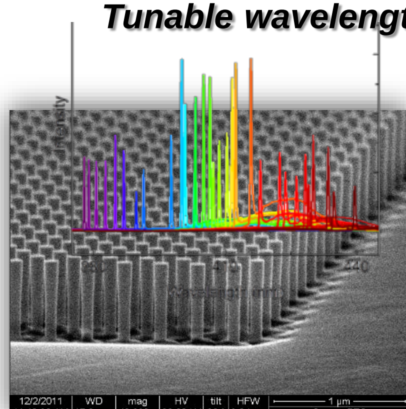
Beam shape control

nanotube lasers

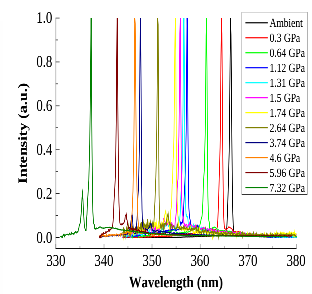


C. Li et al., *ACS Photonics*, **2**, 1025 **(2015)**.

Tunable wavelength nanowire lasers

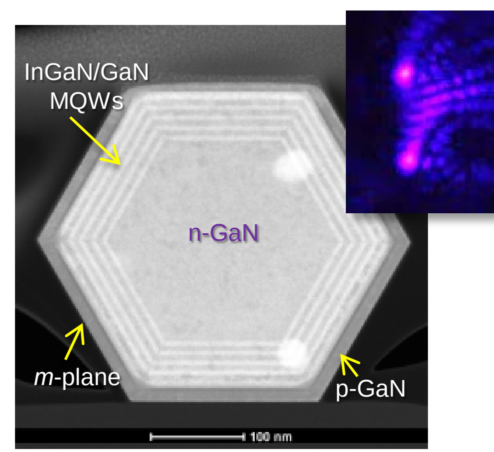


J.B. Wright et al., *Sci. Reports* 3, Art no. 2982 (2013) doi:10.1038/srep02982



S. Liu, C. Li, J. J. Figiel, S. R. Brueck, I. Brener, G. T. Wang, *Nanoscale*, *7*, 9581 **(2015)**.

Nonpolar core-shell nanowire lasers



C. Li et al., *Nano Lett*, submitted

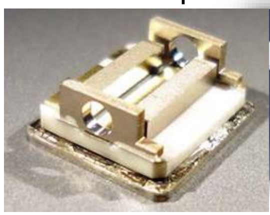
George T. Wang

Why Nanowire Lasers?

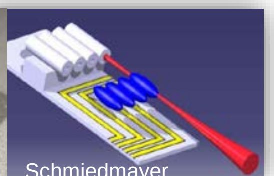
Integrated coherent nanophotonic elements for communications, sensing, imaging, lithography, lighting, etc.

Integrated nanophotonics, atom trapping, optical MEMS

atom trap



P. Schwindt (1725)

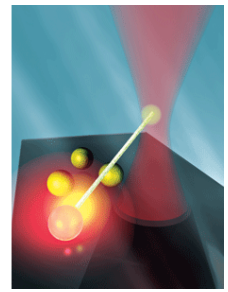


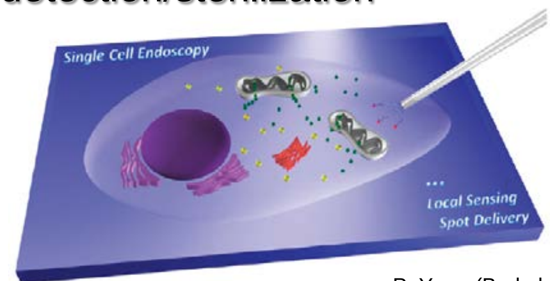
Schmiedmayer

J. Ford, Lucent

Small UV-visible laser for trapping, interconnects, switching, etc.

Optical nanoprobes & chem/bio detection/sterilization

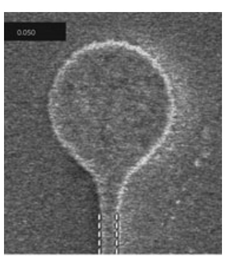




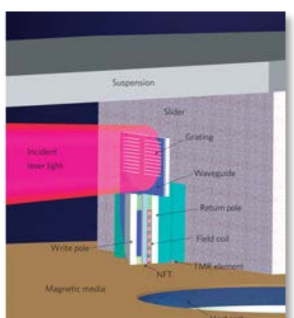
P. Yang (Berkeley)

Sub-λ resolution probes for localized excitation, detection

Nanolithography & Heat-assisted Magnetic Recording



Nature Photonics 3, 220 (2009)



Seagate

Subwavelength & efficient laser spot

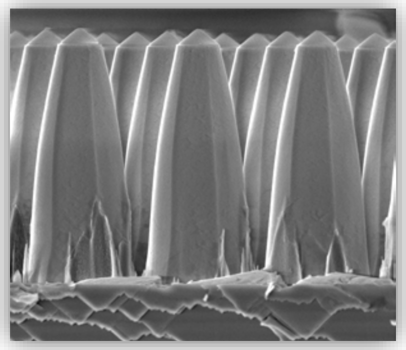
Lighting, projection, & holographic displays

Sony Crystal LED Display prototype

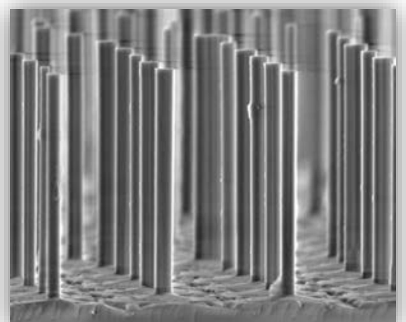
Low-power, speckle-free low coherence (random laser), multicolor pixels

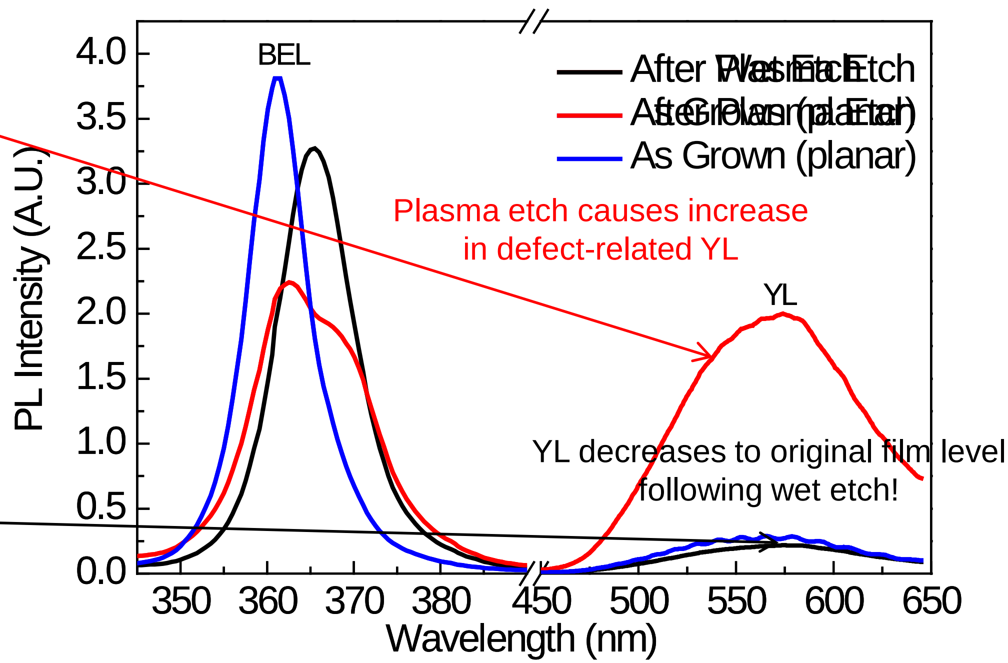
Wet etch step removes plasma etch damage

Plasma etch only



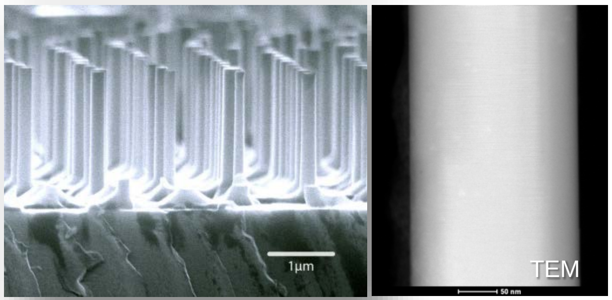
After wet etch





Summary

Precision top-down fabrication *controlled geometries*



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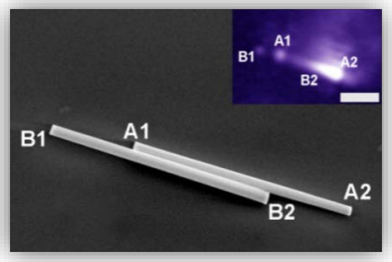

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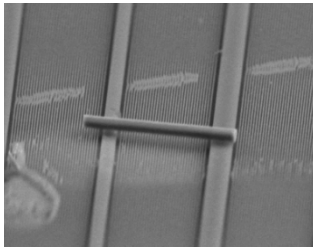
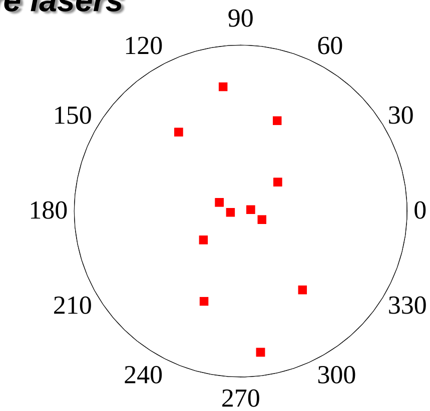
H. Xu et al., *Appl. Phys. Lett.* **101** 221114 (2012)

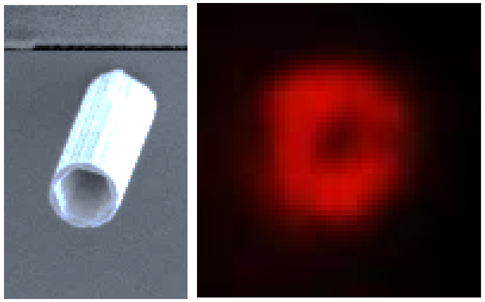


J.B. Wright et al., *Appl. Phys. Lett.*, 104, 041107 (2014).



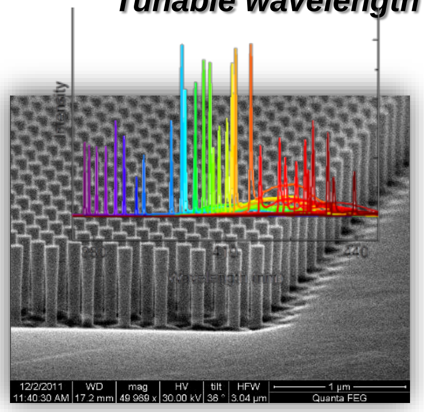
Li, Changyi, et al. *Nanoscale* ***8***, 5682 (2016).

Beam shape control *nanotube lasers*

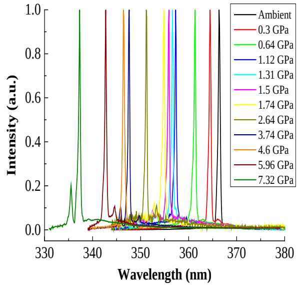


C. Li et al., *ACS Photonics*, **2**, 1025 **(2015)**.

Tunable wavelength nanowire lasers



J.B. Wright et al., *Sci. Reports* 3, Art no. 2982 (2013) doi:10.1038/srep02982



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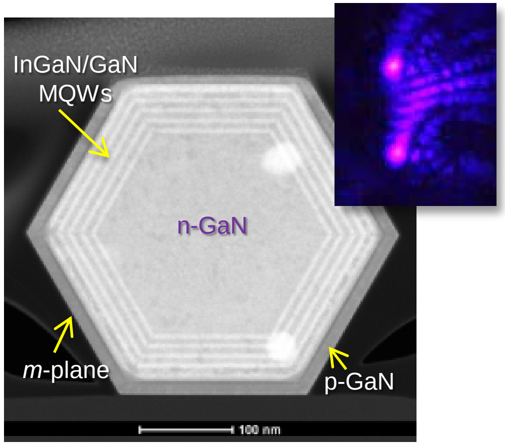
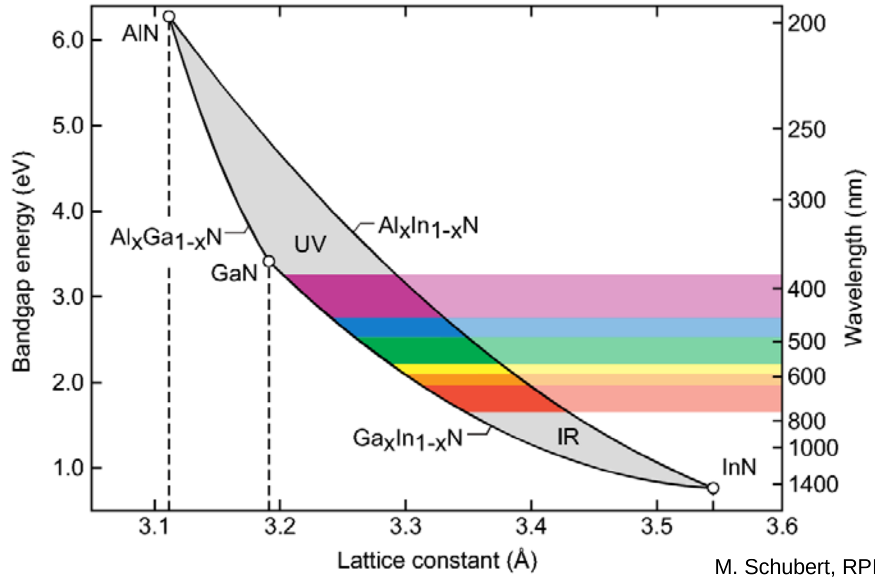


III-Nitride (AlGaInN) semiconductors – The Most Interesting Materials System in the World?

- **Direct RT bandgaps: ~0.7-6.2 ev**
- Solid alloy system (tuneable bandgaps)
- High breakdown field, mobility, thermal conductivity, melting temperature
- Radiation resistant and chemically inert
- InGaN covers entire visible & bulk of solar spectrum (PV material?)



M. Schubert, RPI

- *Commercially used in LEDs, blue laser diodes, high power electronics, HEMTs*

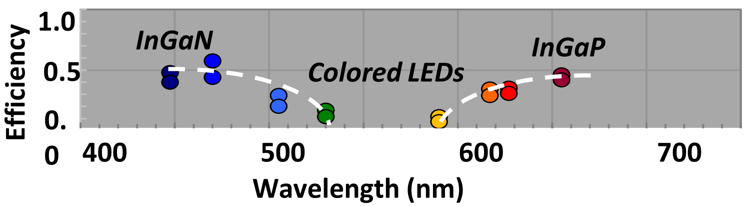
Nitronex GaN power transistor

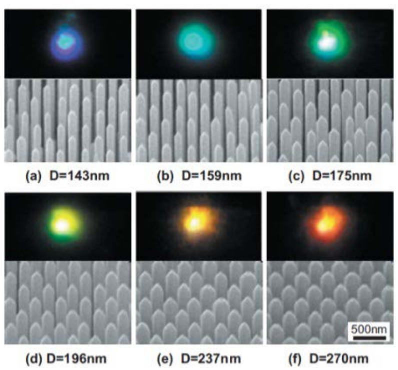
Why III-nitride nanowires for SSL?

Advantages due to enhanced strain accommodation in nanowires

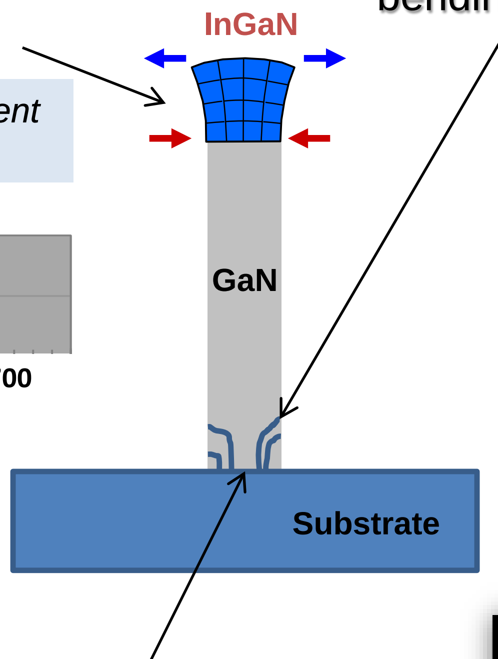
elastic strain relaxation at surface

benefit: heterostructures with high In content (e.g. green-yellow-red gap)





Sekiguchi et al., APL **96**, 231104 (2010) – Sophia U.

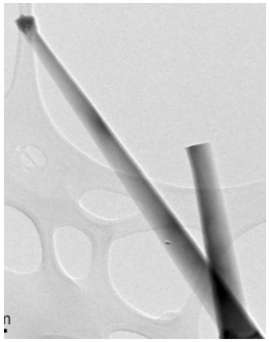


small interfacial area

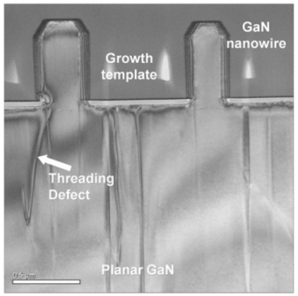
benefit: can grow on cheaper, lattice mismatched substrates; integration with Si devices

bending of dislocations (TDs) toward surface

benefit: reduced TDs, higher IQE

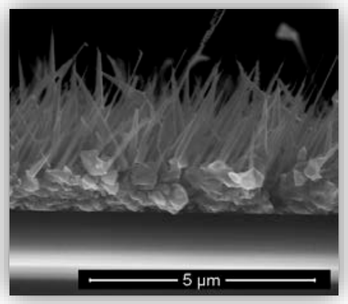


VLS-grown TD-free GaN NWs - Sandia

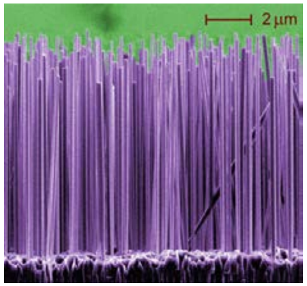


Hersee et al., J. Mat. Res. **17**, 2293 (2011) - UNM

Bending & termination of TDs at nanowire base



GaN NWs on tungsten foil - Sandia

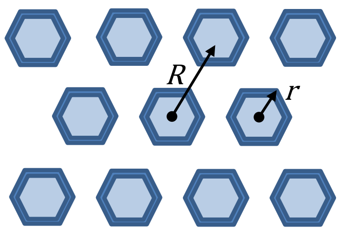


GaN NWs on Si - NIST

Why III-nitride nanowires for SSL?

vertical device integration

benefit: *higher device area per chip (cost/droop)*

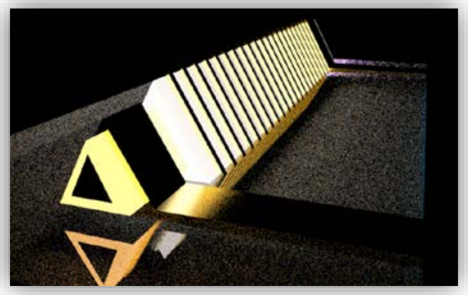


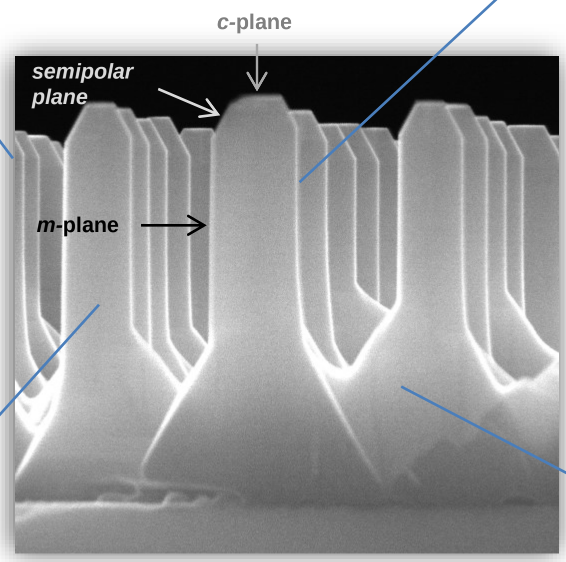
$$\frac{A_{sidewall}}{A_{substrate}} = \frac{6rh}{2.6R^2} \cong \mathbf{4.6F \cdot AR}$$

*example: 4.6·0.5·3=**6.9× increase!***

nanolasers

benefit: *ultracompact, low power coherent light source*

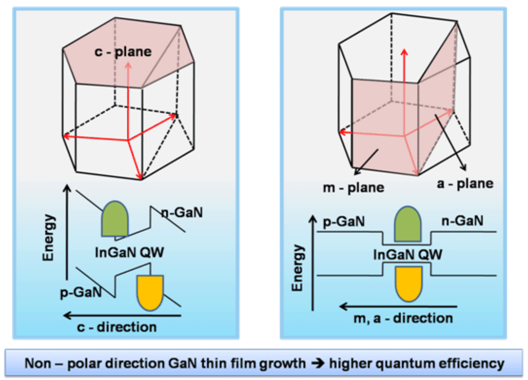




Radial GaN/InGaN MQW NW (Sandia)

access to nonpolar & semipolar planes

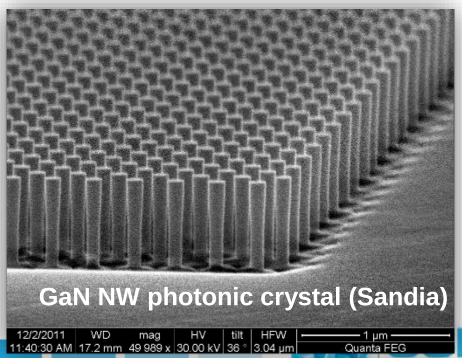
benefit: *higher IQE, reduced wavelength shift*



http://csel.snu.ac.kr/research/LED.php

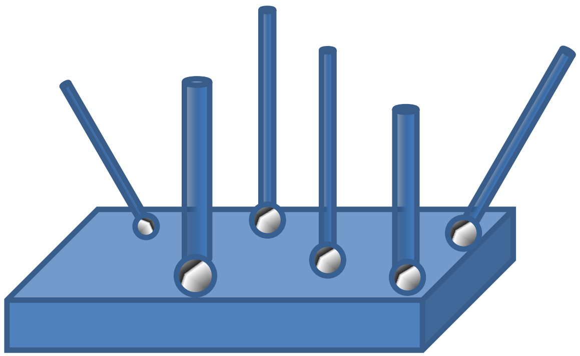
2D arrangements (photonic crystals)

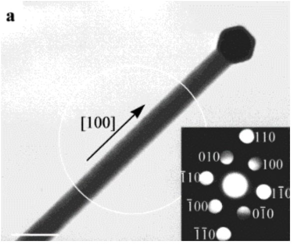
benefit: *higher light extraction, IQE, wavelength tuning*



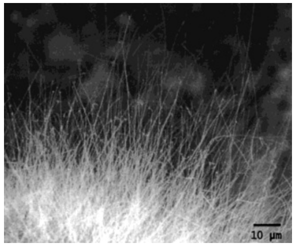
Bottom-up growth of GaN-based nanowires

Metal catalyzed (vapor-liquid-solid)





C. Lieber, 2000

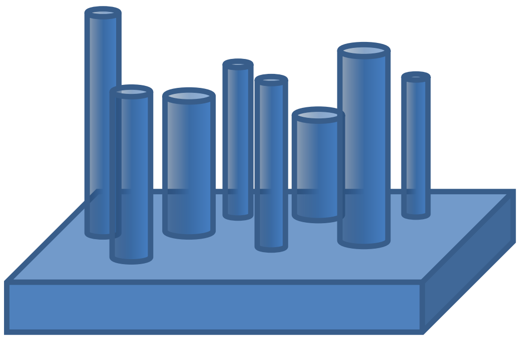


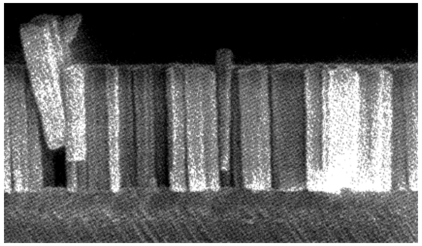
C.C. Chen, 2001

Methods: PLD, thermal evaporation, (MO)CVD

Historically popular (ease & low cost of entry), but falling out of favor due to limitations

"Self-catalyzed"



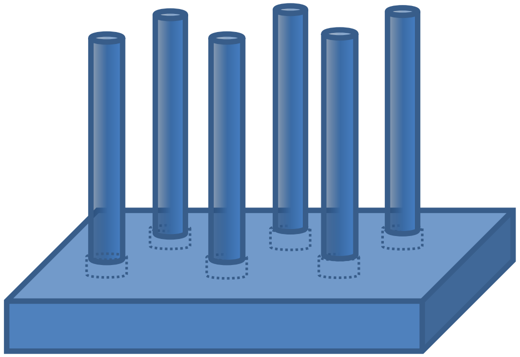


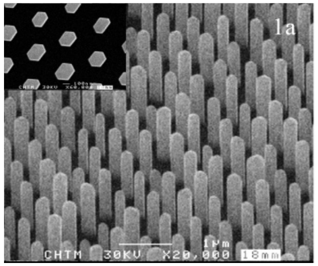
K. Kishino, 1997

Methods: (PA)MBE, HVPE, (MO)CVD

Cons include slow growth rates and random ordering

Selective area growth (SAG)





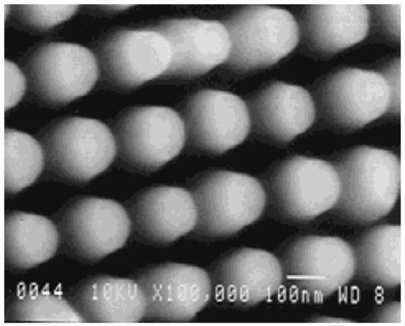
S. Hersee, 2006

Methods: (PA)MBE, MOCVD

Currently popular, but may require growth conditions that limit materials & architectural flexibility

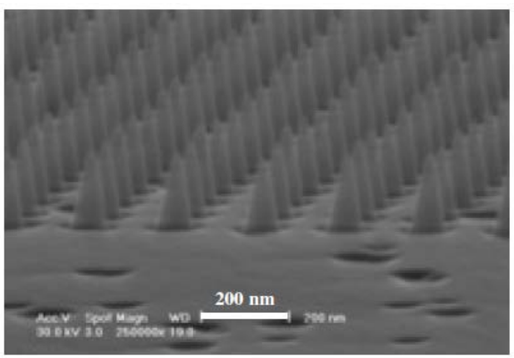
Previous top-down nanorod/nanopillar/nanopost work

InGaN/GaN MQW nanoposts/nanopillars



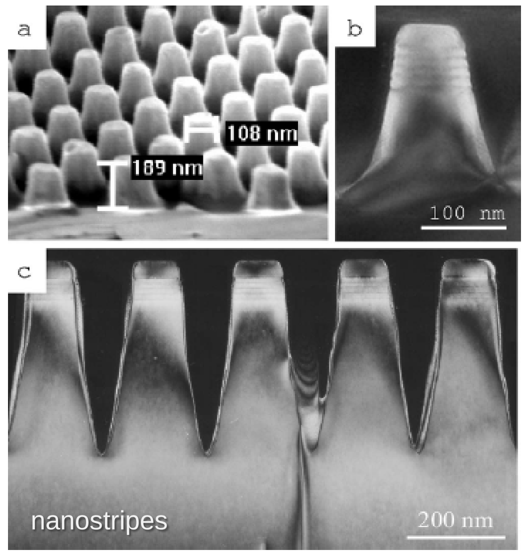
L. Chen et al., PSSa **188** 135 (2001) (Brown, Yale)

- E-beam Ni mask, Cl-based RIE etch



H. S. Chen et al., Nanotechnology **17** 1454 (2006) (NTU)

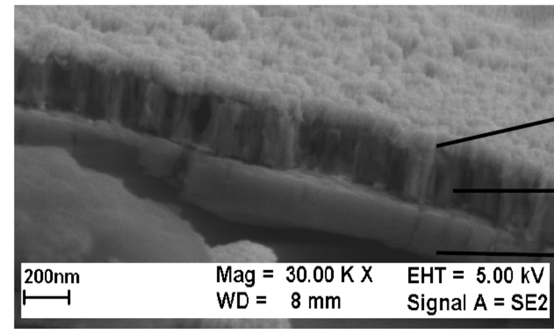
- E-beam Cr mask, Cl-based ICP-RIE etch
- Observed PL blue-shift w/decreasing diameter due to strain relaxation (reduction of QCSE)

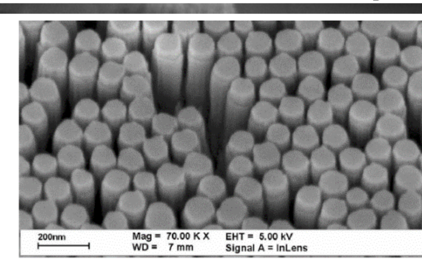


S. Keller et al., J. Appl. Phys. **100** 054314 (2006) (USCB)

Holographic lithography, SiO_2 mask, CHF_3 ICP etch to make strips and pillars

InGaN/GaN MQW nanorod LED





C. Y. Wang et al., Opt. Expr. *16,* 10549, 2008. (NTU)

M. Y. Ke et al., IEEE J. Sel. Top. Quantum Elec. **15** 1242 (2009) (NTU)

SiO_2 nanosphere lithography, Cl-based ICP-RIE etch; electrically injected array

- Tapered, short aspect ratio nanopillars via plasma etching
- Plasma etch causes sidewall damage

Chemical wet etching of the III-Nitrides: Is it useful ?

Wet etching typically not considered viable due to very low etch rates (< Å/min)

Table 1
GaN and AlN etching results in acid and base solutions

Etching solutions	GaN etch rate (nm/min)
Citric acid	0 (75 °C)
Succinic acid	0 (75 °C)
Oxalic acids	0 (75 °C)
Nitric acid	0 (85 °C)
Phosphoric acid	0 (82 °C)
Hydrochloric acid	0 (80 °C)
Hydrofluoric acid	0
Hydroiodic acid	0
Sulfuric acid	0 (82 °C)
Hydrogen peroxide	0
Potassium iodide	0
2% Bromine/methanol	0
n-Methyl-2-pyrrolidonone	0
Sodium hydroxide	0
Potassium hydroxide	0
AZ400K photoresist developer	0
Hydroiodic acid/hydrogen peroxide	0
Hydrochloric acid/hydrogen peroxide	0
Potassium triphosphate	0 (75 °C)
Nitric acid/potassium triphosphate	0 (75 °C)
Hydrochloric acid/potassium triphosphate	0 (75 °C)
Boric acid	0 (75 °C)
Nitric/boric acid	0 (75 °C)
Nitric/boric/hydrogen peroxide	0
$HCl/H_2O_2/HNO_3$	0
Potassium tetra borate	0 (75 °C)
Sodium tetra borate	0 (75 °C)
Sodium tetra borate/hydrogen peroxide	0
Potassium triphosphate	0 (75 °C)
Potassium triphosphate/hydrogen peroxide	0

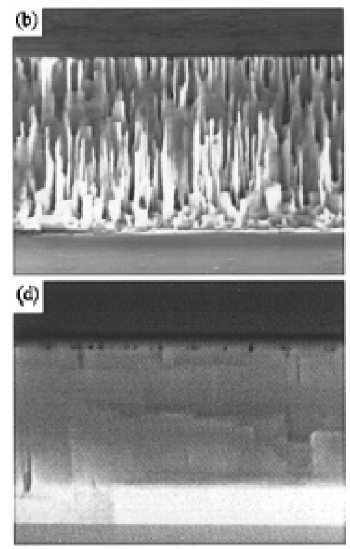
Etching was conducted at room temperature (25 °C) unless otherwise noted (after [31]).

Zhuang and Edgar, MSE R 48 1 (2005)

But actually…

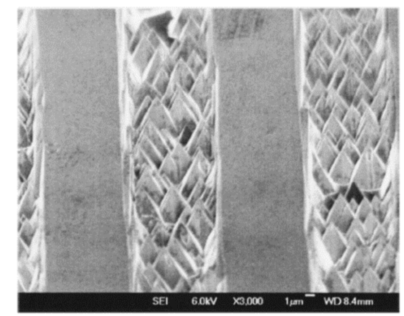
Significant etch rates, but extremely crystallographically anisotropic

m-plane sidewalls



Stocker et al. *APL* **77**, 4253 (2000)

Ga-polar and N-polar GaN



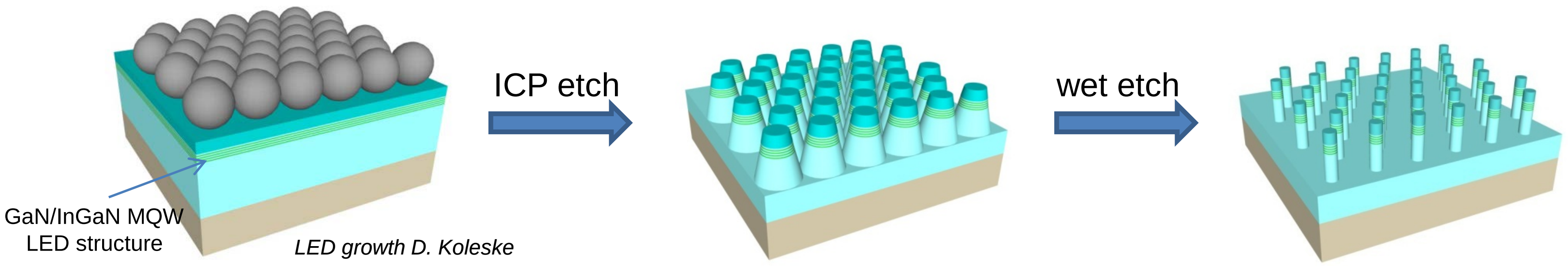
H. Ng et al., J. Appl. Phys., 94, 650 (2003)

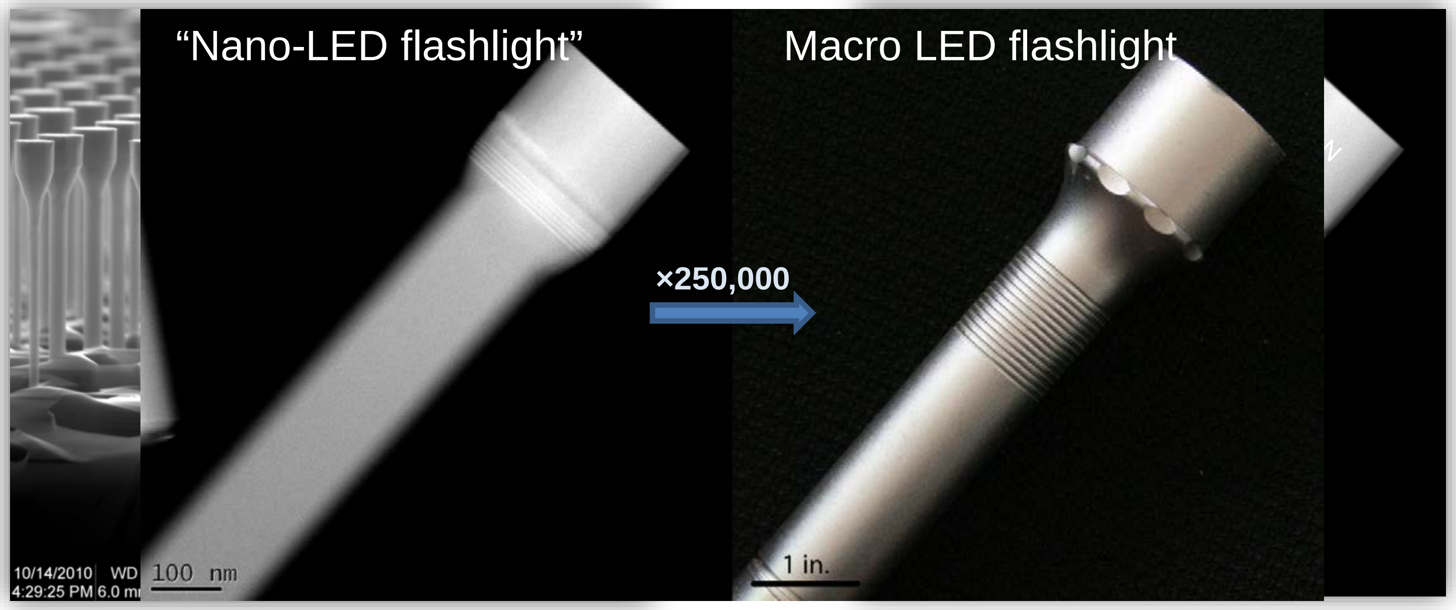
KOH-based wet etches

Anisotropy is **> 2000:1** (GaN/KOH),
as compared to maximum anisotropies of
160:1 (Si), and 4:1 (GaAs)

This property, however, makes wet etching *ideal for micro/nanostructure fabrication* of the III-nitrides !

Etching of Axial Heterostructures - GaN/InGaN nanowire LEDs



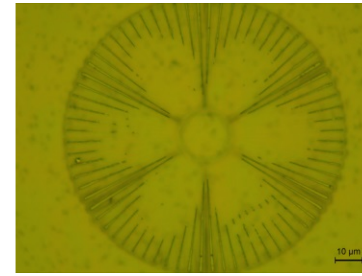


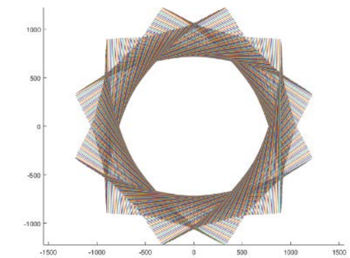
Q. Li et al., *Optics Express* **19**, 25528 (2011)

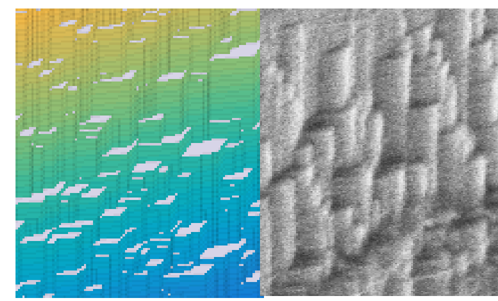
Wet etch affected by doping (composition)

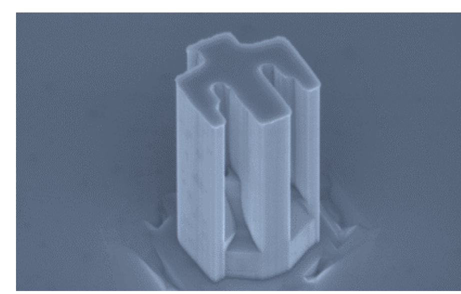
Outline

- Direct measurement of orientation resolved etch rates for GaN
- Prediction of etched geometries
- Elucidate facet evolution during wet etching
- Measurement of facet smoothness
- Application of chemical wet



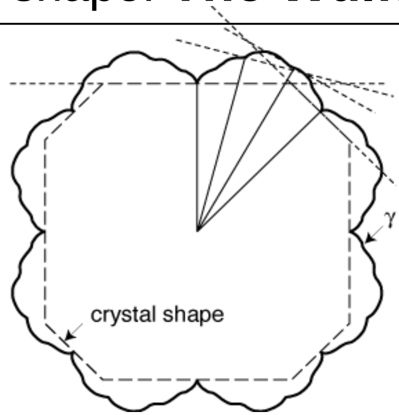






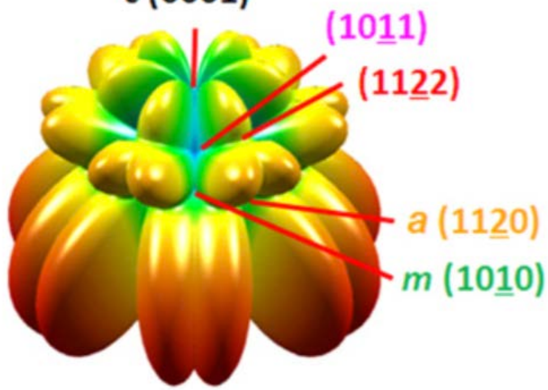
The appearance and evolution of facets

Predicting the equilibrium crystal shape: **The Wulff Plot**



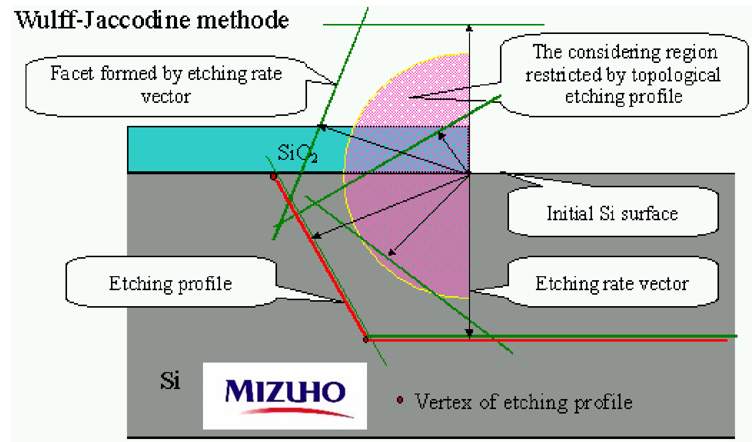
Wulff et al., Semicond. Z. Kristallogr 34, 449 (1901)

For MOVPE growth, use the *kinetic* Wulff plot



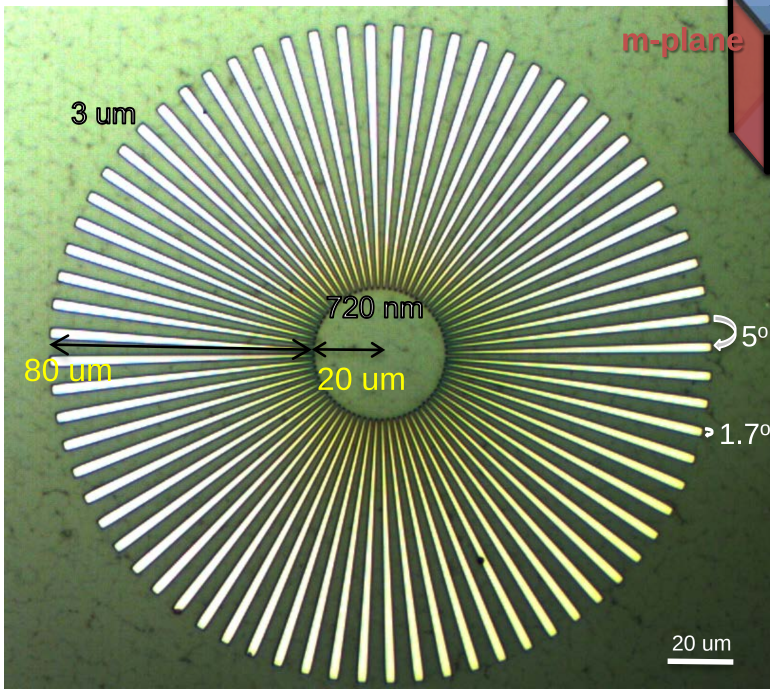
B. Leung et al., Semicond. Sci. Tech. 27, 024005 (2012)

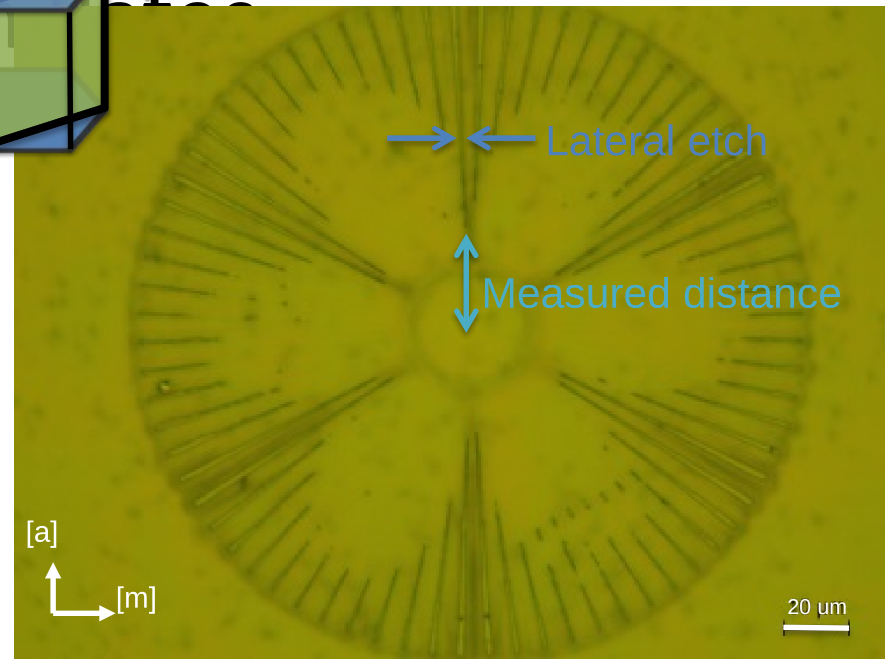
Same method used in commercial silicon wet etch simulators



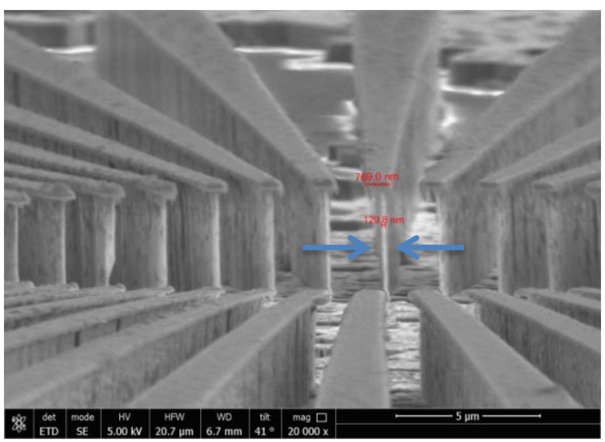
Can we map the "Wulff plot" for etching?

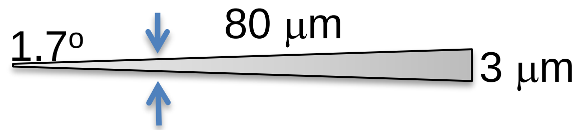
Directly Measuring Orientation Resolved





5% KOH in ethylene glycol





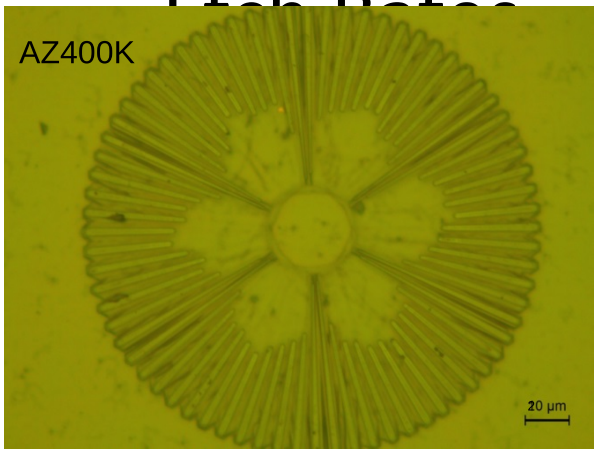
Lateral etch of 100 nm/min →
Wedge retraction of 6.7 µm/min

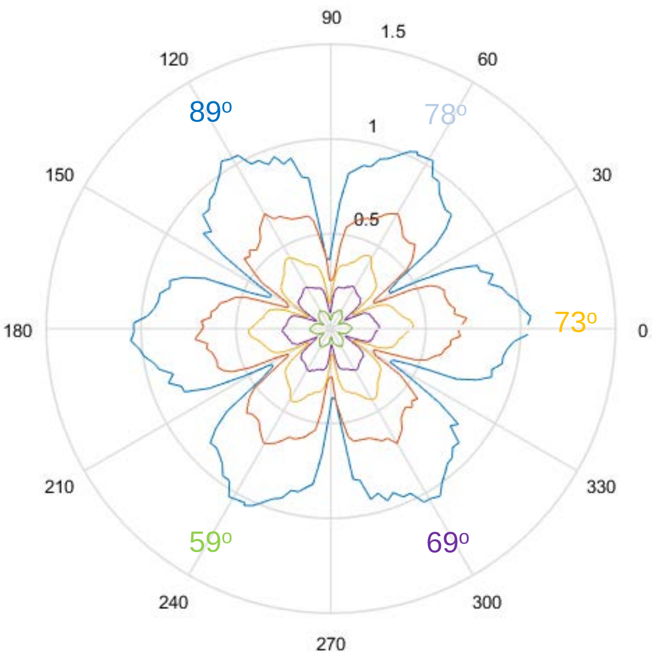
Etch rate is amplified by a factor of 67

Direct measurement of orientation resolved etch rates for the first time in GaN

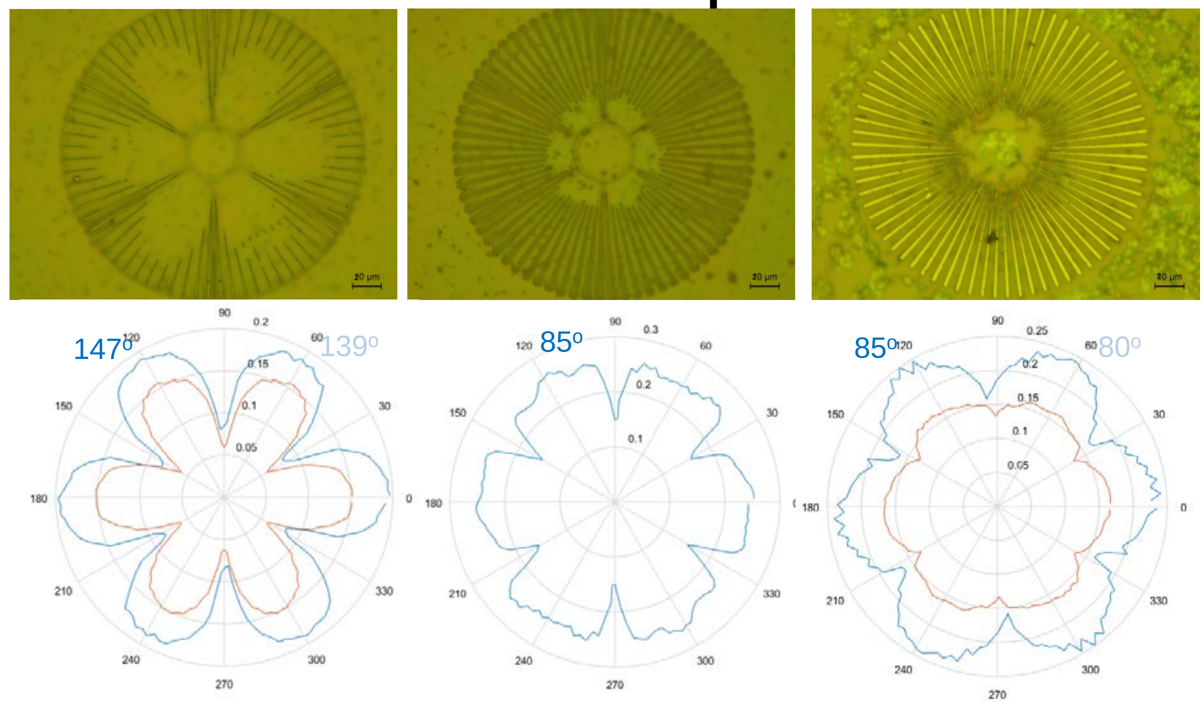
Direct Measurements of Orientation Resolved Etch Rates – Chemistries and Temperatures

AZ400K





5% KOH in ethylene glycol
5% KOH in H_2O
25% TMAH



m-plane: Atomically smooth etch facet

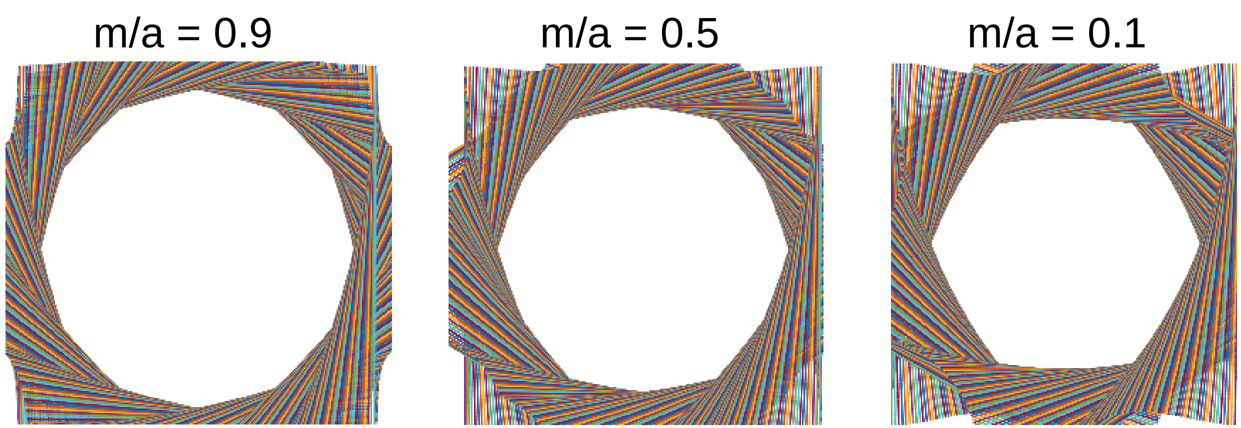
a-plane: Transition from step-flow highly dependent on chemistry

m/a: 0.3 ~ 0.9

Prediction of Etched Geometry: Implementing the Wulff-Jaccodine Method

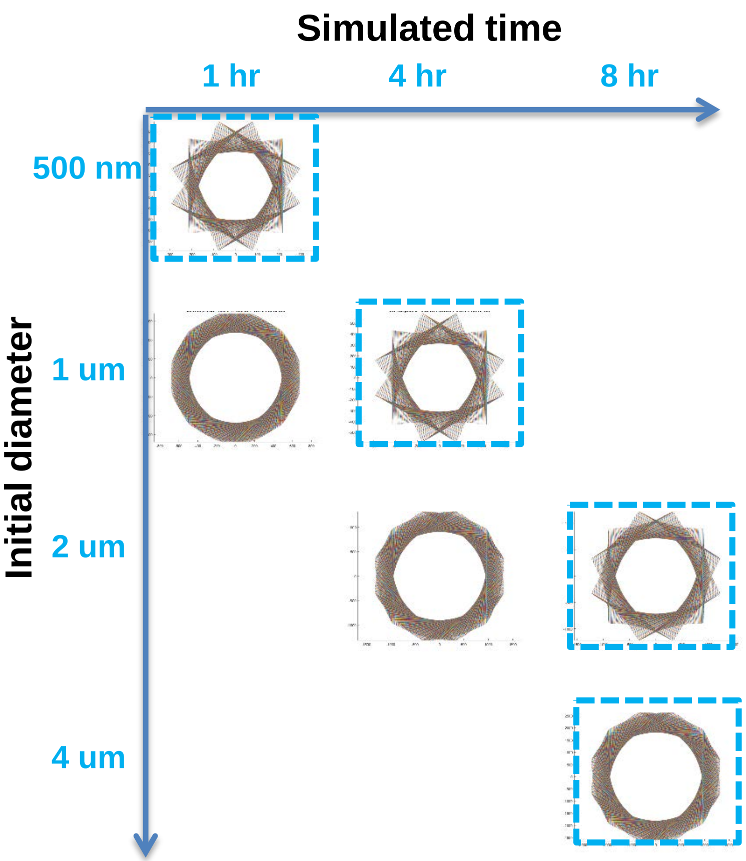
Define initial geometry → *Input orientation dependent etch rates for etch condition*

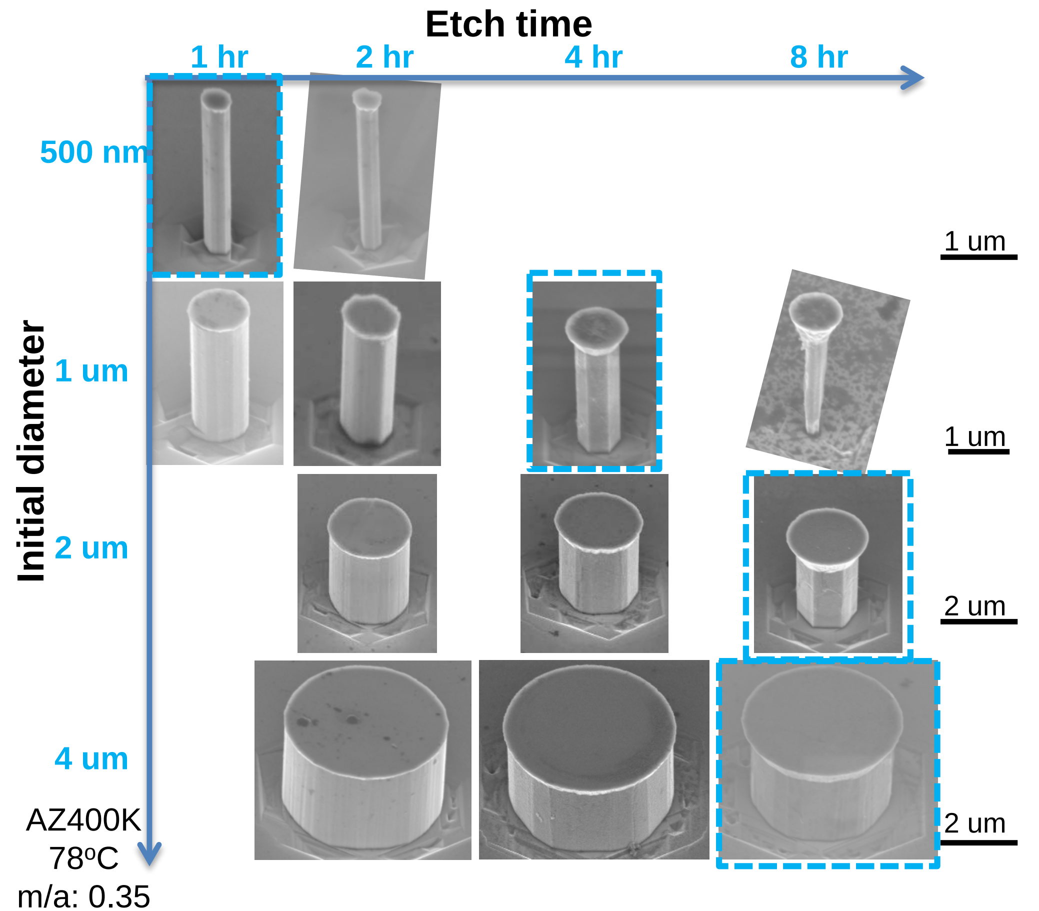
→ *Plot evolution of facets over specified time* → *Final etch geometry predicted by bounding facets*



The white region represents GaN

Prediction vs. Experiment of Etched Geometry – Time and Initial Diameter

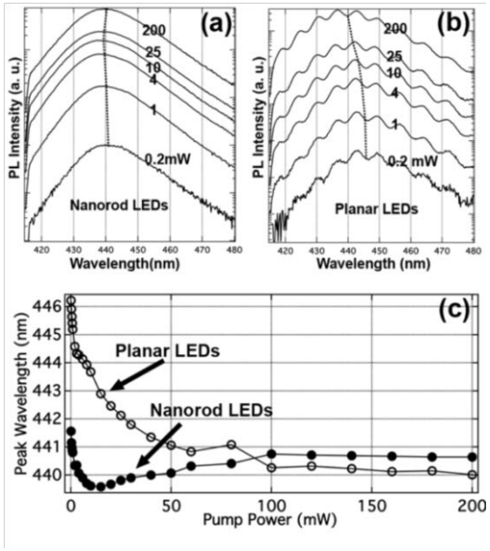




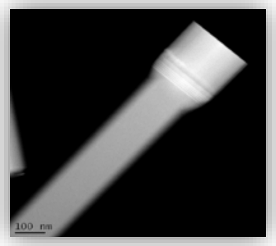
Method provides accurate prediction of pillar etch geometry (transition from circular to hexagonal by appearance of a-plane facets) as a function of initial pillar diameter and time

Optical performance – axial nanowire LEDs vs. planar LED

InGaN peak position vs pump power

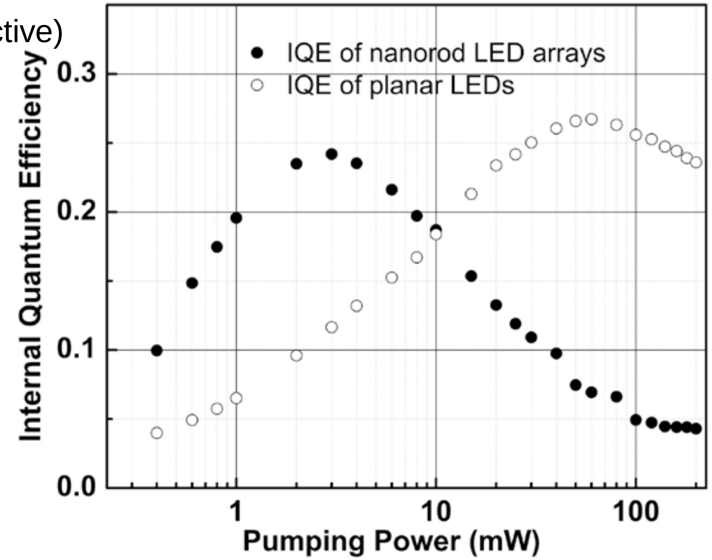


413 nm pump (InGaN selective)



Q. Li et al., Optical performance of top-down fabricated InGaN/GaN nanorod light emitting diode arrays, "*Opt. Express* **19**, 25528 (2011)

IQE – nanowire vs Film



PL, IQE: K. Westlake, M. Crawford

- XRD shows ~16±4% strain reduction in InGaN QWs in nanowire LEDs

XRD measurement courtesy Steve Lee

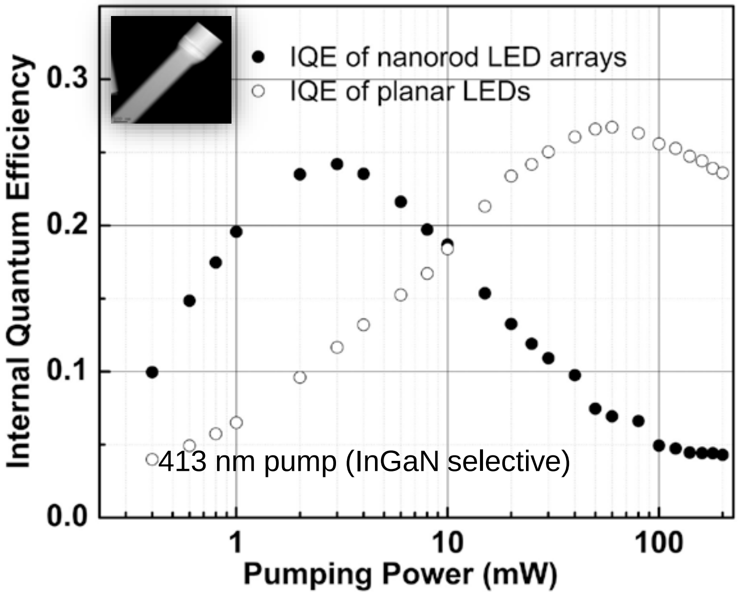
- Little wavelength shift at higher pump powers for nanowire LEDs (no/reduced QCSE)

- nanowire LED: comparable IQE to planar LED but peak IQE occurs at much lower pumping power (enhanced light absorption, heating)

No obvious benefit for LED performance/cost ratio (loss of device area), but possibility for very high IQE single NW emitters for nanophotonic applications

Optical performance – axial nanowire LEDs vs. planar LED

IQE – nanowire vs Film



PL, IQE: K. Westlake, M. Crawford

Q. Li et al., *Optics Express* **19**, 25528 (2011)

- XRD shows ~16±4% strain reduction in InGaN QWs in nanowire LEDs *XRD measurement :Steve Lee, Sandia*
- TEM shows ~94% of nanowire LEDs are dislocation free
- Little wavelength shift at higher pump powers for nanowire LEDs (no/reduced QCSE)
- But nanowire LED shows only comparable IQE to planar LED; peak IQE occurs at much lower pumping power (enhanced light absorption, heating)

How much is surface passivation likely to improve IQE of NW LED?

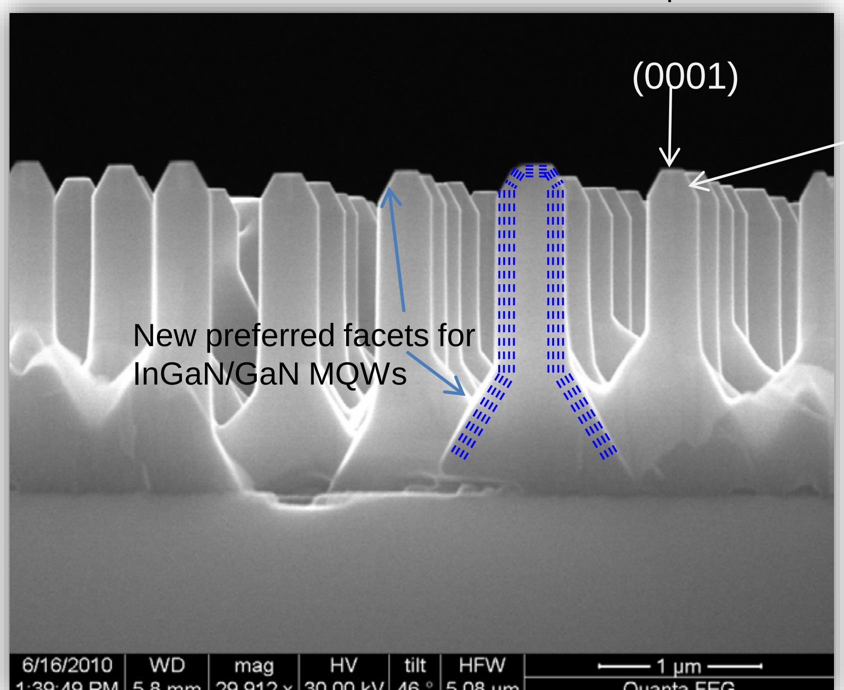
No obvious benefit for blue LED performance/cost ratio (loss of device area), but possibility for very high IQE single NW emitters for nanophotonic applications by starting with commercial quality planar LEDs

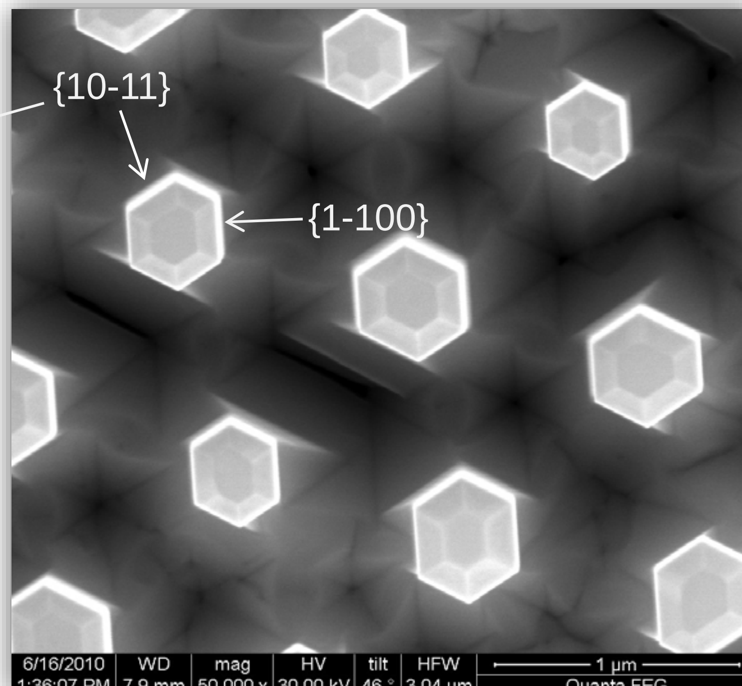
Radial core-shell InGaN/GaN MQWs on top-down NWs

<u>Radial core-shell NW LEDs:</u>

- Much higher active area than axial NW or planar structures
- Reduced strain InGaN growth for higher In incorporation
- Being pursued by a number of groups/companies (MOCVD)

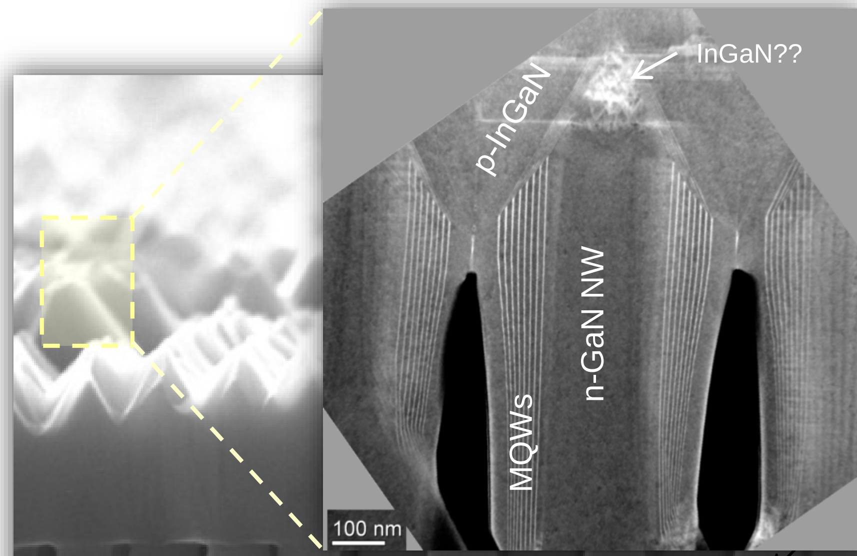
After 5-period MQW GaN/InGaN shell growth

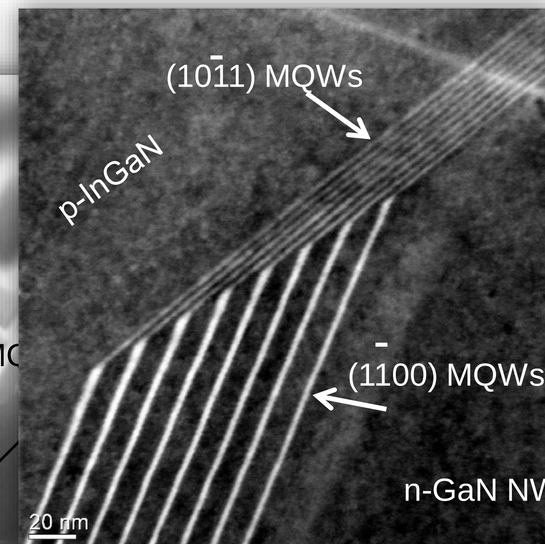




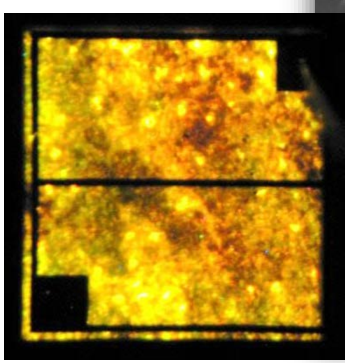
New semipolar facets form with InGaN/GaN MQW growth

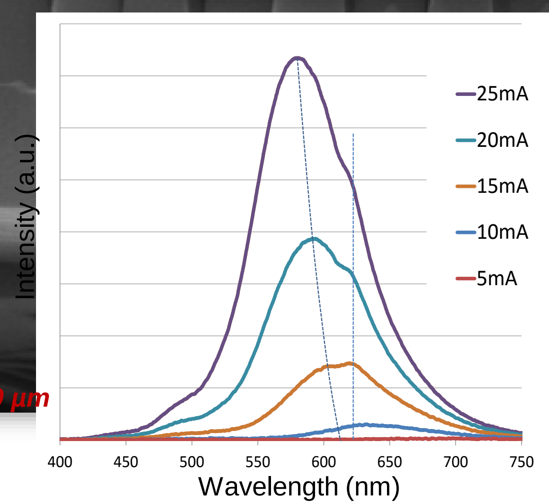
Electrically injected core-shell nanowire based "3D" LED emitting at yellow-red wavelengths





J. Wierer et al., *Nanotechnology* **23** 194007 (2012)





- Yellow-red electroluminescence!
- Two EL peaks:
  - 615 nm (const) red
  - 600-565 nm (shift) yellow

G. T. Wang et al., "Top–down fabrication and characterization of axial and radial III-nitride nanowire LEDs," *Phys. Stat. Solidi A*, **211**, 748 (2014)

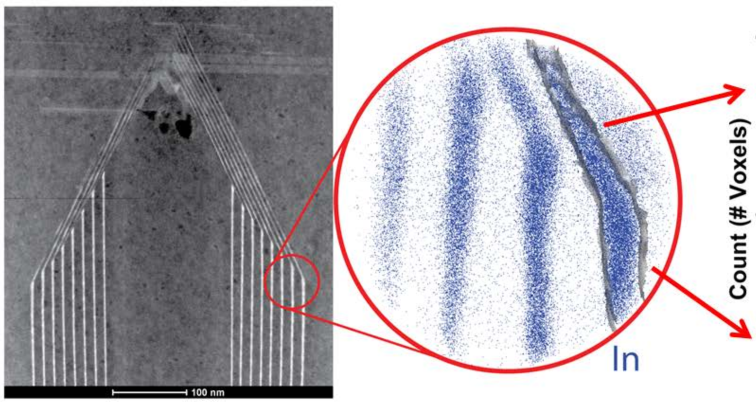
SSLS EFRC

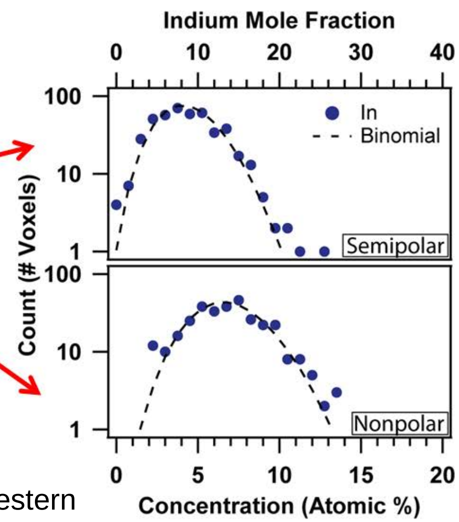
3-D Mapping of Quantum Wells in a GaN-InGaN Core-Shell Nanowire – a Correlated Study

STEM-EDS

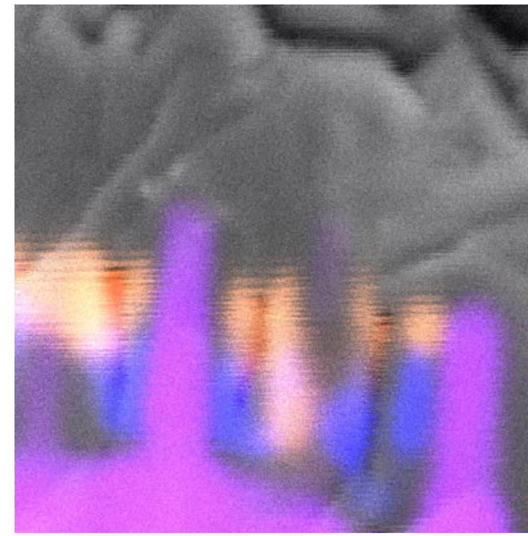
Atom probe tomography (APT)

Cathodoluminescence





APT: L. Lauhon, Northwestern

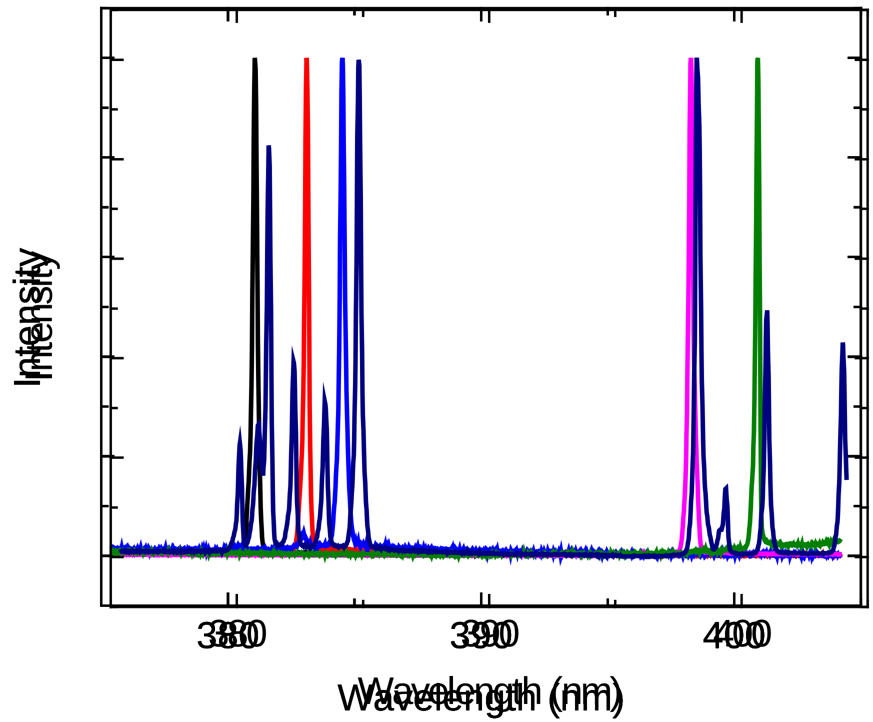


365 nm, 402 nm, 442 nm

Riley, J.; Padalkar, S.; Li, Q.; Lu, P.; Koleske, D. D.; Wierer, J. J.; Wang, G. T.; Lauhon, L. J., *Nano Lett.* **14**, 4317 (2013).

- Nonpolar and semipolar QWs from radial NW LED were imaged by APT and correlated with scanning TEM and cathodoluminescence data
- Results suggest yellow-red emission originates from In-rich tip regions, not sidewalls
- How to get long wavelength emission from m-plane sidewalls?

III-N Nanowire Photonic Crystal Lasers

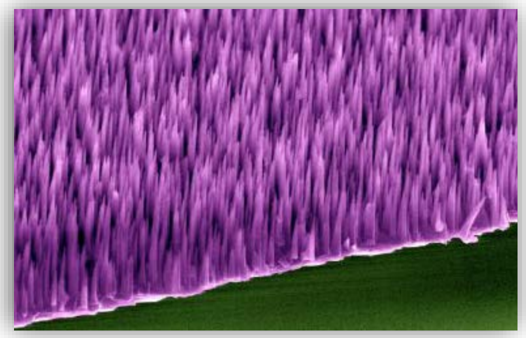


J.B. Wright et al., Scientific Reports 3, Article number: 2982 (2013) doi:10.1038/srep02982

pitch: 320 nm
diameter: 130-140nm

Previous SNL research: bottom-up III-nitride nanowires

Vertically-aligned growth

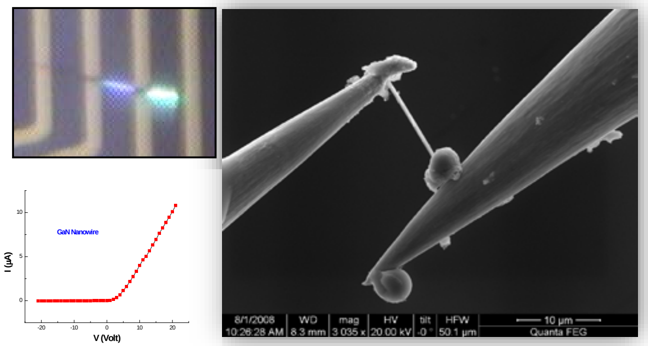


G. T. Wang et al., *Nanotechnology 17 5773-5780* (2006)
Q. Li, G. T. Wang, *Appl. Phys. Lett. 93, 043119* (2008)
Q. Li, J. R. Creighton, G.T. Wang. *J. Crys. Growth 310 3706-3709* (2008)

Nanowire-templated growth

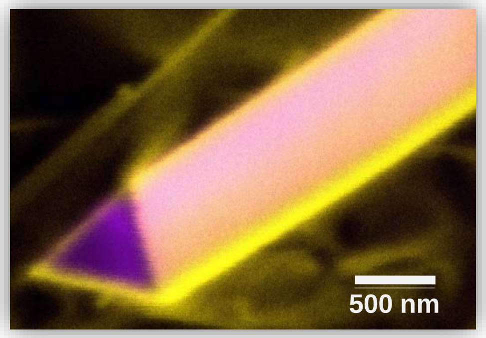
Q. Li, Y. Lin, J.R. Creighton, J. Figiel, G.T. Wang, *Adv. Mat.*, **21** 2416–2420 (2009)

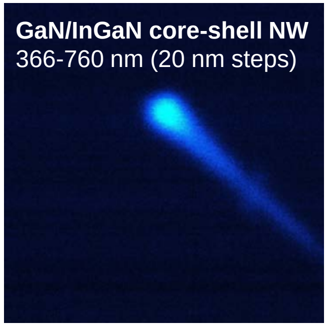
Electrical characterization



A. A. Talin, G. T. Wang, E. Lai, R. J. Anderson, *Appl. Phys. Lett.* **92** 093105 (2008)
Y. Lin, Q. Li, A. Armstrong, and G. T. Wang, *Solid State Commun.*, ***149***, *1608 (2009)*

Optical imaging and spectroscopy





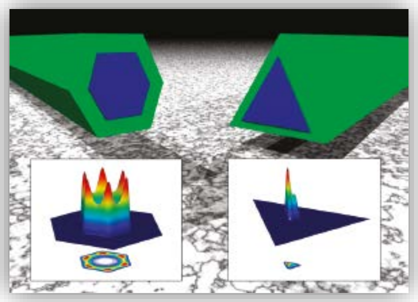
Q. Li, G. T. Wang, *Nano Lett.*, 2010, 10 (5), 1554 [GaN defect CL]
Q. M. Li, G. T. Wang, *"Appl. Phys. Lett.*, ***97***, 181107, 2010. [Gan/InGaN]
P.C. Uppadhya et al. *Semicond. Sci. Tech.* **25** 024017 (2010) [Ultrafast]
A. Armstrong, Q. Li, Y. Lin, A. A. Talin, G. T. Wang, APL **96**, 163106 (2010). [DLOS]

In-situ TEM

T. Westover et al., *Nano Lett.*, **9**, 257 (2009). [*in-situ* NW breakdown]

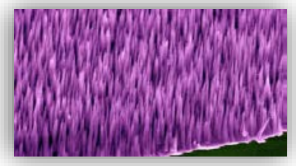
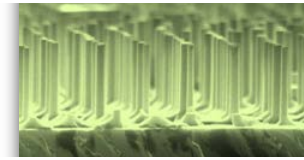
J. Y. Huang et al., *Nano Lett*, ***11 (4),*** *1618* ***(2011).*** [*in-situ* nanomechanics]

Theory

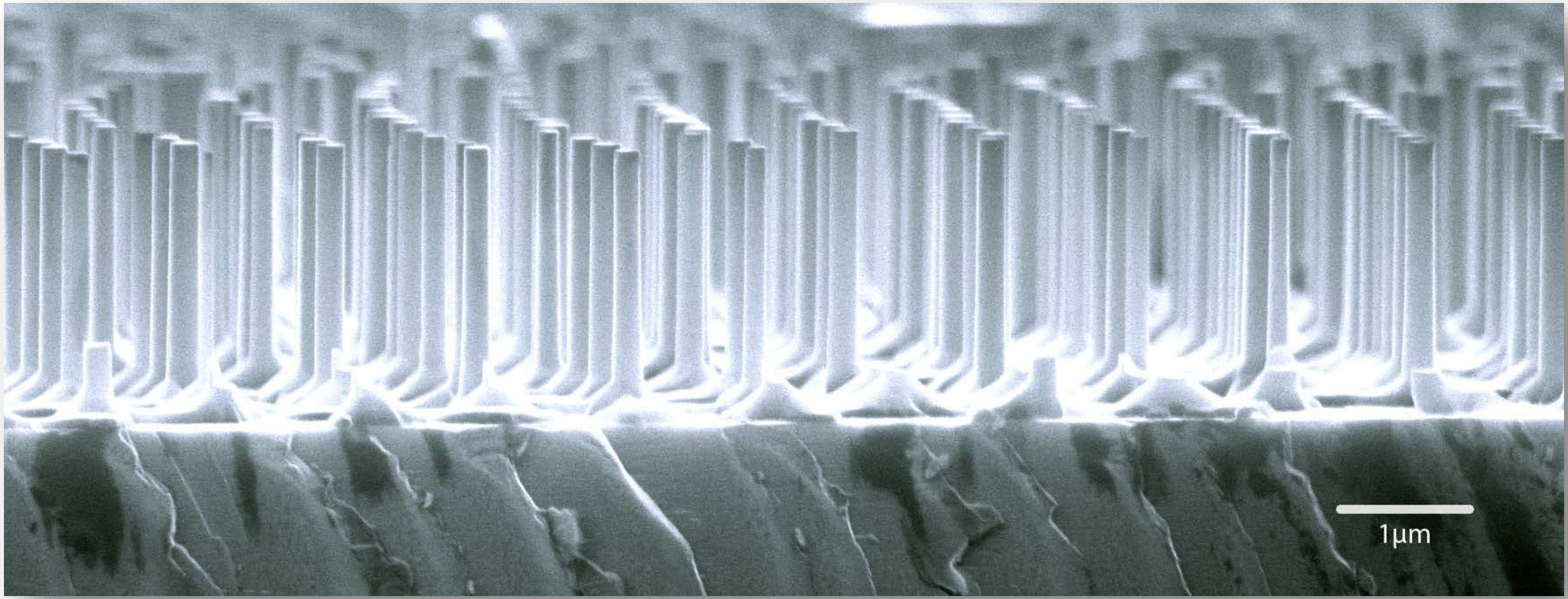


B. Wong et al., *Nano Lett* ***11 (8)***, *3074, 2011*

Bottom-up vs. Top-down nanowires

	Bottom-up 	Top-down (+ regrowth) 
Length	Up to hundreds of µm	Several µm
Diameter	~30 nm to microns	~10 nm to microns
Dislocations	Few or none	Depends on density in film
Radial architectures	Yes, typically by MOCVD	Yes, by MOCVD regrowth
Axial architectures	Yes, typically by MBE	Yes, but not strain relaxed
Substrate	Lattice matching less critical	Lattice matched
Characterization	Some properties difficult to measure (e.g. doping)	Starting material can be measured using standard techniques
Material Quality:	Point defect density may be high due to growth conditions needed	Starting material can be grown under optimal growth conditions
Uniformity	Wire-to-wire variations may occur based on pitch, diameter	Good, although regrowth has same issues as bottom-up

Advantages of new top-down nanowire fabrication method

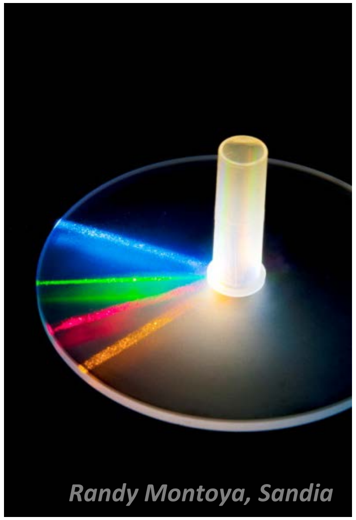


- Wider range of growth conditions, material tunability (1D growth cndns not needed)
- Lower point defect densities (higher growth temperature)
- Ordered/periodic arrays (difficult with catalyst/VLS-based methods)
- Axial III-nitride nanowire heterostructures possible by MOCVD
- Better control of geometry (independent control over height, width, & pitch)
- Improved uniformity
- Easier vertical device integration (height uniformity, base GaN-layer)

Lasers for Solid State Lighting

Advantages of lasers for lighting:

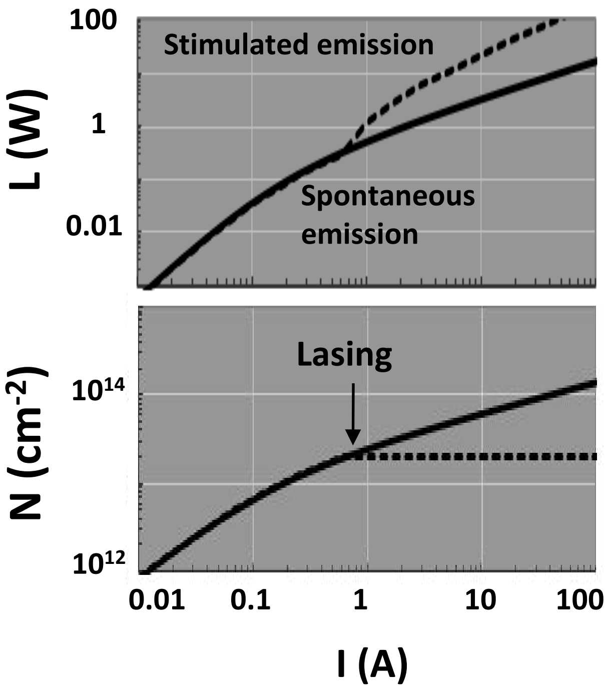
- Lasers show very high efficiency at high power
- LED and LD current densities are converging
- Carrier density is clamped at threshold
  - Circumvent the droop problem in LEDs
  - Need to reduce threshold to avoid losses
- After threshold slope efficiency is one
- Directionality, polarized emission, modulation



Laser Sources For SSL:

- **High efficiency**
- **Low threshold**
- **Focus on III-nitrides**
- **Nanowire lasers**
  - **Low threshold**
- **Polariton lasers**
  - **Ultralow threshold**
  - **New physics**

Clamped carrier density

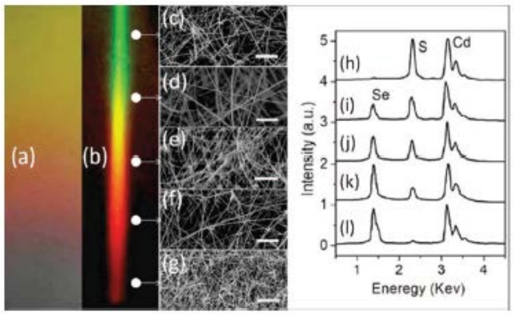


Are narrow linewidth sources acceptable?

A.J. Fischer, Sandia

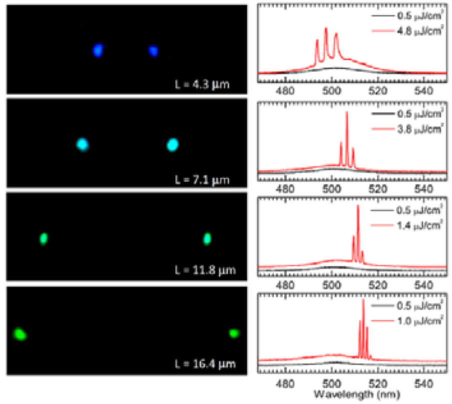
Previous wavelength “tuning” (selection) in nanowire lasers

CdSSe NWs with different compositions across substrate



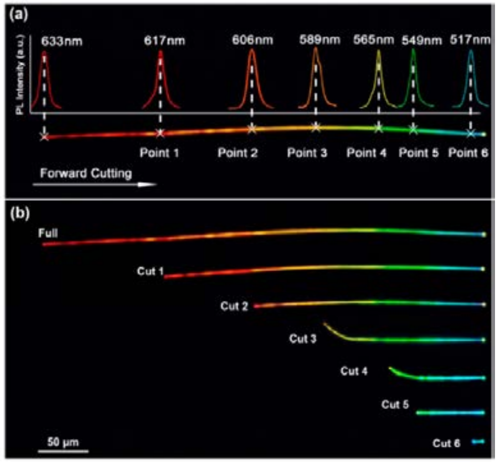
Pan, A. et al., *Nano Letters* **9**, 784 (2009). (ASU)

Different length CdS NWs (intrinsic self absorption effect)



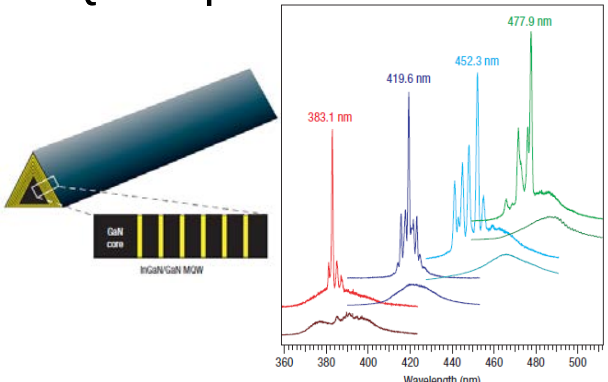
Liu, X et al., *Nano Letters* **13**, 1080 (2013) (Nanyang Tech. U.)

Cutting bandgap graded CdSSe NWs



Yang, Z. et al. *Nano Letters* **14**, 3153 (2014)

Core-shell NWs with different InGaN MQW compositions

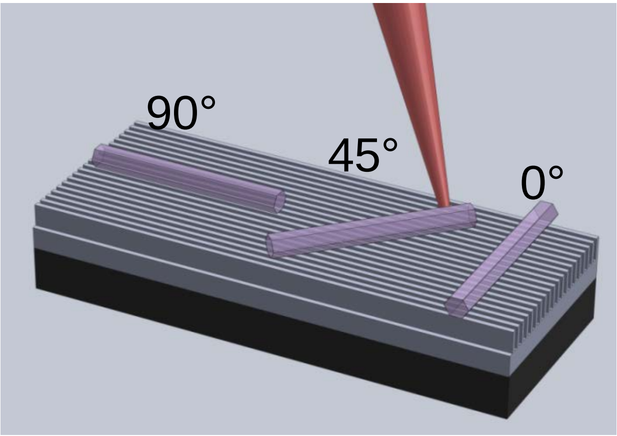


F. Qian et al., *Nat. Mat.*, **7**, 701 - 706 (2008) (Harvard)

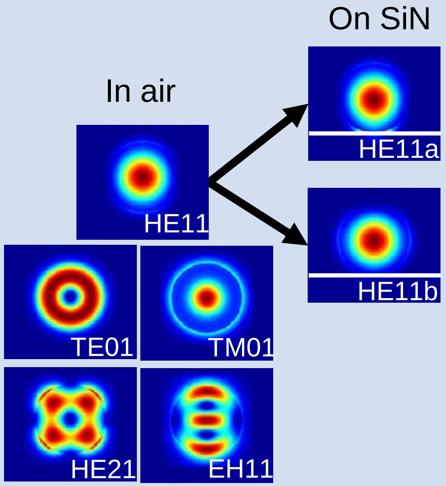
Wavelength selection in nanowire lasers has been demonstrated, but not true continuous and reversible tuning within the same nanowire

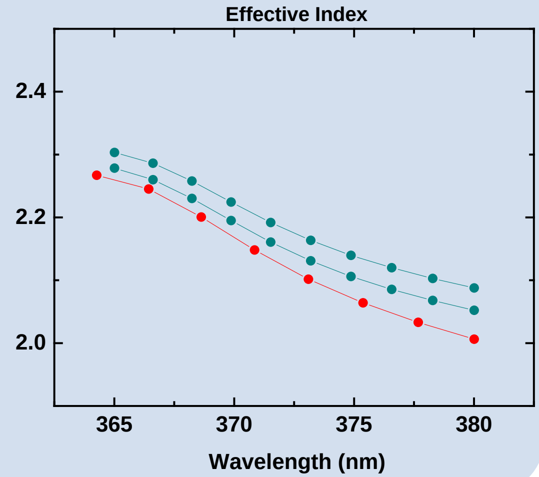
Method 3: Distributed Feedback (DFB) Nanowire Laser

- Standard method for mode control in semiconductor lasers
- Here, single nanowires are coupled to grating substrate to achieve distributed feedback
- Stop-band position and width tuning via nanomanipulation of the angular alignment

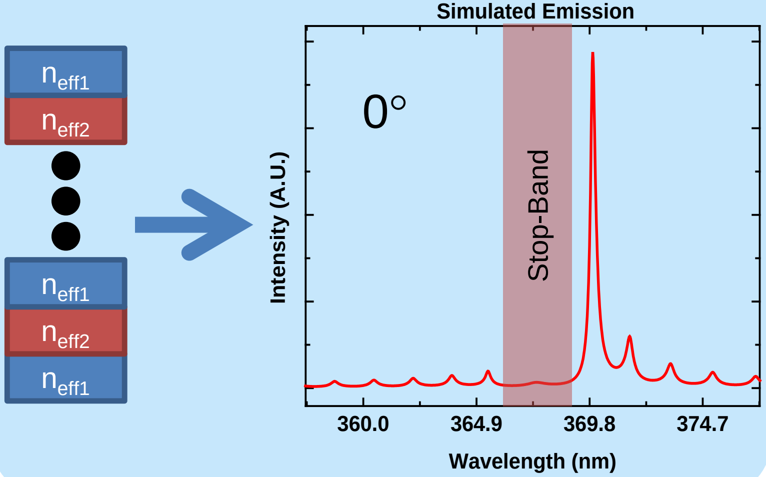


Mode Calculations

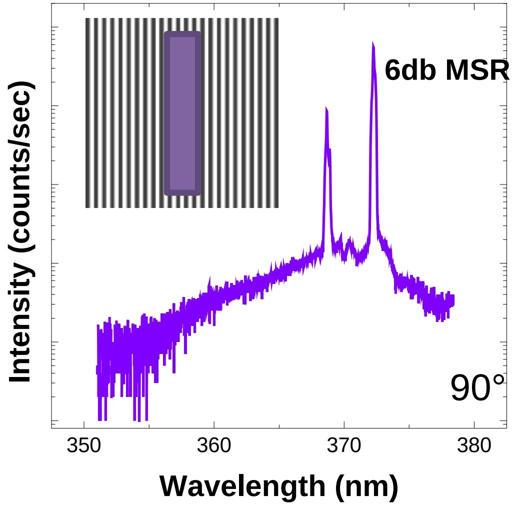


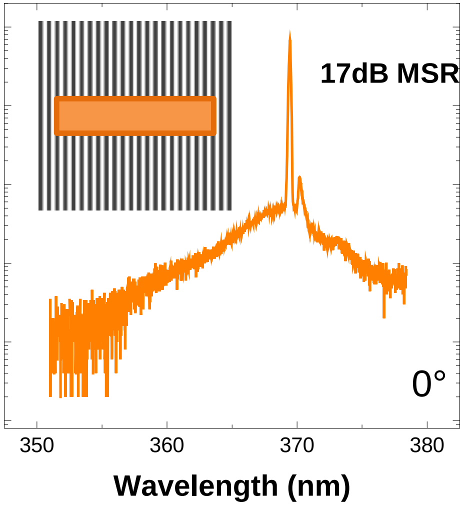


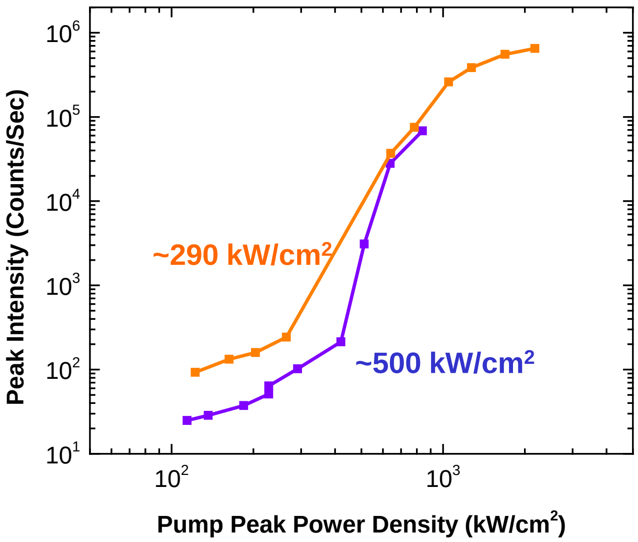
Transfer Matrix Method

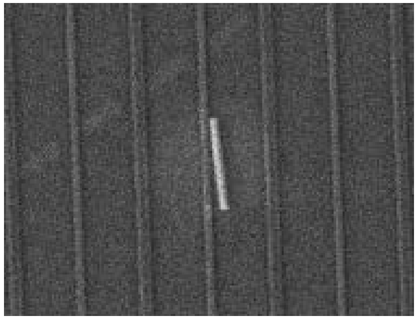


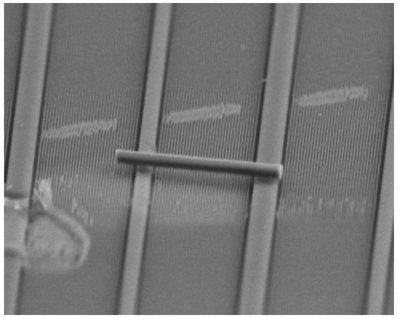
Method 3: DFB Nanowire - Optical Results











NW diameter ~180 nm

- At the designed alignment single-mode lasing was achieved with a 17dB mode suppression ratio.
- Observed reduction in the lasing threshold

J.B. Wright et al. "Distributed feedback gallium nitride nanowire lasers," *Appl. Phys. Lett.*, 104, 041107 (2014).