

### Geographic location of St. Petersburg





### St. Petersburg the capital of Russia since 1703 till 1918



Mariinskii theater Pushkin Hermitage Pavlovsk Russian Museum Peterhof Palaces and Squares of St. Petersburg

## Parks and Palaces of St. Petersburg Suburbs



## Treasures of









1918 — Physical-Technical Department of the State Institute of X-Rays and Radiology was founded by Profs. M. I. Nemenov and A. F. loffe

Nobel Prize Winners which works in the loffe Institute : N. N. Semenov, L. D. Landau, P. L. Kapitsa, I. E. Tamm, Zh. I. Alferov

Soviet Nuclear Programme Leaders : I. V. Kurchatov, A. P. Aleksandrov, Ya. B. Zel'dovich

Total staff - 2000 researchers - 1100

Several hundred of former loffe researchers













<b>MBE grow</b>	th &	processing	Total	24	Optical &	& Electrica	al Studies
	8	Senior Re	esearchers	and	Researchers	5	
	3		PhD stu	dents	S	2	
	3	unde	ergraduate	e stud	lents	1	
	1	mar	nager, tecl	hnicia	an	1	



### **Group Activity Scope**

Main goal: Molecular beam epitaxy and fundamental studies of semiconductor heterostructures (with quantum wells, quantum dots and superlattices) based on

- narrow gap III-V compounds (AI,Ga,In)(As,Sb) for mid-IR optoelectronics and HEMTs (MBE setup Riber 32P, France);
- wide gap II-VI compounds (Zn,Cd,Mg)(S,Se,Te) and ZnO for visible (blue-green) and UV spectral range optoelectronics, including lasers with electron beam and optical pumping, as well as spintronic studies of diluted magnetic semiconductor heterostructures (double chamber III-V/II-VI MBE setup, Semiteq, Russia);
- hybrid III-V/II-VI structures with a heterovalent interface in the active region for mid-IR applications, solar cells, and spintronics (double chamber III-V/II-VI MBE setup, Semiteq, Russia);
- III-nitrides (Ga,In,AI)N for optoelectronic applications in visible (green-red) and deep UV spectral ranges as well as fundamental studies of In-riched compounds and metalsemiconductor composite nanostructures (PA MBE setup Riber Compact 21T, France).

### Projects

Russian Foundation for Basic Research (basic & applied research) – 12 (1 with PKU) Presidium of RAS, Physical Sciences Department of RAS – 5 Russian Agency for Science and Innovations – 2 International contracts (OSRAM, Germany; ETRI, Korea; KACST, Saudi Arabia) - 3 Marie Curie training network on Spinoptronics (FP7) – 1 Russian Ministry on Industry and Trade, Ministry of Deffence - 2 *Peking University, May 15, 2014* 



Plasma-assisted molecular beam epitaxy of AI(Ga)N layers and quantum well structures on  $c-AI_2O_3$  for mid-UV emitters and solar-blind photodiodes

### S.V. Ivanov



#### **Acknowledgments**

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### Outline

Applications of UV-optoelectronics and state-of-the-art of AlGaN-based

UV LED and laser structures obtained by both MOVPE and PA MBE

PA MBE growth and surface morphology control of III-Nitrides

- > Al-rich growth of thick AlN/c-Al<sub>2</sub>O<sub>3</sub> buffer layers
- Growth kinetics of AlGaN layers (2D-3D transition)



### Market and main applications of UV-optoelectronics





### Main applications of UV-lasers



**Biomedical applications** 

#### Peking University, May 15, 2014

14



### State of art of UV-LEDs (2013)





### State-of-the-art of UV-lasers (2013)





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### Peculiarities & Advantages of Plasma-Assisted MBE of AlGaN



High vacuum growth conditions

No parasitic gas-phase reactions (AI+NH<sub>3</sub>)

- Hydrogen- and carbon-free growth environment Easy Mg doping without post-growth treatment
- Rapid change of growth atom fluxes
- Low growth temperature (700-800°C)
  - + Sharp interfaces with atomic resolution
  - Complexity of the 2D step-flow growth
- Growth under different stoichiometric conditions:

nitrogen-rich (III/N<1) **3D growth** metal-rich (III/N≥1) **2D growth** 

• Variable polarity growth:

Ga-polar and N-polar depending on growth nucleation



### Linear control of plasma-activated nitrogen flux



N mace flow (a) c



Jmerik et al., Techn. Phys. Lett. 33,333 (2007)



### Stoichiometrical conditions in PA MBE of III-nitrides



### Ga-rich PA MBE of GaN



Heying et al., J. Appl. Phys. 88, (2000), 4. Northrup et al., Phys.Rev.B, 61, (2000), 9932.



Pan et al., Chin.Phys.Lett., 28, (2011), 068102.



## Al-rich growth of thick AIN layers with periodically supplied Al-flux and continuous N-flux









### Polarities of Al<sub>x</sub>Ga<sub>1-x</sub>N (x=0-1) layers grown by PA MBE



Mizerov et al., Semiconductors, 43, 1096 (2009)



# AIGaN layers with different morphologies and Al-content grown at both N- and Ga-rich conditions



### N-rich AlxGa1-xN

 $F_{III}/F_N=0.9$  $T_S=700^{\circ}C$  $F_N=0.5ML/s$  $F_{AI}/F_N=0.7\neq x$ 

x depends on

- Substrate temperature
- Stress in heterostructures
- Growth rate

Mizerov et al., J. Cryst. Growth 323, 686 (2011).



 $\frac{\text{Ga-rich Al}_{0.7}\text{Ga}_{0.3}\text{N}}{F_{III}/F_{N}=1.8}$  $T_{S}=700^{\circ}\text{C}$  $F_{N}=v_{g}=0.5\text{ML/s}$  $F_{AI}/F_{N}=x=0.7$ 





## 3D-2D phase diagram of PA MBE growth of $AI_xGa_{1-x}N$ (x=0-0.8) layers in Ga-rich conditions





### Metal-rich growth of thick 2D-AIGaN layers with temperature and flux modulated epitaxy





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### Growth of AIN nucleation layers on c-AI2O3 substrates at the different substrate temperatures & stoichimetric conditions





## Lowering the TDs density in AIN/c-AI<sub>2</sub>O<sub>3</sub> heterostructure by changing stoichiometric conditions 3D 2D





### TDs density in 390-nm-thick AIN/c-AI203 heterolayers with different nucleation layers (NL)

### XRD rocking curves



# General TEM view of AIGaN/AIN/c-AI<sub>2</sub>O<sub>3</sub> SQW structure with NL grown in regime III







# Filtering TD densities in thick AIN buffer layers by multiple compressively-strained 3nm-thick GaN layers



# Filtering TDs in thick AIN buffer layers by multiple compressively-strained 3nm-thick GaN layers





### Microscope images of the 2-µm-thick GaN/AIN heterostructures with GaN interlayers of non-optimized design and growth conditions



detachment



Strain relaxation versus surface morphology of 3-nm-thick GaN interlayers in 2D AIN buffer



### Structural property of Al<sub>0.8</sub>Ga<sub>0.2</sub>N(100nm)/AIN(2µm)/c-Al<sub>2</sub>O<sub>3</sub> heterostructure grown on optimized AIN/GaN buffer





- Metal-flux modulated Al-rich growth of 260nm-thick AIN layers separated by six 3-nmthick GaN interlayers grown under the slightly N-rich conditions (F<sub>Ga</sub>/F<sub>N</sub>~0.9).
- Screw&mixed TD density reduced to ~10<sup>8</sup> cm<sup>-2</sup>

### XRD rocking curves of AIN buffer layer in SQW structure grown on c-sapphire at optimized AIN/GaN buffer structure design and growth conditions

symmetric (0002) reflex

skew-symmetric (10-15tw) reflex



Screw TD : ~1.5 10<sup>8</sup>cm<sup>-2</sup>

Edge & Mixed TD : ~3 10<sup>9</sup>cm<sup>-2</sup>

$$N_{\text{TD}} = w^2/4.35b^2$$
 AIN:  
b = 0.498 nm (for screw TD)  
b = 0.311 nm (for edge TD)



### Suppression of TDs by using $30x\{AI_{0.77}Ga_{0.23}N/AIN\}/AIN$ superlattices (T = 10nm, $x_{av} = 0.9$ )





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## Submonolayer Digital Alloying (SMDA) growth of $AI_xGa_{1-x}N/AI_yGa_{1-y}N$ MQW heterostructures









*Jmerik et al., Semiconductors* **42**, 1452 2008), *PSS A* **210**, 2013).



### HAADF STEM and HRTEM images of AIGaN-based SQW heterostructure







### Structural properties of AlGaN-based SQW heterostructure grown on c-Al<sub>2</sub>O<sub>3</sub>







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### Valence band structure of $AI_xGa_{1-x}N(x=0-1)$ alloys and anisotropic polarization of output UV radiation from GaN&AIN



### PL spectra of AIGaN-based heterostructures with the different compressive strains on c-Al<sub>2</sub>O<sub>3</sub>



46



# Kinetic limitations for generation of threading dislocations

Misfit dislocations generation	$\frac{dN}{dt}$	- <u>to</u>	be influenced by the:						
Stress due to misfit strain	eff								
Density of nucleation sites	$N_{0}$		2D	Metal-rich PAMBE !					
Growth temperature	Т		Low T (<800 ) PA MBE !						
This can be described by the semiempirical expression :									
$\frac{dN}{dt}  B  N_0$	n  0	exp	$\frac{E_a}{kT}$						
			Houghto	n, J.Appl.Phys. 70, (1991), 2136					



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### Optically pumped mid-UV lasing in AIGaN heterostructures grown by PA MBE (previous results)



APPLIED PHYSICS LETTERS 96, 141112 (2010)

Low-threshold 303 nm lasing in AlGaN-based multiple-guantum well structures with an asymmetric waveguide grown by plasma-assisted molecular beam epitaxy on c-sapphire

V. N. Jmerik, <sup>1,a)</sup> A. M. Mizerov, <sup>1</sup> A. A. Sitnikova, <sup>1</sup> P. S. Kop'ev, <sup>1</sup> S. V. Ivanov, <sup>1</sup> E. V. Lutsenko, <sup>2</sup> N. P. Tarasuk, <sup>2</sup> N. V. Rzheutskii, <sup>2</sup> and G. P. Yablonskii<sup>2</sup> <sup>1</sup>Ioffe Physical-Technical I<sup>-</sup>



### Design and TEM image of AIGaN-based SQW structure optimized for sub-300nm UV-lasing





#### Key factors

- Reduction of TDs density (10<sup>8</sup>-10<sup>9</sup>cm<sup>-2</sup>) by using (1-3)-µm thick AIN buffer with incorporated optimized 3-nm-thick GaN layers and AlGaN/AIN SL, grown under the optimized conditions.
- Employing a SQW structure with optimum QW thickness <3 nm and slightly inhomogenious morphology facilitating appear nce of localization states.







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### Electrical properties of as-grown PAMBE Al<sub>x</sub>Ga<sub>1-x</sub>N:Mg

**C-V (300K)**: p-type  $p = 3 \times 10^{18} \text{ cm}^{-3}$ 

Hall (250K): p-type p =  $1 \times 10^{18}$ cm<sup>-3</sup>,  $\mu_H$  = 3 cm<sup>2</sup>/Vs Seebeck (300K): p-type

## $AI_{0.42}Ga_{0.58}N$

C-V (300K): compensated, although at the reverse bias above 5V p-type conductivity was observed

Hall (250K): p-type p =  $4 \times 10^{17}$  cm<sup>-3</sup>  $\mu_{H} = 1.4$  cm<sup>2</sup>/Vs

Seebeck (300K): p-type

This is the first observation of p-conductivity by Hall effect in MBE-grown Al<sub>x</sub>Ga<sub>1-x</sub>N with x>0.3

Komissarova et al., Montreux, 2008, pss(c) 6, S466 (2009)



### **Design and characteristics of UV LED**



### Development of solar-blind p-i-n UV-photodiode







### Manufacturing both Schottky and p-i-n UV-photodiodes with the different level of Mg-doping





### Conclusions

- Advanced PA MBE growth technology of several-micron-thick AIN buffer layers with atomically-smooth and dropletfree surface morphology on c-Al<sub>2</sub>O<sub>3</sub> substrates has been developed, which employs pulsed supplied AI-flux under the quantitative control by laser reflectometry
- 2D-3D phase diagram of PA MBE of  $AI_xGa_{1-x}N$  epilayers (x=0.2-1) on c- $AI_2O_3$  substrates under the group III-rich conditions has been elucidated and substrate-temperature modulated epitaxy has been proposed to avoid Ga droplet formation.
- The most optimum combination of GaN interlayer parameters to maintain the 2D and cracking-free morphology of GaN/AIN buffer layers is as follows: mixed 2D-3D growth mode (streaky-dotty RHEED pattern), thickness of about 3nm, and interlayer spacing above 250 nm. TEM analysis has exhibited positive effect of the GaN insertions providing (i) inclination of both screw and edge TDs and (ii) blocking of vertical propagation of TDs due to bending in c-plane.
- Threading dislocations reduction and filtering using HT-MEE nucleation layer, optimized ultra-thin GaN interlayers in AIN buffer layer and strained AIN/AIGaN SL resulted in reduction of TD density in the active region grown atop down to 1.5 10<sup>8</sup> and 3 10<sup>9</sup> cm<sup>-2</sup> for screw and edge types, respectively. Lowest reported values for PA MBE.
- AIGaN SQW and MQW structures have been fabricated by SMDA technique, which demonstrate RT PL within the wavelength range of 230-320 nm, PL decay time around 1 ns up to RT and intensity reduction just by 2.5 times from 77 to 300K.
- Pseudomorphic growth of AlxGa1-xN/AIN (x>0.5) heterostructures has been implemented on c-Al<sub>2</sub>O<sub>3</sub> substrates to suppress transition from TE to TM polarization of photoluminescence as the wavelength decreases.
- Advanced AlGaN SQW and MQW structures grown by PA MBE on c-sapphire demonstrate optically-pumped stimulated emission within the 255-300 nm wavelength range with typical threshold power densities of 240-480 kW/cm<sup>2</sup> (295K) and TE polarization. The lowest laser threshold power density achieved at 289 nm was 150 kW/cm<sup>2</sup>.
  - Mid-UV LEDs and solar-blind photodiodes have been demonstrated.



## Thank you for the attention



## Main effects determining the light emission mechanisms in AlGaN-based (0001) QWs



Varied technological parameters:

- Al-composition in Barrier layers: y=0.4-0.6 QWs: x=0.3-0.5
- Thickness of QW: 2.5-6nm
- Morphology: plane vs corrugated
- Growth temperature: 670-730°C



# 6-nm-thick $AI_{0.3}Ga_{0.7}N/AI_{0.4}Ga_{0.6}N$ SQW structures with plane and corrugated morphology grown by SMDA



2D AIN buffer growth mode 3D AIN buffer growth mode

For both structures  $N_{\rm D}$ ~10<sup>10</sup> cm<sup>-2</sup>



### CL images and cw-PL spectra of the AIGaN SQW structures with different morphologies

