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| 13. ABSTRACT (Maximum 200 words)<br>AlGaIn alloys with high Al contents, covering from 350 nm to 200 nm, are ideal materials for the development of efficient ultraviolet (UV) light sources/sensors. There are many problems and questions that still stand in the way of the practical device implementation of UV photonic devices. Among these, the attainment of highly conductive Al-rich AlGaIn remains one of the biggest obstacles for the III-nitride research. The objectives of this program were to address some of the fundamental material and device issues and to explore potential applications of III-nitrides for UV micro- and nano-photonic devices. The KSU team has achieved<br>1. n-type Al-rich AlGaIn alloys with record high conductivities.<br>2. converted highly insulating AlN to n-type conductive AlN by Si doping.<br>3. nano-fabrication and characterization of III-nitride photonic crystals (PC) and demonstrated the first current-injected III-nitride PC emitter operating below 330 nm.<br>4. p-type conduction in Al-rich Al <sub>x</sub> Ga <sub>1-x</sub> N for x up to 0.7.<br>5. nano-fabrication of deep UV photonic crystals on AlN wafers.<br>6. achieved 280 nm UV LEDs that are among the best in the world.<br>7. demonstrated the operation of 200 nm DUV Schottky detectors based on AlN having a detectivity that is comparable to those of photomultiplier tubes. |                                                                    |                                                                   |                                                                            |                                                                    |
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## Final Report

Award No: DAAD19-03-1-0337  
Project Title: Wide Bandgap III-Nitride Micro- and Nano-Photonics  
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### I. Summary of Progress

III-nitride wide bandgap semiconductors, with energy band gap varying from 0.9 eV (InN) to 3.4 eV (GaN) to about 6.2 eV (AlN), have been recognized as technologically important materials. Photonic devices based on III-nitrides offer benefits including UV/blue emission; large band offsets of GaN/AlN or InN/AlN heterostructures allowing novel quantum well (QW) device design, and inherently high emission efficiencies. Furthermore, due to their mechanical hardness and larger band gaps, III-nitride based devices may operate at much higher temperatures and voltages/power levels for any dimensional configuration and in harsher environments than other semiconductor devices and are expected to provide much lower temperature sensitivities, which are crucial advantages for many applications. AlGaIn alloys with high Al contents, covering from 350 nm to 200 nm, cannot be replaced by any other semiconductor system due to the fact that no other semiconductor possesses such a large direct bandgap (diamond is 5.4 eV with indirect bandgap), as well as the ability of bandgap engineering through the use of alloying and heterostructure design. Efficient ultraviolet (UV) light sources/sensors are crucial in many fields of research. For instance, protein fluorescence is generally excited by UV light; monitoring changes of intrinsic fluorescence in a protein can provide important information on its structural changes.

However, there are many problems and questions that still stand in the way of the practical device implementation of UV photonic devices. Among these, the attainment of highly conductive p-type AlGaIn, especially in high Al content AlGaIn alloys, remains one of the biggest obstacles for the III-nitride research. Methods for improved material qualities, which would enhance the doping efficiencies and device performance, need to be further explored. The objectives of this ARO research program is to address fundamental material and device issues that are expected to profoundly influence our understanding of the fundamental properties of III-nitrides and their potential applications in new areas beyond UV/visible emitters as well as semiconductor electronics.

### Accomplished Milestones

During the supporting period, the KSU team has achieved

- n-type  $\text{Al}_x\text{Ga}_{1-x}\text{N}$  alloys (of high Al contents) with record high conductivities.
- n-type AlN epilayers with measurable conductivity (resistivity  $\rho = 10^2$  ohm-cm).
- a better understanding for the band structure and the associated optical properties of  $\text{Al}_x\text{Ga}_{1-x}\text{N}$  alloys and AlN.
- nano-fabrication and characterization of III-nitride photonic crystals and demonstrated the first current-injected III-nitride photonic crystal (PC) emitter operating at  $\lambda$  as short as 330 nm.

- a better understanding of roles of vacancies in Al-rich AlGa<sub>x</sub>N and methods for their identification.
- p-type conduction in Al-rich Al<sub>x</sub>Ga<sub>1-x</sub>N for x up to 0.7.
- nano-fabrication of deep UV photonic crystals on AlN wafers.
- achieved 280 nm UV LEDs that are among the best in the world.
- demonstrated the operation of 200 nm DUV Schottky detectors based on AlN having a detectivity that is comparable to those of photomultiplier tubes.

We highlight a few examples of our studies below:

➤ Material growth by MOCVD, device fabrication and characterization

Over the last several years, our group has developed comprehensive techniques for the MOCVD growth of III-nitride materials. By optimizing the epilayer crystalline quality, carrier mobility and optical emission properties, we have produced GaN, Al<sub>x</sub>Ga<sub>1-x</sub>N (x up to 1), In<sub>x</sub>Ga<sub>1-x</sub>N, and In<sub>x</sub>Al<sub>y</sub>Ga<sub>1-x-y</sub>N epilayers, InGa<sub>x</sub>N/GaN, GaN/AlGa<sub>x</sub>N, AlGa<sub>x</sub>N/AlN and In<sub>x</sub>Al<sub>y</sub>Ga<sub>1-x-y</sub>N/GaN QWs with device qualities. The output powers of our light emitting diodes (LEDs) with a standard chip size (0.3 x 0.3 mm<sup>2</sup>) are about 10 mW and 0.4 mW at 20 mA for 408 nm/460 nm and 280 nm devices, respectively. Our 200 nm deep ultraviolet (DUV) Schottky detectors based on AlN have a detectivity that is comparable to those of photomultiplier tubes.

➤ Integration of micro-photonic components

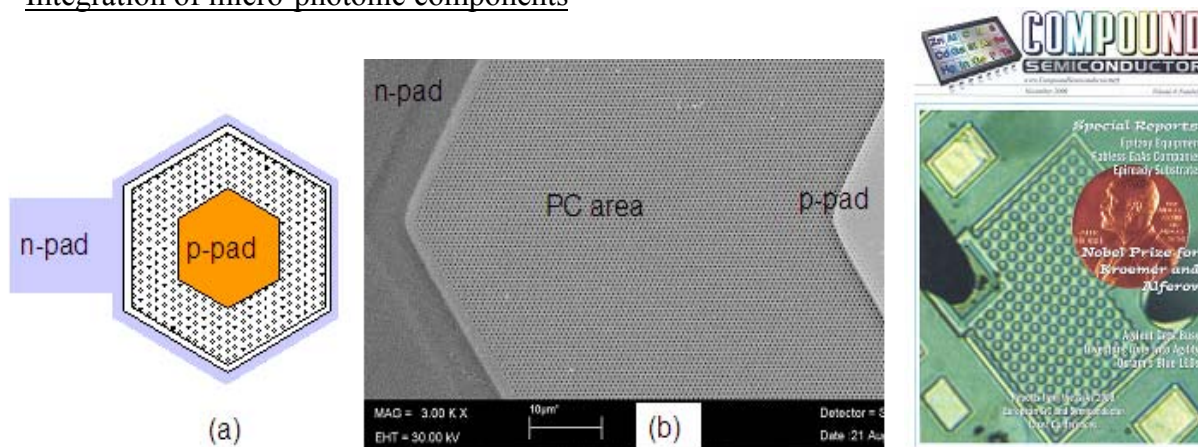


Fig.. 1 (Left) Schematic of photonic crystal (PC) incorporation on III-nitride emitters and scanning electron microscopy (SEM) image of PCs created by nanofabrication. ***Our work on nitride PCs was also reported by the January 2004 issue of Photonic Spectra, February 2004 issue of SPIE's oemagazine, March 2004 issue of Compound Semiconductors, and March 2004 issue of III-V Reviews.*** (Right) An example of our interconnected  $\mu$ -LED for boosting LED extraction efficiency (on a cover page of the *Compound Semiconductor*).

We have pioneered the fabrication of micro- and nano-photonic structures based on III-nitride wide bandgap semiconductors by e-beam and photo-lithography patterning and plasma etching as well as by selective-area epitaxial overgrowth. We have patented micro-size LED technology and applied the interconnected micro-size LED technology for boosting the nitride emitter efficiencies (Fig 1 - Right) and individually addressed micro-LED arrays for microdisplay applications.

Our group was the first to successfully achieve nanofabrication of photonic crystals (PCs) based upon III-nitrides and exploited PCs to enhance the extraction efficiency of UV emitters [Fig. 1 - Left]. We achieved a 20-fold enhancement of light extraction using optical pumping. For UV PC-LEDs under current injection, a 3-fold enhancement in light extraction and a 4-fold enhancement in modulation speed have been realized by PC formation. Our research has established nanofabrication processes for nitride materials, provided a cornerstone for achieving significant enhancement in quantum efficiency of UV emitters through chip-scale integration/architecture and expanded photonic bandgap materials into the DUV range.

Research on III-nitride PCs is now being actively pursued by many research groups worldwide and PC-LEDs are now being recognized as one of the most prominent technologies to boost the LED efficiency for solid-state-lighting technology, which is expected to revolutionize the lighting paradigm with the benefits of high energy efficiency, longevity, and high reliability.

➤ High crystalline quality AlN epilayer growth technology development

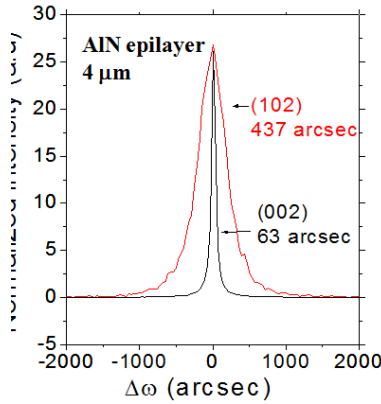


Fig. 2 XRD rocking curves of (002) and (102) planes for a 4  $\mu\text{m}$  AlN epilayer.

We have carried out the growth and systematic studies of the optoelectronic and structural properties of AlN epilayers through the measurements of x-ray diffraction (XRD), photoluminescence (PL) and the dark current of the fabricated AlN DUV photodetectors. The results revealed that the threading dislocation (TD) density, in particular the edge TD density, decreases considerably with increasing the epilayer thickness. XRD rocking curves of the (002) and (102) reflections of a 4  $\mu\text{m}$  epilayer are shown in Fig. 2 and FWHMs are as small as 63 and 437 arcsec, respectively. These are among the smallest values reported for AlN epilayers and are even smaller than those of the best GaN epilayers grown on sapphire (GaN (002) reflection peak has a FWHM of about 150 arcsec).

From the tilt (out-of plane rotation) and twist (in-plane rotation) spread caused by the mosaicity of the AlN film, the dislocation density was estimated. The screw dislocation density was  $\sim 5 \times 10^6 \text{ cm}^{-2}$  in the 4  $\mu\text{m}$  thick AlN epilayer and is more than one order of magnitude lower than that in GaN of the same thickness ( $\sim 10^8 \text{ cm}^{-2}$ ). This clearly indicates that AlN epilayer is an effective dislocation filter. This reduction in screw dislocation density is particularly important for vertical optoelectronic devices such as LEDs, laser diodes (LDs), and Schottky detectors because screw dislocations are one of the major sources of current leakage paths, which increase with increasing current density. Screw dislocations also behave as non-radiative recombination centers that reduce the output intensity from optical devices. We have also established epitaxial growth conditions for obtaining high quality a-plane AlN epilayers and photonics structures on r-plane sapphire substrates.

➤ Identification of cation vacancies and n-type conductivity control in AlGaIn alloys

In the past, AlGaIn alloys with high Al contents and pure AlN were known as excellent insulators due to their large energy bandgap up to 6.1 eV. It has been very difficult to convert them to semiconductors due to the large activation energies of dopants and the simultaneous generation of free electron traps during crystal growth. We have utilized DUV picosecond time-

resolved PL spectroscopy as an effective approach to identify the presence of these compensating centers for materials grown under different conditions. We have identified three types of Al vacancies that are free electron traps in AlGa<sub>x</sub>N alloys with high Al contents and in pure AlN. By monitoring/minimizing the Al vacancy related emission peaks, optimal growth conditions and layer structures for obtaining highly conductive n-type AlGa<sub>x</sub>N alloys were obtained. We have achieved record high room temperature n-type conductivity for Al<sub>x</sub>Ga<sub>1-x</sub>N alloys with high x. Furthermore, guided by PL and Hall effect measurements, we have achieved a value of room temperature n-type resistivity in AlN as low as 6 Ωcm (with a free electron concentration of about  $3 \times 10^{17} \text{ cm}^{-3}$ ) and hence establish the fact that it is possible to obtain n-type conduction in pure AlN with Si doping.

➤ Fundamental limits of p-type doping in AlN and AlGa<sub>x</sub>N

In Mg doped AlGa<sub>x</sub>N alloys, native defects such as nitrogen vacancies, ( $V_N^{3+}$ ) and ( $V_N^{1+}$ ), limit p-type conductivity of AlGa<sub>x</sub>N. Fig. 3 compares the 300 K PL spectra of (a) an undoped AlN epilayer, (b) a Mg-doped AlN epilayer with high resistivity, and (c) a Mg-doped AlN epilayer with measureable p-type conductivities (or reduced resistivities) at elevated temperatures. Undoped AlN has a strong band-edge emission peak at 5.98 eV due to the recombination of free excitons and exhibits virtually no impurity transitions in the low energy region, ensuring a good optical quality. AFM revealed an atomically smooth surface with a root mean square (RMS) roughness of about 7 Å within a 2 μm x 2 μm scan. These undoped AlN epilayers were employed as templates for the subsequent growth of Mg-doped AlN epilayers. For Mg-doped AlN epilayers, the PL spectra presented in Figs. 3(b) and 3(c) encompass an emission peak at around 4.7 eV, in addition to the band-edge emission at 5.94 eV. The band-edge emission peak at 5.94 eV has been identified and is due to the recombination of excitons bound to neutral Mg acceptors (or acceptor-bound excitons  $I_1$ ). We have identified that the 4.7 eV emission line is related to nitrogen vacancies ( $V_N^{3+}$ ).

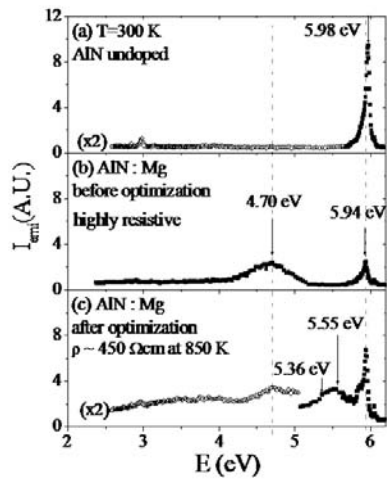


Fig. 3 PL spectra of (a) an undoped and highly resistive AlN epilayer, (b) Mg-doped and high resistive AlN epilayer and (c) Mg-doped AlN epilayer with measureable p-conductivity at  $T > 600 \text{ K}$ .

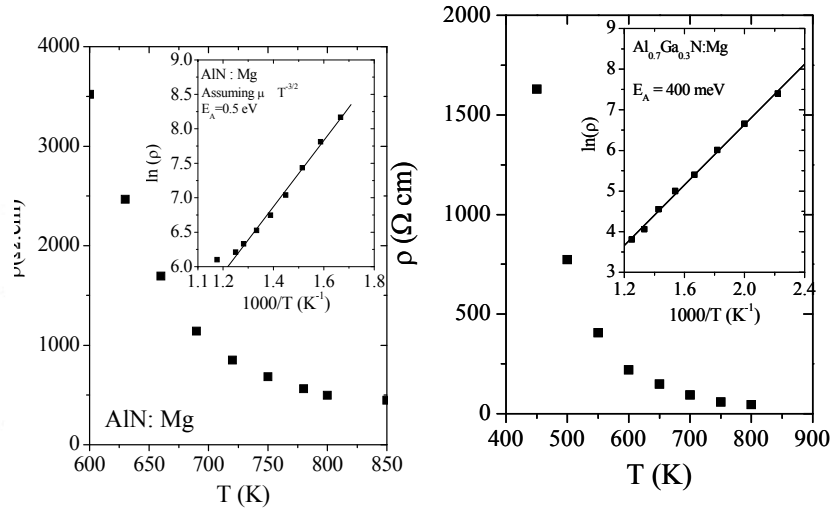


Fig. 4. Temperature dependent resistivity of an Mg-doped AlN epilayer (Left) and Al<sub>0.7</sub>Ga<sub>0.3</sub>N epilayer (Right). Insets: Estimated activation energy,  $E_A$ , of Mg acceptor is  $\sim 0.5 \text{ eV}$  in AlN and is  $\sim 0.4 \text{ eV}$  in Al<sub>0.7</sub>Ga<sub>0.3</sub>N. Optimized Mg-doped Al<sub>0.7</sub>Ga<sub>0.3</sub>N and AlN epilayers have measureable p-conductivity at  $T > 400 \text{ K}$  and  $T > 600 \text{ K}$ , respectively.

A clear correlation between the electrical and optical properties of Mg-doped AlN epilayers has been observed. Samples exhibiting strong emissions at 4.7 eV, such as that shown in Fig. 3(b), are generally highly insulating. Hall-effect measurements were carried out at elevated temperatures. The resistivity for one of our Mg-doped AlN epilayers, in which the intensity of nitrogen vacancy ( $V_N^{3+}$ ) related emission line at 4.7 eV was minimized, was measured in the temperature range between 400 and 900 K and the result is shown in Fig. 4 (Left), from which an activation energy of about 0.5 eV for Mg acceptor in AlN was obtained. This represents the first electrical measurement result for Mg acceptor activation energy in AlN.

Furthermore, by monitoring the  $V_N^{3+}$  related PL emission to the band edge emission intensity and p-type resistivity at elevated temperatures, we have confirmed p-type conduction in  $Al_{0.7}Ga_{0.3}N$  alloys at elevated temperatures and a p-type resistivity of about 40  $\Omega$ -cm at 800 K was observed, as illustrated in Fig. 4 (Right). Although Mg doped AlGaN alloys with high Al contents are generally highly resistive at room temperature, our NSF work has provided a more coherent picture for the conductivity control of AlGaN and AlN and determined the Mg acceptor energy level in AlGaN of the entire alloy range, as shown in Fig. 5.

By incorporating Al-rich  $Al_xGa_{1-x}N$  cladding layers with improved conductivities, we have obtained practical DUV LEDs. Fig. 6 shows the performance results of our 280 nm DUV LEDs under continuous (CW) operation with an optical power of 0.4 mW and forward voltage of 6.1 V at 20 mA, which are currently among the best devices in the world.

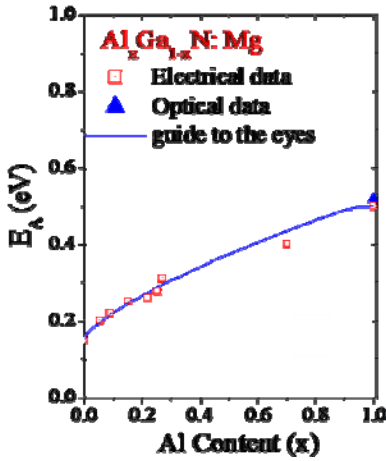


Fig. 5 Mg acceptor energy level in  $Al_xGa_{1-x}N$  alloys as a function of  $x$ .

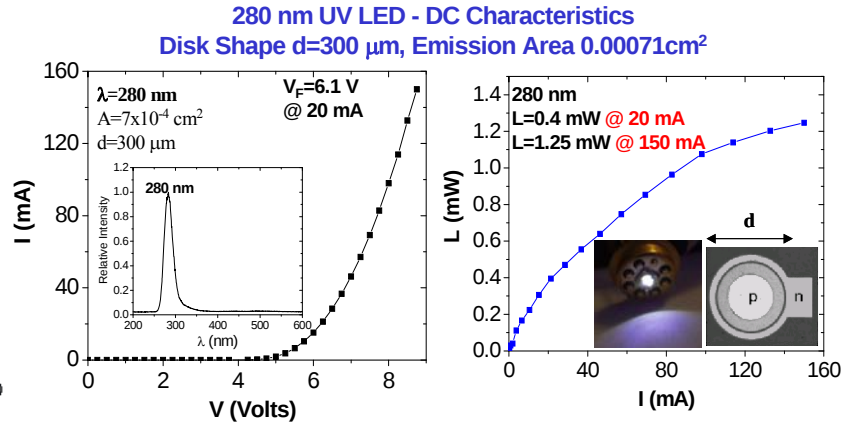


Fig. 6 L-I and I-V characteristics of our 280 nm DUV LEDs (275  $\mu$ m in diameter) based on AlGaN. The inset shows the EL spectrum and photo of a packaged KSU 280 nm DUV LED.

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25. J. M. Zavada, N. Nepal, C. Ugolini, J. Y. Lin, H. X. Jiang, R. Davies, J. Hite, C. R. Abernathy, S. J. Pearton, E. E. Brown and U. Hömmerich, "Optical and magnetic behavior of erbium-doped GaN epilayers grown by metal-organic chemical vapor deposition", Appl. Phys. Lett. 91, 054106 (2007).

26. Z. Y. Fan, J. Y. Lin, and H. X. Jiang, "III-nitride micro-emitter arrays: development and applications," Special Issue, J. Phys. D: Appl. Phys. **41** 094001 (2008).
27. H. X. Jiang and J. Y. Lin, "III-Nitride Micro-Cavity Light-Emitters," – in *"Wide Bandgap Light-Emitting Materials and Devices,"* edited by G.F. Neumark, I. Kuskovsky, and H. X. Jiang, published by Wiley –VCH Verlag GmbH, 2007.

#### Tutorials/short courses offered

1. "Processes and Devices of GaN Materials on Si," H. X. Jiang, Spring MRS meeting 2008 Tutorial, San Francisco.
2. "III-Nitride deep UV photonics," H. X. Jiang & J. Y. Lin, Short Course, SPIE - Photonic West Meeting, San Jose Jan. 2006.

#### Invited talks

1. "III-nitride photonic crystals," H. X. Jiang, University of Illinois, Urbana-Champaign, April 2008.
2. "Recent Development and Applications AC-LEDs," H. X. Jiang, 2007 International workshop on new materials research and industrialization," Wenzhou, China, December 2007.
3. "Progress and challenges of p-type doping in III-nitrides," H. X. Jiang, III-Nitride Workshop, Richmond, VA, Oct. 2007.
4. "III-nitride deep UV nano-photonics," H. X. Jiang, the 54<sup>th</sup> Midwest Solid State Conference, University of Nebraska – Lincoln, Oct. 2007.
5. "III-Nitride UV Photonics," H. X. Jiang, 3<sup>rd</sup> Asian Pacific Workshop on Wide Gap Semiconductors, March 11-14, 2007, Jeonju, Korea.
6. "Achieving highly conductive Al-rich AlGaN alloys for deep UV photonics," H. X. Jiang Photonic, West Meeting, San Jose, Jan. 2007.
7. "Er-doped GaN synthesized by MOCVD," IBEDM, Japan, Nov. 2006.
8. "Nitride Deep UV Photonics," H. X. Jiang, H. X. Jiang, the 53<sup>rd</sup> Midwest Solid State Conference, University of Missouri, Kansas City, Oct. 2006.
9. "III-Nitride Wide Bandgap Semiconductors for Optical Communications," H. X. Jiang, Lasers & Electro-Optics Society, LEOS 2006 Annual Meeting, Montreal, Canada, Oct 2006.
10. "Conductive high Al-content AlGaN alloys for deep UV photonics," 2006 Lester Eastman Conference on High Performance Devices, Cornell University, August 2006.
11. "Fundamental doping issues in Al-rich AlGaN alloys for deep UV photonics," H. X. Jiang, OIDA (Optoelectronics Industry Development Association) Roadmap Forum on Nitride LED and Laser Technology, Palo Alto, CA, May 2006.
12. "III-nitride deep ultraviolet micro- and nano-photonics," H. X. Jiang, Delivered in the Symposium on Quantum Sensing: Evolution and Revolution from Past to Future, SPIE - Photonic West Meeting, San Jose, Jan. 2006.
13. Nitride Photonic Crystals," H. X. Jiang, Meijo International Symposium on Nitride Semiconductors, Meijo University, Japan, Dec. 2005.
14. "Si and Mg-doped Al-rich AlGaN alloys for deep UV photonics," H. X. Jiang, 5<sup>th</sup> Akasaki Research Center Symposium, Dec., Nagoya, Japan, Dec. 2005.
15. "III-Nitride Photonic Crystals and AC LEDs," H. X. Jiang, 2005 International Forum on LED & Solid-State Lighting, Xiamen China.

16. "III-Nitride Photonic Crystals," H. X. Jiang, SPIE Symposium on Integrated Opto electronic Devices 2005 Conference on Light-Emitting Diodes – Research, Manufacturing, and Applications IX, San Jose, CA 2005.
17. "III-nitride deep UV photonics," H. X. Jiang, presented in Institute of Electron Technology, Warsaw, Poland, Aug. 2005.
18. "Recent advances in III-Nitride UV photonics," H. X. Jiang, NASA Goddard Space Flight Center, May 2005.
19. "Recent advances in III-nitride micro- and nano-photonics," H. X. Jiang, Navel Research Lab., May 2005.
20. "III-Nitride Photonic Crystals and AC LEDs," Presented in 2005 International Forum on LED & Solid-State Lighting, Xiamen China, April 2005.
21. "Nitride Photonic Crystals," J. Y. Lin, Eighth International Symposium on Contemporary Photonics Technology, Tokyo, Japan (January 2005).
22. "III-Nitride Ultraviolet Micro- and Nano-Photonics," J. Y. Lin, 2005 Conference on Lasers and Electro-Optics Quantum Electronics & Laser Science Conference, Baltimore, MD (May, 2005).
23. "III-nitride deep UV photonics," Air Force Workshop on III-nitrides, Alaska, Aug. 2004.
24. "Recent advances in III-Nitride UV photonics," OCPA meeting, summer 2004, Shanghai, China
25. "III-Nitride Photonic Crystals," Physics Colloquium, Taxes Tech, Fall 2004.
26. "Recent advances in III-Nitride UV photonics," delivered in Focused Session "Wide Bandgap Semiconductors," American Physical Society, 2004 March Meeting, Montreal, Canada.
27. "Recent advances in III-Nitride UV photonics," 2004 The Electrochemical Society, San Antonio, Texas, May 2004.

#### Books edited:

1. Ultrafast Phenomena in Semiconductors VII," Volume 4992 (2003), edited by K. T. Tseng, H. X. Jiang, and J. J. Song. Published by The International Society for Optical Engineering.
2. "Optical Materials," Vol. 23, Issues 1-2 - Proceedings of the 8th International Conference on Electronic Materials, 2002, Xi'an, China, edited by R. Zhang, T.F. Kuech, H. Jiang, J. Xu, D. Kip, Q. Sun, and J. Wang, published by Elsevier.
3. "Advances in III-V Nitride Semiconductor Materials and Devices," MRS Fall 2006 Meeting Proceedings Vol. 955E, Edited by C.R. Abernathy, H. Jiang, J.M. Zavada.
4. "Wide Bandgap Light-Emitting Materials and Devices," edited by G.F. Neumark, I. Kuskovsky, and H. X. Jiang, published by Wiley –VCH Verlag GmbH, 2007.