

History of blue LED consisted of nitride semiconductors

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Abstract— The electric energy used for illumination is said to be a quarter of total electric energy consumed on the earth. It is extremely effective to raise the luminous efficiency in order to realize the sustainable society. Furthermore, in the case of night illumination by using electric batteries charged from solar cells in the developing countries where electricity is not supplied, the high efficient emission is indispensable. The history of electrical illumination begins with an incandescent electric lamp. By inventing a fluorescent lamp, the luminous intensity has been improved from 15 lm/W to 95 lm/W. Presently, white light-emitting-diodes (LEDs) consisted of nitride semiconductors have been widely used because their efficiency is at least twice higher than that of a fluorescent lamp. The key device for realizing a white LED is a blue one. In this paper, the history of blue LED consisted of nitride semiconductors is reviewed.

Keywords—blue LED, white LED, Nitride semiconductor, GaN

I. INTRODUCTION

The electric energy used for illumination is said to be a quarter of total electric energy consumed on the earth. In order to realize the sustainable society, raising up the luminous efficiency is extremely effective. Furthermore, in the developing countries where electricity is not supplied, the high efficient emission is indispensable for night illumination by using electric batteries charged from solar cells. The history of electrical illumination began with an incandescent electric lamp. By inventing a fluorescent lamp, the luminous intensity has been improved from 15 lm/W of an incandescent electric lamp to 95 lm/W.

With respect to the light emission from semiconductors, SiC is the first [1]. In 1962, N. Holonyak, Jr. reported for the first time on the light-emitting-diode (LED) constructed with p-n junction of Ga(As_{1-x}P_x), which LED emitted red light [2]. In 1971, M. G. Craford succeeded in the green p-n junction LED of Ga(As_{1-x}P_x) doped N as an isoelectronic trap [3].

The research on SiC was not progressed because the crystal growth of SiC was difficult. Instead of SiC, II-VI group semiconductors, mainly ZnSe, for the blue emission was closed up. The powders of these materials have been already applied for phosphor in fluorescent lamps for a long time. The direct emission from ZnSe was also studied in 1960s. Since the p-type ZnSe was difficult to be fabricated, a metal-insulator-semiconductor (MIS) diode was fabricated and the blue

emission was successfully observed [4]. For a while afterwards, the research was focused on how to fabricate p-type ZnSe. At last, p-type ZnSe was obtained for the first time by controlling the supply ratio of Se to Zn (VI/II ratio) and supplying radical nitrogen [5] [6]. In 1971, a p-n junction diode of ZnSe was fabricated [7]. In parallel, on SiC, p-n junction LEDs were developed. Since the high efficient emission cannot be expected because its optical transition is indirect, the research on SiC aborted. Meanwhile, the nitride semiconductor, mainly GaN, has been studied. The drastic improvement in GaN, InGaN single crystal growth as a material for the blue emission, the materials design for constructing a double-hetero (DH) structure which is indispensable for the highly efficient emission, and p-type conduction were achieved. However, in 1991, 3M group suddenly reported the first laser oscillation from the ZnCdSe/ZnSe DH structure [8]. The device lifetime of this laser diode strongly depended on the VI/II ratio. For the long device lifetime, the value in the two-places of decimals had to be precisely controlled [9]. Sony group proposed ZnMgSSe for an electron barrier in the LD structure and fabricated a high efficient LD. This ZnMgSSe is lattice matched to a GaAs substrate used and p-type ZnMgSSe is very effective as a barrier for injected electrons. Blue-green LD was fabricated with the structure of the separate-confinement heterostructure (SCH). However, the longest device lifetime of only 480 hours was obtained. This reason has been considered that it is difficult to control point defects in II-VI semiconductors. These point defects worked on dark spots. As a result, the research on VI-II semiconductors has declined. Instead of II-VI semiconductors, nitride semiconductors has become to be earnestly studied. Blue LEDs and LDs have successfully fabricated and become commercially available.

Presently, white LEDs consisted of nitride semiconductors have been widely used because their efficiency is at least twice higher than that of a fluorescent lamp. The key device for realizing a white LED is a blue one because the white light is generated with the combination of a blue LED and a yellow phosphor. This blue LED won the Nobel Prize 2014 in physics according to the contribution to the energy saving for lighting. In this paper, the history of blue LED consisted of nitride semiconductors is reviewed.

II. MATERIAL DESIGN AND EPITAXIAL GROWTH

To realize blue LEDs, there is the long history on the material design and the epitaxial growth. The first synthesis of GaN was performed in 1932 [10]. The gas phase epitaxial growth of a GaN thin film which is a main material in nitride semiconductors constructing blue LEDs was performed on a sapphire substrate in 1969 [11]. The blue

emission was obtained from LEDs with MIS-type GaN grown on a sapphire substrate in 1971 for the first time [12]. After this event, the GaN research was focused on the development of the growth technique for high-quality GaN on a sapphire substrate. At first, the growth method of GaN through an AlN thin layer on a sapphire substrate by molecular beam epitaxy (MBE) was studied [13]. The meaning of AlN was that its lattice constant was intermediate between GaN and sapphire, and that AlN can fit to sapphire better than GaN because Al and N of AlN was a component of GaN and sapphire, respectively. By using the growth method of metalorganic vapor phase epitaxy (MOVPE) which is used for the mass production of devices with the conventional semiconductors such as GaAs and InP, even the transparent GaN film cannot be obtained for a long time because GaN had to be heteroepitaxially grown on a (0001)-plane sapphire substrate for the lack of a GaN substrate, differently from the homoepitaxial growth in conventional semiconductors. 13.8% of the lattice-mismatch between GaN and sapphire makes it difficult to grow high-quality GaN. In 1986, by adopting an AlN thin film as a buffer layer and introducing the concept of a low-temperature grown buffer layer which has been already developed in the growth of GaAs on an Si substrate [14], a transparent GaN film with a smooth surface was successfully obtained for the first time [15]. Really speaking of this way, in the growth process at the constant temperature, the equipment did not work order firstly, and later the equipment was accidentally recovered, and the temperature raised up to the usual growth temperature. Eventually, high quality GaN with a smooth surface was obtained [16]. In Fig. 1, this procedure using a GaN buffer layer is shown. A GaN buffer layer grown at low temperature was shown to be as effective as an AlN one [17]. This GaN buffer layer has the advantage in the annealing process for the crystallization lower temperature than that of an AlN buffer layer. Of course, this GaN buffer layer can be used in MOVPE [18].

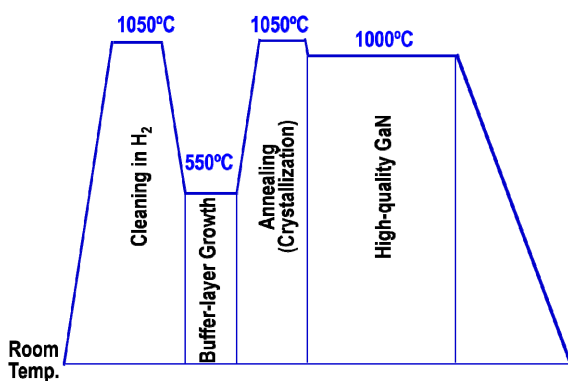


Fig. 1. Growth procedure of two-step growth for GaN.

The epitaxially grown GaN usually shows n-type conduction. For fabricating LEDs, a p-type material is also necessary. Generally speaking, a p-type conduction material with wide band-gap is difficult to be fabricated because point defects have to be precisely controlled for obtaining the p-type conduction. Amano *et al.* tried to dope Mg as a p-type dopant.

At the initial stage, Mg-doped GaN became only highly resistive, however, this material was patiently investigated with cathode luminescence (CL). By this investigation, this material was found to show p-type conduction with Hall measurement in 1989 [19]. This method was named “low-energy electron beam irradiation (LEEBI)”. It cannot be believed that irradiated electrons transport Mg atoms from interstitial positions to substitutional ones and finally doped Mg atoms can be activated because the mass of an electron is too small in comparison with that of an Mg atom. A few year later, the mechanism of this phenomenon was proved that the thermal energy generated during irradiation makes hydrogen atoms passivating Mg atoms desorb from Mg atoms [20]. This phenomenon is the same as that of the hydrogen passivation of acceptors in Si [21].

To construct a DH structure indispensable for high-efficient LEDs, two more elements in addition of two elements forming compound semiconductors such as GaN are necessary for satisfying two freedoms of controlling the band-gap energy and the lattice-constant. The latter is important for the growth lattice-matching to each layer. The former is for the band engineering in a DH structure. As a result, four elements are needed. Therefore InGaAlN quaternary system was proposed in 1989 because AlN and InN have the same crystal structure of wurtzite as GaN [22],[23]. The relation between the lattice-constant along a-axis and the band-gap energy E_g of InGaAlN is shown in Fig. 2, compared with conventional III-V semiconductors such as GaAs and InP, and II-VI semiconductors.

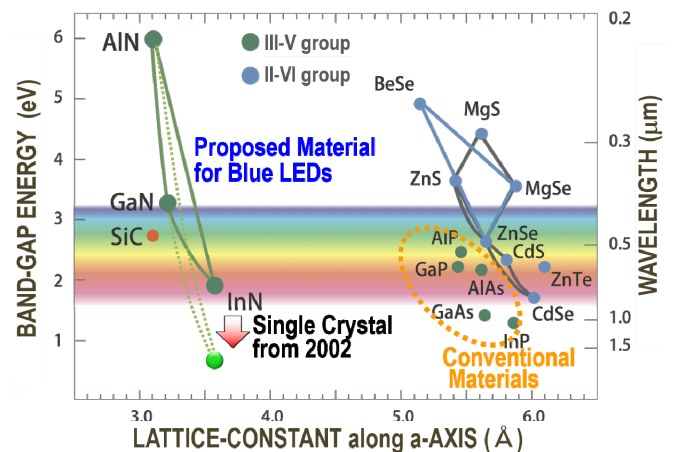


Fig. 2. Lattice-constant along a-axis and band-gap energy [12].

From this figure, it is clearly found that the simplest material for a blue emission material in InGaAlN is InGaAlN because InAlN is predicted to have severe phase separation due to the difference of atom sizes between Al and In. The growth of InGaAlN had been already studied [24], however, a single crystal InGaAlN was not obtained. This reason is explained as follows. In the growth conditions for the heterostructure of GaAs and InP, all the growth conditions such as temperature were constant. The flow rate ratio of the group V to the group III (V/III ratio) was also constant of 100 to 200. The carrier gas to transport the source gases to the reactor was always hydrogen

because hydrogen can take out oxygen and water from the reactor. The growth conditions in the gas phase strongly depends on the equilibrium vapor pressure of elements composed in materials. The equilibrium vapor pressure of elements between the gas and solid phases has to be mostly paid attention in the gas-phase growth. The equilibrium vapor pressure of nitrogen in AlN, GaN, and InN which are the end materials in nitride semiconductors are shown in Fig. 3 in comparison with the equilibrium pressures of As in GaAs and P in InP [22].

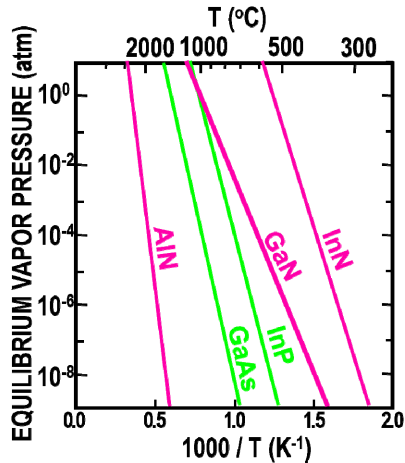


Fig. 3. Fig. 3 Equilibrium pressure of group-V elements between gas and solid phases [12].

The growth temperature of GaAs and InP is about normally 600 °C. On the other hand, the growth temperature of GaN is over 1000 °C. The equilibrium vapor pressure of nitrogen in GaN at the growth temperature becomes by a several order magnitude higher than that of As in GaAs. In an extreme case of InN, this value raises up a several order higher than that of GaN. From the equilibrium vapor pressure, the growth of InGaN can be easily imaged to be considerably difficult. The ammonia used as a nitrogen source makes it more difficult to grow InGaN because ammonia is dissolved based on the following reaction. This dissolution efficiency is said to be less than 1 %. These situations also make it difficult to fabricate bulk GaN and a GaN substrate. This means that the homoepitaxial growth cannot be performed, but GaN has to be heteroepitaxially grown on a foreign substrate.



If hydrogen is used as a carrier gas as is the case with the growth of GaAs, the dissolution is suppressed to be understood from the reaction (1). From this consideration, nitrogen has been adopted as a carrier gas for promoting the dissolution of ammonia. The growth temperature has been felt down to 800 °C for the growth in the low equilibrium pressure of nitrogen. The V/III ration has been raised up to 16,000 for supplying the much quantity of nitrogen. As a result, the growth of a single crystalline InGaN film has become available for the first time [22]. The control of In mole-fraction in InGaN has been

realized as shown in Fig. 4 [25]. In this figure, TMIn and TEGa is trimethylindium and triethylgallium as an indium source and a gallium one, respectively.

The photoluminescence from InGaN grown at 800 °C was successfully observed [26]. As mentioned above, the basic technologies for the LED fabrication were established up to 1989. The key technologies and the affiliations developed them are summarized with the affiliations developed them in TABLE I.

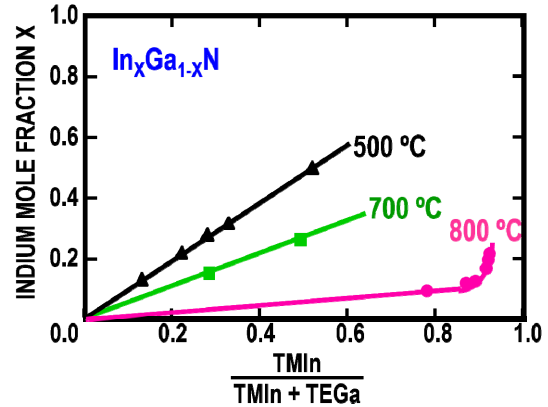


Fig. 4. Control of In-mole-fraction in InGaN [15].

TABLE I. KEY TECHNOLOGIES DEVELOPED FOR BLUE LEDs AND LDS

	Issues	Year	Affiliation
High Quality GaN	AlN Buffer Layer	1986	Nagoya Univ.
Material for Blue Light	Proposal of InGaAlN	1987	NTT
	InGaN Growth	1989	NTT
Conduction Type Control	p-GaN Growth	1989	Nagoya Univ.

However, InGaN with all the composition from GaN to InN cannot be obtained because there is the severe phase separation as shown in Fig. 5 [27]. This phase separation occurs due to the difference between lattice constants of AlN, GaN, and InN.

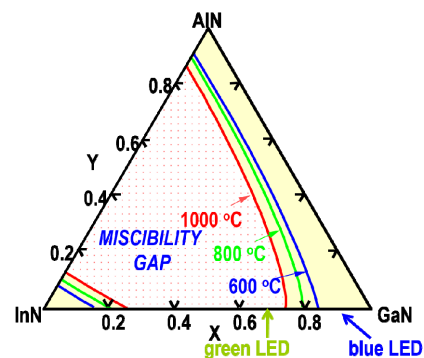


Fig. 5. Phase separation of InGaAlN [17].

III. DEVICE FABRICATION

The growth conditions mentioned in the previous section have been widely used in the mass production of blue, green, and white LEDs. By combinations of all the technologies mentioned above, the blue LED with a double heterostructure was fabricated in 1993 [28],[29]. This blue LED with the shell type became commercially available in November, 1993 from Nichia Corporation. The light output power was 1.2 mW and the external quantum efficiency 2.2 %. The green LED also became commercially available in September, 1996 from the same company. The light output power was 2mW and the external quantum efficiency 4.2 %. Thereafter the device structure was progressed for obtaining high power. S. Nakamura *et al.* of Nichia Corporation introduced the multiple-quantum well structure into the emitting layer of LEDs [30]. The typical LED structure is shown in Fig. 6. For obtaining higher power, an AlGaIn layer sometimes is used between GaN and p-type GaN for suppressing the overflow of electrons to the p-type side.

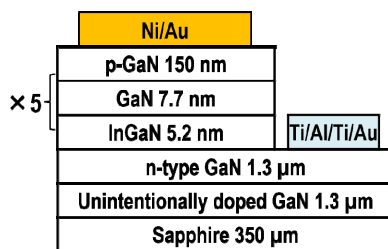


Fig. 6. Typical structure of LED.

For an example, the relation between the light output power and the injection current for a blue LED is shown in Fig. 7 [31].

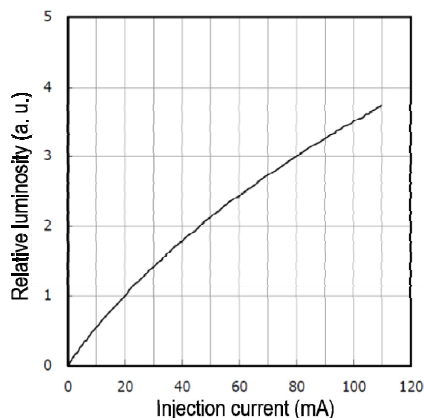


Fig. 7. Relation between relative luminosity and injection current for blue LED. The luminous intensity is normalized by 6.0-8.4 Cd [31].

The external quantum efficiency in all the visible region is shown in Fig. 8. In $\text{In}_x\text{Ga}_{1-x}\text{N}$, the efficiency decreases in the longer wavelength side because of the phase separation as shown in Fig. 5. In $(\text{Al}_x\text{Ga}_{1-x})_{0.52}\text{In}_{0.48}\text{P}$, the efficiency decreases in the shorter wavelength side because the difference in the band-gap energy between an emitting layer and cladding one in the DH structure decreases and the carrier overflow occurs. The efficiency of green LEDs is low. Fortunately, the

visibility with human eyes is very high in this color region and as a result we feel green LEDs are also bright.

A white LED was invented by Nichia Corporation in 1996 [32]. Its basic concept is shown in Fig. 9. It was considered that this invention was based on the experience of the top maker in the phosphor field. This white LED was simultaneously sailed as reported. The light output power of a shell-type white LED was 1.4 mW and the external quantum efficiency 3.5 %. In the TABLE II., the characteristics of the present LEDs commercially available from Nichia Corporation are shown [33]. They have been drastically improved in comparison with the initial stage. Even today, the characteristics of LEDs have been continued to be improved.

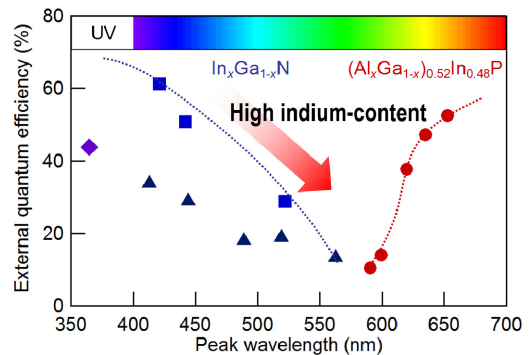


Fig. 8. External quantum efficiency of LEDs in all the visible region.

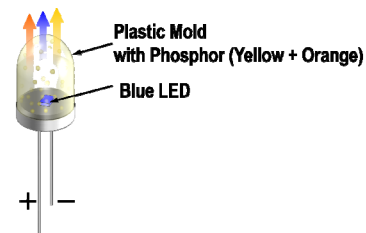


Fig. 9. Concept of white LED.

TABLE II. CHARACTERISTICS OF PRESENT LEDs COMMERCIALY AVAILABLE

LED color	blue	green	white
light output power (mW)	45	19	24
external quantum efficiency (%)	85	41	54

The laser diodes (LDs) have been also developed. In 1997, a blue LD was reported on the oscillation. In 2001, this blue LD with the light output power of 5 mW and the external quantum efficiency of 2 % became commercially available from Nichia Corporation. The green LD with 10 mW was also commercially available. For an example, the relation between the light output power and the injection current for a blue LD is shown in Fig. 10 [34].

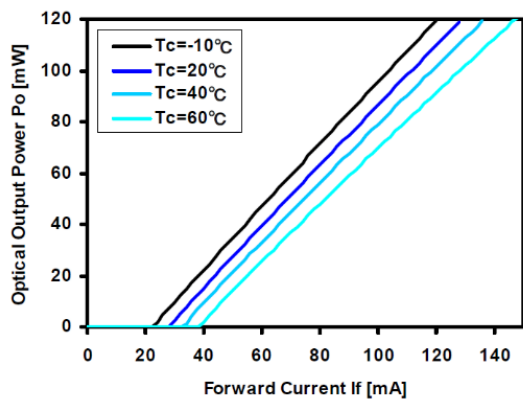


Fig. 10. Relation between the light output power and the injection current for blue LED at 25 °C [34].

The present blue and green LEDs with 45m W and 85%, and 19mW and 41% have been commercially available, respectively [35].

IV. CONTRIBUTION OF WHITE LEDS TO SUSTAINABLE SOCIETY

The transition of light sources is shown in Fig. 11 [36]. Presently, the efficacy of incandescent lamps and fluorescent ones is 15 lm/W, and 95 lm/W, respectively. For the white LEDs sold in 2014, this value increased to 180 lm/W. In the research level, the efficacy reaches to 315 lm/W in 2014 [25]. This value is twenty times and three times larger than those of incandescent lamps and fluorescent lamps, respectively. In this way, white LEDs have much higher efficiency than other lamps for lighting.

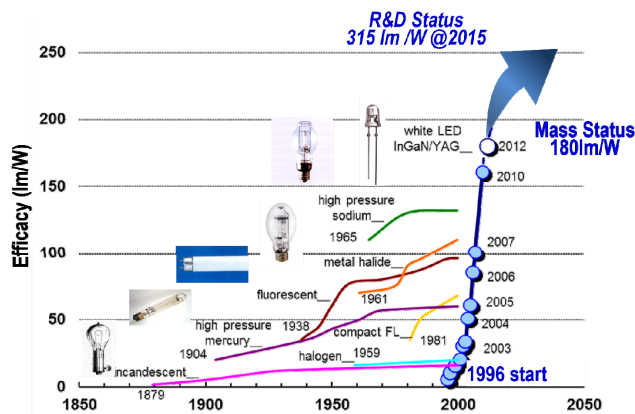


Fig. 11. Transition of light sources [25].

Since the electric energy used for illumination is said to be a quarter of the total electric energy consumed on the earth, white LEDs are very useful for energy saving. TABLE III. shows the status of white LEDs for lighting [26]. In 2025, the energy saving by the penetration of white LEDs into lighting is predicted to be the same as the total electric generating capacity in Japan. Another important point of white LEDs is much longer device lifetime than the other lamps.

TABLE III. ENERGY SAVING BY USING LEDS [26]

Year	2005	2010	2015	2020	2025
LED Penetration (%)	0.05	2	12	30	55
Energy Saving (RWh/yr)	2	67	330	720	1,100 ^a
Energy Cost Saving (MS/yr)	200	6,700	33,000	72,000	110,000
Energy Generating Capacity Saving (GW)	0.2	8	38	82	125

^a Total Electric Generating Capacity in Japan

V. CONCLUSIONS

A blue LED which is the main theme in this paper has been commercially available through the history as described above. There are two routes for this commercialization. One was Nichia Corporation. They began to sell LEDs in 1993. The other was Toyota Gosei to which Nagoya university group transferred the developed technologies. They collaborated and this company succeeded in the commercialization of LEDs in 1995. Green LEDs began to be sold in 1995. Regarding with blue and green LEDs, they became commercially available in 2001 and 2010, respectively.

White LEDs became commercially available in 1996. The penetration of these into solid-state lighting have been strongly desired for energy saving.

For a long time, InGaAlN has been believed to cover the color region from the orange of InN to the ultraviolet of AlN. The band-gap energy of InN with the radio-frequency (rf) sputtering and the reactive cathodic sputtering was reported to be 1.9 eV [38] and 2.1 eV [39], respectively. From the band-gap energy measurement of single-crystalline InGaN with the indium mole fraction of 0 to 42%, its band-gap energy was predicted to be much larger than the reported value [12]. For obtaining single-crystalline InN, 12 years was needed after this prediction because of its tremendously high equilibrium vapor pressure of nitrogen. In 2002, the band-gap energy of InN has been experimentally proved to be 0.7-0.8eV as shown in Fig. 2 [40]. This result opens the window for a nitride semiconductor InGaAlN to be applied for the range from infrared to ultraviolet. In addition to the field of optics, a nitride semiconductor is promising in the field of transport switching devices such as high-frequency, high power, and high breakdown voltage transistors. Recently, GaN/AlGaN high electron mobility transistors (HEMTs) have been already penetrated to the basic stations for cellular phones [41].

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