CHAPTER 8

LIGHT EMITTING DIODE (LED) CHIP FABRICATION USING LOW TEMPERATURE PROCESSES

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ABSTRACT

Commercial light emitting diode (LED) chips are fabricated using metal organic chemical vapor deposition (MOCVD) process, which require high temperature process of above 1000 degree Celsius (°C). Therefore, alternative processes such as pulse sputtering deposition (PSD), thermal annealing and electron beam have been investigated. Although a series of investigations have been reported on fabrication of LED chips employing various type of deposition, to our knowledge, most of these investigations are still not excluded from the high temperature process as their secondary process. Hence, this chapter reveals the investigation of LED chip fabrication at low temperature (which is below 1000 °C) and its possibility and reliability to emit light at wavelength of 442 nm, 460 nm and 520 nm. At this point, the use of combination of various techniques such as thermal annealing, PSD and electron beam helps to avoid expensive and high temperature techniques such as MOCVD. Development of low-cost production and commercial fabrication tools for LED and optoelectronic devices is a promising subject for future research. In addition, basic LED and device structure are also explained and illustrated in this chapter.

Keywords: Low temperature, Blue-LED, sputtering deposition, light emitting diode
8.1 INTRODUCTION

It is very clear from the current trend that the source of light will be replaced by LED or light emitting diode. Recently, gallium nitride (GaN) based LEDs have attracted a great interest because of its wide application in solid state lighting, biomedical research, agriculture, aquaculture, biomass production, phototherapy, and environmental applications [1]. Remarkable breakthroughs of GaN based LED had been achieved since 1996 when Nichia Chemical Corporation, Japan (team of Prof. Shuji Nakamura, Nobel Prize winner in 2014) developed the first white LED and it was successfully commercialized around the world [2]. This achievement creates new motivation to all researchers to explore and enhance the current knowledge and technology of GaN based materials more attractive for electronic applications until today. Despite the breakthrough progress of blue-LED over the decades, ultimate goal of attaining low temperature for LED chip fabrication process is still remains challenging. One of the difficulties in current LED fabrication is the usage of expensive metal organic chemical vapor deposition (MOCVD) techniques. MOCVD techniques require high temperature process to grow the thin films from precursor gas and to obtain the solid epitaxial layer.

Compared to MOCVD process, magnetron sputtering deposition represents a low-cost growth techniques and simple source materials. Magnetron sputtering also has great ability to preparing uniform thin films in low temperature process [3]. However, until today LED epitaxial layer that directly grown using magnetron sputtering techniques is not well studied. Only few papers reported the used magnetron sputtering technique for blue-LED chip fabrication [4]. In this chapter, basic LED operation and device structures are explained and illustrated. The fabrication of conventional chip LED is also reviewed in the aspect of process temperature. Although a series of investigations has been reported on fabrication of LED chips employing various types of deposition, most of these investigations are focused on high temperature process. Hence, this chapter is focused to review low temperature led chip fabrication process for its future application on plastic or fabric substrates.

8.2 LED WORKING PRINCIPLE, FABRICATION AND PACKAGING

Currently, the LED lamp is popular due to its efficiency and now it becomes
a replacement for other white light sources such as fluorescent lamp and incandescent lamp. LEDs are available in various colors, designs and sizes depending on its requirement. LEDs are usually used for traffic lights, signages, street lights, general lighting, indication light on devices and large video screen. The standard LED commonly has a cylindrical shape and the outer packaging is closed by hemisphere shape at the spot where the light is emitted. LED chip consist of several layer, reflector tray with contact to cathode, gold wire contact to anode and plastic lens to combine and holds all the components inside.

A LED is an electronic device that use electric current pass through it to emit visible light. LED operates in forward bias like a normal pn-junction diode. This process occurs when the charges that carry the electric current which consist of holes in p-type semiconductors and electron in n-type semiconductors combine within a pn-junction in semiconductor materials and release energy in the form of light. In order to use LED device, n-type material must be connected to the negative terminal of the battery and p-type material must be connected to the positive terminal of the battery. Figure 1 is a simple LED pn-junction configuration under forward bias condition.

![Figure 1: Schematic of simple forward biased LED p-n junction and LED basic operation when electrons and holes recombined in the active region to produce photons][5],[6]

Fabrication of LED chip structure itself is much more complicated. Figure 2 shows the basic LED chip structure contain several layers made up from compound semiconductors which consist of n-type layer, active layer and p-layer. This entire layer has different mode of electrical conductivity so that
the pn-junction can emits its energy in the form of light. The films of chip LED structure normally grown epitaxially using metal organic chemical vapor deposition (MOCVD) and metal organic vapor phase epitaxy (MOVPE) in high temperature condition. Generally, GaN-based materials are used for n-layer and p-layer. To create the n-type and p-type GaN layers, it was doped using silicon (Si) to obtain n-type GaN layer while magnesium (Mg) to obtain p-type GaN layer. The doping techniques such as thermal annealing, impurity diffusion, ion implantation is able to alter the undoped GaN to have high density of electrons or holes. Another important layer is the active layer where the pn-junction in semiconductor materials releases energy in the form of light. Normally, active layers materials use semiconductors such as gallium arsenide (GaAs), gallium phosphide (GaP), indium gallium nitride (InGaN), aluminum gallium nitride (AlGaN) and aluminum gallium indium nitride (AlGaInN). All these materials have different band gap which capable to emits light in different wavelength ranging from infrared to visible light through until ultraviolet. As for the rest, the substrates also can affect the quality of chip LED. Typically, the substrates in LED chip have a different in lattice mismatch between the substrates and the epitaxial layers. This cause stacking faults and threading dislocations in GaN epilayers [7] and lead to cracking [8] in LED chip. Hence, the substrates of LED chosen normally have close lattice match to the GaN layer. Common substrates are GaN, sapphire (Al2O3), GaAs, Si and silicon carbide (SiC) [8–11].

The structural design for LED packaging is also important. Through packaging, LED can improve the luminescence efficiency and protect the LED chip from defect, die cracking, dopant diffusion, and electromigration [12]. Electromigration happen when there is momentum transfer between conducting electrons and diffusing metal atoms during condition of high current densities and high temperatures in metals. During the condition mentioned, the ions in the metal is gradually drift in the direction of electron flow and result in mass transportation, thus will cause dislocations, leakage current, point defects and non-radiative recombination along the LED chip edges [12]. This packaging structure successfully makes sure the normal operation of diode chips and emission of visible light. Commonly, LED packaging includes a transparent lens which is made from a transparent epoxy and a reflector cup into which the LED chip is mounted, as shown in Figure 3.
Figure 2: Basic structure of LED chip using InGaN/AlGaN double heterostructure for blue emission LED [6],[13]

Figure 3: Final LED structure type pinned LED. Cross sectional image of an LED packaging. LED chip sits on a reflector cup and is embedded in a transparent epoxy whose shape favors light extraction to air. Combination of LED chip emission pattern, shape of reflector cup and refractive index of transparent lens influence the radiation pattern [14]
8.3 FABRICATION OF CONVENTIONAL BLUE-LED AND UV-LED

The conventional LED fabrication for high efficiency blue-light emission in the 90s commonly use hydride vapor phase epitaxy (HVPE), molecular beam epitaxy (MBE) and MOCVD methods to grow the epitaxial layer. All these methods were using high temperature process up to 1000 °C in return to achieve high quality and solid epitaxial layer. The epitaxial growth of GaN for LED application using MOCVD has been firstly reported by Nakamura, Senoh and Mukai in 1993 [15],[16]. Figure 4 shows schematic diagram of blue-LED by Nakamura, Senoh and Mukai in 1993. It was reported that the sapphire substrates were heated first to 1050 °C in hydrogen stream, then the GaN buffer layer is grown using 510°C and followed by Si doped n-type GaN layer using 1020°C in trimethyl-gallium (TMG), SiH₄ and NH₃ precursor gas. After the GaN layer, the InGaN layer is growth using 800°C in TMG, NH₃ and trimethyl-indium (TMI) working gas. The process temperature then increased to 1020 °C to growth Mg doped p-type GaN. After the film growth, low energy electron beam irradiation (LEEBI) treatment was performed to obtain highly p-type GaN layer. Finally, the fabrication of LED chip accomplish as gold (Au) contact layer was evaporated to p-type GaN and aluminum (Al) contact onto n-type GaN layer. Prior to the contact pad deposition, a small portion of p-type GaN layer is etched until partially n-type GaN layer was exposed.

![Figure 4: Schematic diagram blue-LED by Nakamura, Senoh and Mukai in 1993 [15][16]](image)

The growth technique shown by Nakamura’s experiment is not much different with other growth technique that has been published [15], [17–19].
Most of the fabrication processes, until today, using high temperature and high flow rate working gas.

After the development of blue-LED, ultraviolet-LED (UV-LED) is the latest issue that has been addressed by many researchers. Recent publication in [3] used high temperature for UV-LED chip epitaxial layers. Figure 5 shows the schematic of UV-LED and fabrication method with respective temperature. In brief, the aluminum nitride (AlN) thin films which act as buffer layer were deposited on sapphire substrates using reactive radio frequency magnetron sputtering deposition. Prior to deposition, the substrate was heated to 600 °C. Then, the film was transferred to MOCVD chamber to grow 20 periods of AlN/Al 0.65 Ga 0.35 N at 1130 °C. After that, a silicon-doped n-type Al 0.56 Ga 0.44 N layer was grown on the AlN/AlGaN at 1005 °C. Next for the active layer, 5 pairs of AlGaN were grown followed by Mg doped p-AlGaN and p-GaN. At the end of the process, the structure was annealed at 800 °C to activate the Mg acceptor.

![Figure 5: Schematic diagram of UV-LED [3]](image)

**8.4 FABRICATION OF LED CHIP USING LOW TEMPERATURES PROCESS**

In this section, fabrication of LED chips using low temperature is discussed. In general, the development of III-V compound semiconductors group materials that use low temperature process provide more uniform thickness across the substrate materials and overcome the large lattice mismatch.
between the substrate and GaN epilayers by introducing nucleation layer between the thin films [20]. One of the techniques for growing these group materials at relatively low temperature is using magnetron sputtering deposition. There are three types of sputtering process which are direct current (DC) sputtering, radio frequency (RF) sputtering and high power impulse magnetron sputtering (HiPIMS) [21]. The sputtering process generally produced from the bombardment of target surface with energetic gaseous ions that cause the physical ejection of surface atoms. The number of ejected atoms is depends on the incident energy of ions [22]. Sputtering technique is also applied for ion etching, analytical techniques and thin film deposition. Further details of magnetron sputtering deposition can be found in various reference books [23–27].

8.4.1 LED Chip Fabrication Using Low Temperature Process

Fabrication of LED chip using low temperature is not well established yet and there are several papers reported on the experimental works. To date, researchers are focusing to fabricate a blue-LED using temperature below 1000°C with an output power of 1.5 mW, an equivalent quantum efficiency (EQE) of 2.7% and 450 nm of emission wavelength, which is the standard specification of commercial LED [2]. The main reason of LED chip using high temperature is because of the process of MOCVD itself required high temperature to deposit the precursor gas to grow the epitaxial layer and obtain the solid epitaxial layer. Hence, further studies to improve the technique for fabrication of LED using lower temperature must be established so that LED can be fabricated on a plastic or fabric in the near future.

8.4.2 Low Temperature Blue-LED Produced Using p-ZnO/(CdZnO/ZnO) MQWs/n-ZnO Configuration

There are several reports on deposition of single layer structure for optimization of LED especially for p-type layer and contact layer [28-30]. However, the experimental work on fabrication of the complete LED chip using all low temperature process which is below than 1000 °C is not reported yet. Recently, blue-LED based using p-ZnO/(CdZnO/ZnO) MQWs/n-ZnO configuration at room temperature using dual ion beam sputtering had been fabricated by [31]. The n-layer structure was made up from Ga-doped ZnO which was deposited on Si substrates by using substrate temperature of 400 °C [32]. After that, two-period cadmium zinc oxide (CdZnO) multi quantum well (MQW) was deposited consecutively
with i-ZnO barrier which is grown using same temperature and method with n-layer structure. Lastly, p-layer structure was deposited on top of the CdZnO/ZnO MQW at 500 °C using Sb doped ZnO (SZO) target. Each layer undergoes in-situ annealing process at 800 °C in vacuum to activate the dopant. To complete the LED as a device, an indium electrode was used as an ohmic contact on n-Si substrate and p-type SZO. The temperature to form the ohmic contact on both layers is 400 °C in high vacuum condition. The device was observed to emit UV electroluminescent (EL) emission until 442 nm at 130 mA injection current using turn-on voltage of 4.69 V. Figure 6 shows the schematic diagram of their fabrication processes.

![Figure 6: Schematic diagram of blue-LED based on p-ZnO/(CdZnO/ZnO) MQWs/ n-ZnO using low temperature][31]

### 8.4.3 Fabrication of GaN-based LED on Flexible Metal Foils

Recently, H. Kim et. al. performed fabrication of full color GaN based LED diodes on almost lattice-matched flexible hafnium (Hf) metal foils using pulse sputtering deposition (PSD) in the temperature range of 400-700 °C [33]. As shown in Figure 7, the n-type GaN layer was deposited on Hf metal foil at temperature 700 °C. Prior to deposition, the Hf metal foil was annealed at 1000 °C for 60 minutes in vacuum to produce highly c-axis oriented structure. Then, five periods of InGaN/GaN MQW was deposited and topped by Mg-doped p-type GaN layer all using 400 °C. Lastly, In and Pd/Au electrodes were used to form ohmic contacts using e-beam evaporation. The LED structures have a turn-on voltage of 5 V and exhibits good rectifying characteristic with leakage current of 0.1 mA at -5 V. It was found that the blue light EL intensity increase (460 nm) with an increasing of the injunction current between 4 and 8 mA. This result indicates that flexible Hf foils can be used for blue-LED fabrications with fairly operation.
Figure 7: Schematic diagram of full-color GaN-based LED on nearly lattice-matched flexible hafnium metal foils [33]

8.4.4 Fabrication of InGaN-based LED on Amorphous Substrates

Fabrication of full-color InGaN-based LED using low temperature process has been presented by J. W. Shon et. al. [4]. The highest temperature used for the nitride films in this experiment is only at 760 °C by pulsed sputtering deposition (PSD) techniques. High crystalline nitride films successfully prepared on amorphous fused silica substrates. It was done by growing the multilayer graphene layers on nickel (Ni) foil using CVD then transferred onto amorphous fused silica substrates or amorphous SiO$_2$ which was prepared by thermal oxidation of Si. AlN buffer layer was deposited followed by GaN layer using PSD with a growth rate of 1-2 mm/h to form n-type layer AlN/graphene/amorphous SiO$_2$ structures. Then, 5 periods of alternating InGaN/GaN MQW were grown on n-type layer followed by Mg doped p-type GaN layer is grown on the top of MQW, as shown in Figure 8. The nitride layers were deposited at substrate temperatures range of 550-760 °C. To perform electroluminescence measurement on LED structures, Pd/Au and In electrodes on p- and n-type GaN surfaces was deposited. It has been reported that green LEDs was successful created, and it was found the emission spectra intensity increase as increasing of injection current from 2.1 to 10.1 mA with increasing of photoluminescence from 405 nm to 520 nm. In addition, blue and red LEDs also have been fabricated and successfully operated by changing the indium composition in PSD-InGaN layers. The study showed that the combination of growth techniques can lead to the development of large area flexible inorganic devices in the future since LED can be fabricated on amorphous substrates.
Figure 8: Schematic diagram of full-color InGaN-based LED fabricated on amorphous substrates using pulsed sputtering deposition [4]

8.5 SUMMARY

In summary, the basic component of LED that enables it to emit light is the charges that carry the electric current which consist of holes in p-type semiconductors and electron in n-type semiconductors which combine within a pn-junction in semiconductor materials. The fabrication of LED chip using low temperature below 1000\degree C is possible and reliable as the emission wavelength of LED are 442 nm, 460 nm and 520 nm. The combination of various deposition techniques such as thermal annealing, PSD and electron beam enable to avoid the use of expensive and high temperature techniques when compared to commonly used MOCVD. Hence, these understanding are beneficial to the development of low-cost production and commercial applications for future LED and optoelectronic devices.

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