INFRARED EMISSION COMPARISON BETWEEN HALF-STACK AND FULL-STACK ZnS:REF$_3$ ACTFEL DEVICES

By

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INFRARED EMISSION COMPARISON BETWEEN HALF-STACK AND FULL-STACK ZnS:REF₃ ACTFEL DEVICES

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Major Department:  Materials Science and Engineering

The effects of a single dielectric layer (half-stack) versus both a top and bottom dielectric layer (full-stack) on the electroluminescent (EL) spectra and brightness of ZnS:ErF₃ and ZnS:TmF₃ alternating current thin film electroluminescent (ACTFEL) devices have been studied. The doped ZnS thin films are radio frequency (RF) planar magnetron sputter deposited onto a Corning 7059 glass substrate pre-coated with an indium tin oxide (ITO) transparent conducting electrode and an aluminum titanium oxide (ATO) dielectric layer. ZnS:ErF₃ films were tested for EL properties in the as deposited or annealed (425°C, 1 hour, nitrogen) conditions. For ZnS:ErF₃ ACTFEL devices, near infrared signals at ~1550nm and ~985nm are observed. In addition, another three peaks in the visible region (525nm, 550nm and 660nm) are observed. For ZnS:TmF₃ ACTFEL devices, peaks at ~790nm and ~480nm are observed. Half-stack devices are shown to exhibit lower turn-on voltages and higher infrared intensities. For ZnS:ErF₃ ACTFEL devices, the turn-on voltages are 180V and 220V in the as deposited condition, versus
150V and 190V in the annealed condition for half-stack compared to full-stack devices, respectively. The \( B_{40} \) (brightness at 40V above EL threshold) at 1550nm is 0.28 \( \mu W/cm^2nm \) and 0.02 \( \mu W/cm^2nm \) in the as deposited condition, and 0.38 \( \mu W/cm^2nm \) and 0.04 \( \mu W/cm^2nm \) in the annealed condition for half-stack compared to full-stack devices, respectively. For ZnS:TmF\(_3\) ACTFEL devices, the turn-on voltage is 130V for half-stack compared to 170V for full-stack devices, and the \( B_{40} \) at 790nm is 0.006 \( \mu W/cm^2nm \) and 0.0017 \( \mu W/cm^2nm \) for half-stack and full-stack devices, respectively. Half-stack devices also show a higher infrared/visible peak height ratio. For ZnS:ErF\(_3\) ACTFEL devices, the ratio of 1550nm/550nm peak intensities is 9 and 0.5 in the as deposited condition, versus 25 and 4 in the annealed condition for half-stack and full-stack devices, respectively. For ZnS:TmF\(_3\) ACTFEL devices, the peak height ratio of 790nm/480nm is 0.006 for half-stack compared to 0.003 for full-stack devices. The external conduction and internal charges for half-stack devices were 60% to 90% larger than for full stack devices, suggesting a correlation with the improved brightness. Devices annealed at 425°C also showed an improved infrared intensity, a higher infrared/visible ratio and lower turn-on voltages. Again increased external conduction and internal charges in the annealed device correlated with the improved brightness.
Though the standard cathode ray tubes (CRTs) have been and remain the dominant color displays, more and more attention has been turned towards the development of flat panel display (FPD) such as field emission displays (FEDs), plasma displays (PDs), liquid crystal displays (LCDs), and electroluminescent displays (ELDs). The 1992 FPD market was dominated by LCDs (91%), while PDs (7%) and ELDs (2%) competed only in niche markets. Though LCDs (85%) were still projected to dominate the overall market in 2003, ELDs are expected to grow to 8% while PDs should remain at 7% [1].

The FPD technologies can be classified into two categories. One is non-emissive, of which LCDs are typical examples. However, LCDs have many inherent problems due to their organic nature, such as limited temperature tolerance, limited viewing angle and insufficient contrast, etc. The other is emissive displays, which include FED, PD and ELD. Because of their solid-state instead of liquid-state constructions, these devices have a much wider viewing angle, higher contrast, larger operation temperature range, better ruggedness and longer life span than non-emissive displays. Of these, ELDs are the most prevalent displays [2]. More specifically, thin film EL displays have several qualities that make them particularly attractive for small to midsize flat panel displays. A wider viewing angle (>80°), broader operating temperatures (-25°C~60°C), higher contrast, being more shock and vibration resistant make thin film EL displays the longest lasting, most reliable flat panel display technology on the market by now.
The focus of this research is the thin film electroluminescence display device driven by an alternating current field, i.e., an alternating current thin film electroluminescence device (ACTFELD). The literature review of ACTFELD will be given in Chapter 2. Chapter 3 will present the experimental procedure and sample preparation. The objective of this study is to compare the infrared emission between half-stack and full-stack devices, in which the phosphor layer is ZnS doped with rare earth fluorides. Data are given in Chapter 4. In Chapter 5 conclusions will be presented.
CHAPTER 2
LITERATURE REVIEW

2.1 History of Electroluminescence

Electroluminescence (EL), by definition, is a phenomenon that converts electrical energy to luminous energy without thermal energy generation. Though the first report of electroluminescence is often attributed to a famous paper by Destriau in 1936, the first observations of the effect of electric fields on luminescence were reported by Gudden and Pohl in 1920 [3]. They found that the luminescent intensity increases as an electric field of several KV/cm is applied to a ZnS:Cu phosphor which is insulated from the electrodes. George Destriau in 1936 observed light emission from a suspension consisting of ZnS:Cu and oil [4].

Afterwards, the development of EL experienced a relatively silent period until the 1950s when GTE Sylvania received a patent for an EL powder lamp. However, the devices were limited to powder phosphors embedded in a dielectric matrix. In the 1950s and 1960s more attention is drawn to how to improve EL efficiency reaching or even exceeding the 15 lm/W of the existing incandescent lamp. However, problems in these devices, such as low light output, short operating lifetime, and a rapid deterioration when operated above room temperature led to the virtual abandonment of powder EL studies.

Fortunately, great progress was made in thin film technology in the 1960s, which helped Soxman and Ketchpel fabricate thin film devices. In 1961, Soxman et al. [5] successfully discovered AC thin film electroluminescence using a device containing a ZnS:Mn phosphor with a dielectric. These devices had acceptable lifetimes (several
thousand hours) and multiplexing capabilities, but failed to achieve acceptable reliability.

A milestone in thin film EL (TFEL) devices was achieved by Inoguchi et al (Sharp Corporation) who introduced an ac thin-film approach in 1974 [6, 7]. The team of researchers at Sharp fabricated a device modeled after a capacitor that consisted of manganese-activated zinc sulfide as the phosphor and yttrium oxide for the sandwiching insulators. This was the first high-brightness, long-lifetime device. Sharp succeeded in developing this technology and introduced a monochrome television display in 1978 [8]. Planar systems introduced multicolor and full-color displays in 1982 [9] and 1994 [10], respectively. Thus, today thin film electroluminescent displays are very active in the market place. Major research interest will be focused on improving device brightness, stability and efficiency.

2.2 ACTFEL Device Structure

2.2.1 Monochrome Structure

The most common structure used for alternating current thin film electroluminescent (ACTFEL) devices is illustrated in Fig 2.1(a) [11]. It is also called a MISIM (metal-insulator-semiconductor-insulator-metal) structure. This device usually uses glass as a transparent substrate on which a transparent bottom electrode is deposited. The following step is the deposition of a transparent insulator, the phosphor layer, another insulator layer, and an opaque electrode. Because the substrate is transparent glass, the light can be transmitted through the glass into the eyes of the viewer. Also, glass is cheap and widely available in any size. In addition, it does not contain alkali metals which have been found to diffuse into the semiconducting phosphor layer and cause the devices to degrade over time [12].
Another structure that has been developed is known as the “inverted” structure which is shown in Fig 2.1(b). In this case, the substrate is opaque (e.g., silicon) and therefore a transparent electrode is required on the top of the ACTFEL structure [13] through which light leaves the device toward the viewer. Because there is no requirement for the substrate to be transparent in the inverted structure, ceramics can be used for the substrate materials, which allows higher temperature processing.

The two kinds of devices mentioned above are “full-stack” devices. However, there is another type of device which is known as a “half-stack” device. This type of device is similar to either a standard or inverted device, but lacks one of the two insulating layers. It’s shown in Fig 2.2. Because it lacks the top insulator, the device processing will be simpler. This type of device is normally used for research purposes due to lower reliability.

2.2.2 Full-color Structure

There are several approaches to the development of full color EL displays. Two common approaches will be discussed here, which are patterned-phosphor structure and “color-by-white” filtered structure.

For patterned-phosphor structure, also called a red-green-blue display, the red, green, and blue phosphor layers are patterned so that each pixel is divided up into four sub-pixels that contain a red, green and two blue phosphor regions (Fig 2.3(a)). The advantages of this structure are a simpler drive structure, being identical to that of a monochrome panel and the use of aluminum electrodes for self-healing breakdown [12]. However, because it will require masking and etching, the processing is more complicated.
Fig 2.1 Schematic diagram of (a) typical structure of MISIM, (b) inversed structure of ACTFEL.
Fig 2.2 Schematic diagram of half-stack structure.
In the color-by-white approach, each pixel has a phosphor layer that produces white light that is subsequently filtered by a red, green, or blue filter (Fig 2.3(b)). Since only one phosphor layer is used, the processing is simple. However, as the phosphor layer is filtered, there will be a loss in efficiency.

There are also other approaches to the development of full color displays. Interested readers are referred to Ono [12].

2.3 Device Physics

The processes leading to EL (Fig 2.4) from a device can be divided into four steps, namely:

1. As the applied voltage is raised above the threshold voltage, electrons are tunnel injected into the conduction band from electronic states at the insulator-phosphor interface.

2. Due to the large electric field, these electrons are accelerated to ballistic energies in the phosphor layer. They are called “hot electrons.”

3. The hot electrons with sufficient energy impact excite the electrons in the luminescent centers in the phosphor layer.

4. The excited electrons of the luminescent centers relaxed back to the ground state by radiative recombination and thus generate photons.

2.3.1 Tunnel Emission

To understand tunnel emission, one must consider two questions: One is the source of hot electrons; the other is the mechanism of electron injection. In ACTFEL device, the origin of the hot electrons is generally defect states at the insulator/phosphor interface. Experimental observations have shown that the threshold voltage is a function of the polarity of the applied voltage, so devices have demonstrated asymmetric currents.
Fig 2.3 Schematic structure of (a) patterned phosphor structure, (b) “color-by-white” filtered structure [12].
when different top and bottom insulators are used [14]. As for the mechanism of electron injection, the accepted theory is thermally assisted tunneling. The device current is a strong function of the phosphor field, which suggests the tunneling mechanism dominates. Moreover, experimental observations have shown that there is a slight temperature dependence on the ACTFEL device, which suggests that the tunneling mechanism is thermally assisted [15]. Finally, the fact that the electrical properties are only a weak function of temperature rules out a strict thermionic emission process. In summary, electrons trapped at the phosphor/insulator interface are the carrier source, and a thermally assisted tunneling mechanism is responsible for their injection.

The trap depth and the density of energy states at the interface will affect the device performance. As the applied voltage reaches the threshold voltage, the energy bands are bent enough that electrons tunnel into the phosphor conduction band from interface states. The tunneling emission current, $J$, for Shottky barriers is given by [16]

$$J \sim E^2 \exp \left( \frac{-8\pi\sqrt{2m^*}(q\Phi_B)^{3/2}}{3qhE} \right)$$

where $E$ is the electric field, $m$ is the effective electron mass, $q$ is the charge on an electron, $\Phi_B$ is the barrier height, and $h$ is Planck’s height. For interface trap emission, this equation remains the same except that the barrier height term is replaced with a term that represents the interface trap depth. The trap depth controls how much energy is required to tunnel electrons out of the interface states and into the conduction band of the phosphor. If the trap depth is too shallow, the electrons will be injected out of the interface at a low electric field and will not be accelerated to the high energy required to cause luminescence. However, if the trap depth is too deep, it will take too high an
Fig 2.4 Four main steps of electroluminescence [1].
electric field to inject electrons. The empirical optimum electron trap depth is 1.0−1.3 eV [15].

The density of states at the interface will determine the current injected into the ACTFEL device. A high density of states is desirable for ACTFEL device. Studies by Bardeen [17] and later by Kurtin et al. [18] revealed that surface and interface state properties are dependent on the covalency/ionicity of the semiconductors bonds. Rack et al. [19, 20] also investigated interfacial layers to increase electron injection in \( \text{Ca}_x\text{Sr}_{1-x}\text{Ga}_2\text{S}_4:\text{Ce} \) blue phosphors.

2.3.2 Electron Acceleration

When electrons tunnel into the phosphor layer conduction band, they are accelerated due to the electric field in the phosphor layer and may impact the luminescent centers. During transport in the host, electrons scatter and lose energy by several mechanisms [21-24]. There are two main approaches to understand these mechanisms, namely Monte Carlo simulation and lucky-drift model.

The basic idea of a Monte Carlo simulation is to calculate the electron energy distribution in ACTFEL device by describing the carrier dynamics and kinetics as a function of electric field. In 1988, Brennan [22] performed a full band structure calculation for fields up to 1 MV cm\(^{-1}\) in ZnS. However, his simulation concluded that very few electrons have sufficient energy to excite Mn luminescent centers, which is not consistent with the high luminescence of ZnS:Mn devices. Later, Mach et al. [23] simulated the high field transport properties in ZnS based on the assumption of a parabolic conduction band and a polar optical phonon scattering mechanism. Their simulation indicated nearly loss-free electron transport, which resulted in a very energetic electron energy distribution. Bhattacharyya et al. [21] implemented a more realistic
simulation, where they assumed a non-parabolic conduction band and considered scattering due to polar optical phonons, acoustic phonons, intervalley scattering, and ionized and neutral impurities.

The basic idea of the lucky-drift model is to describe the high field transport properties using the electron mean free path. This approach was originally put forward by Baraff [25], whose theory consists of the two modes. One is the ballistic mode, which occurs before the electron’s first collision. The other is the drift mode, which occurs after the electron has had one collision. The lucky-drift model is a more simplistic analytical model which has made significant contributions to understanding the high field electron transport phenomenon in an ACTFEL device.

2.3.3 Impact Excitation and Ionization

Once the injected electrons accelerate to high enough energy, electrons in the luminescent center will be excited out of their ground states by impact from an injected electron. There are two main mechanisms in ACTFEL device. One is impact excitation [24], in which the electron is promoted into a higher energy atomic state of the luminescent center. The probability of this process is related to the impact cross section of the luminescent center. When the electron experienced impact excitation, it can relax back into its ground state either radiatively or non-radiatively. Impact excitation is desirable in ACTFEL device. Another mechanism is impact ionization [26], in which the electron is promoted into the conduction band of the host material and then accelerated toward the anode by the electric field. Since the exited electron is not localized on the luminescent center, radiative relaxation may be more difficult. In addition, too many electrons in the conduction band may cause avalanche breakdown of the phosphor film, therefore impact ionization is not desired in ACTFEL device.
2.3.4 Radiative Recombination

As mentioned in 2.3.3, once the electron experienced impact excitation, it can relax back into its ground state either radiatively (which produces a photon) or non-radiatively (which generates phonons). For ACTFEL devices, it’s necessary to maximize the former and to minimize the latter, since phonons are vibrations in the phosphor lattice and are deleterious to the radiative efficiency.

In most ACTFEL devices, the radiative mechanism is an atomic transition, which means that the atomic transition energy is dominated by the elemental or free ion quantum properties of the luminescent center. This event can occur by either intershell or intrashell transitions depending on the quantum numbers of the initial and final state.

Generally speaking, there are two selection rules which determine whether an optical transition is allowed. One is called the Laporte selection rule, which states that when the change in the angular momentum quantum number, $\Delta l$, is $\pm 1$, an electronic dipole transition is allowed. Another is the spin selection rule, which states that when the change of the spin quantum number of the electron, $\Delta s$, is 0, optical transitions are allowed. Though these selection rules are generally followed, there are several mechanisms that relax these selection rules and cause otherwise forbidden transitions to be observed. For example, when asymmetry of the lattice exists (for example, ZnS sites are tetrahedral), Laporte-forbidden transitions can be observed.

2.4 Device Characterization

A simple model which accurately describes most of the ACTFEL device characteristics is shown in Fig 2.5. In this model, the insulators are represented by perfect capacitors and the phosphor layer is represented by a capacitor below threshold and a pair of back-to-back Zener diodes above threshold [27]. Below threshold, the device is just a
circuit with three capacitors in series, two from the insulators and one from the phosphor. The voltage drop across each layer is proportional to the dielectric constant and the layer thickness. The electrical threshold voltage is the voltage at which the phosphor layer experiences a Zener breakdown as electrons are injected by tunneling into the phosphor layer. Above threshold, the phosphor layer becomes two Zener diodes back to back.

A typical trapezoidal waveform that is used to drive an ACTFEL device is shown in Fig 2.6. The device is driven at 2.5 kHz with 5 µs rise and fall times and a 30 µs pulse width [28].

2.4.1 Charge-Voltage (Q-V) Diagram

Charge-Voltage data are very useful for electrical characterization of an ACTFEL device. A typical Charge-Voltage diagram is shown in Fig 2.7 [29]. The description of the diagram is as following:

1. Initially, at zero voltage there is zero charge.
2. As the voltage is ramped below threshold, the slope of Q-V is equal to the capacitance of the phosphor and insulators.
3. Above threshold, the slope is equal to the capacitance of the two insulators only since the phosphor layer breaks down.
4. As the voltage is reduced below threshold, the slope again becomes the capacitance of the phosphor and insulators. However, at zero voltage the charge is non-zero because of the trapped charge at the phosphor/insulator interface.

2.4.2 Brightness-Voltage (B-V) Curve

Brightness-voltage data are the most important measurement used to evaluate an ACTFEL device. A typical B-V curve is shown in Fig 2.8. From this curve, several important ACTFEL parameters can be obtained.
Fig 2.5 Equivalent circuit model of an ACTFEL device [27].
Fig 2.6 A typical waveform used to drive ACTFEL device.
1. The EL threshold voltage $V_{th}$ is the intercept at $B=0$ of the maximum slope.

2. The $B_{40}$ value is the brightness at 40V above the threshold voltage.

3. The CIE coordinates $x$, $y$ and $z$ [30] characterize the color of light emitted by the phosphor. Usually, only $x$ and $y$ values are recorded since the $z$ value is given by

$$x + y + z = 1$$

Fig 2.9 shows the CIE diagram. The CIE coordinates are determined by the extent to which emission spectra overlaps each of the tri-stimulus functions [30]. By plotting the CIE coordinates of the different phosphor materials, the range of colors can be determined.

4. The luminous efficiency can be calculated simply by [1]:

$$\eta = \frac{B \pi}{P}$$

where $B$ is the brightness or luminance (cd/m$^2$), $P$ is the power density (W/m$^2$). Since 60 Hz and $V_{th} + 40$ V are common device driving conditions, a simple way to determine the luminous efficiency is to get the $B_{40}$ from the B-V curve, and then get the power at $V_{th} + 40$ V from the Q-V diagram.

2.5 Material Requirements

2.5.1 Substrates

The basic requirements for the substrate of the standard ACTFEL device are that it be transparent and smooth. Normally, Corning 7059 boro-silicate glass [31] is used. However, the softening temperature of Corning 7059 glass is only 598°C [1], which sometimes can not meet the high temperature annealing requirement. In this case, high temperature glass ceramics are used. They are much more expensive than glass and it is reported that the resolution is only about 100µm, versus a resolution on glass of 25µm [2].
Fig 2.7 A typical idealized charge-voltage diagram [29].
2.5.2 Electrodes

Top and bottom electrodes, one of which must be transparent, are required for ACTFEL device. For the standard device, the first layer deposited on the glass substrate is the transparent electrode. The requirements of the electrode are that they are conductive so that it will not affect the RC time constant of the device and transparent so that it can minimize the absorption. Additionally, it must withstand the thermal processing of subsequent layers. The most common transparent electrode is indium tin oxide (ITO), whose resistivity is about $5\,\Omega/\square$ for thickness that range from 300~500 nm. The high conductivity comes from the high concentration of shallow donors located only few meV below the conduction band. These donors can be thermally activated into the conduction band at room temperature. Sn$^{+4}$ sitting substitutionally on an In$^{+3}$ site and oxygen vacancies [32] plus intentionally doped F ions are the primary sources of these donors. Other candidates for transparent electrodes are ZnO and ZnO:( Al, In, Ga) [33].

The top electrode is the last layer that is deposited for the standard ACTFEL device. The main requirement for it is that it has low resistivity and can be resistant to electromigration under high electric fields [34]. The most common top electrode is aluminum due to its good conductivity, resistance to ion migration and good processibility. In addition, because aluminum is highly reflective, it enhances brightness but reduces the contrast in a display.

2.5.3 Insulators

The main purpose of the insulator layer in ACTFEL device is to prevent current flow through the device and protect the phosphor layer from electric breakdown from fields of up to 2 MV/cm. Because of the important role of the insulators, the requirements
Fig 2.8 A typical B-V plot.
Fig 2.9 Schematic diagram of the CIE diagram [30].
for them are rather stringent. However, the most important requirements for them are:

1. High dielectric constant

   Below threshold, the applied voltage is capacitively divided by the phosphor and the insulators as following

   \[ V_p = \frac{C_I}{C_I + C_P} V_a \]

   \[ V_I = \frac{C_P}{C_I + C_P} V_a \]

   where \( V_a \) is the applied voltage and \( V_I \) and \( V_P \) are the voltages across the insulators and the phosphor layer, respectively. \( C_I \) and \( C_P \) are the insulator and phosphor capacitance, respectively.

   To maximize the portion of the applied voltage dropped across the phosphor layer, the capacitance of the insulators should be maximized while that of the phosphor layer should be minimized. To optimize the insulator capacitance, either the dielectric constant should be maximized or the layer thickness should be minimized. Since the layer thickness can’t be set too low due to processing and breakdown issues, a high dielectric constant is desirable.

2. High electric field strength

   Because of the clamp effect, excessive voltage above the threshold voltage will drop across the insulator layer. So, high electric field strength of the material is required to avoid dielectric breakdown.

   However, it should be noted that as the dielectric constant increases, the electric field strength decreases because these properties are usually inversely proportional to each other. A figure of merit for insulator materials was introduced by Howard [35]. The
figure of merit is the product of the dielectric constant and the electric field strength of the insulator. Figures of merit for several insulators [36] are listed in table 2.1.

Additionally, a uniform thickness and a pinhole-free layer are also important for the insulators. These are process dependent.

2.5.4 Phosphor

The phosphor layer is the light emitting material which is sandwiched between two insulators. This material consists of two constituents: the host material which dominates the electrical properties of the phosphor layer, and the luminescent center which dominates the optical properties of the phosphor layer. Since the radiative recombination event in the phosphor layer is an atomic transition which occurs locally at the luminescent center, the host materials and the luminescent centers must satisfy certain requirements.

2.5.4.1 Host Materials

One important requirement that the host material must satisfy is that the bandgap must be larger than the energy for photon generation. For blue emission, this bandgap must be at least 3.1 eV to avoid absorption. Another requirement is that the host material must have a high dielectric strength, usually greater than 1 MeV/cm. This is because the host must allow for efficient acceleration of electrons. After electrons are injected into the phosphor, they must be accelerated to sufficiently high energy states to impact excite the luminescent centers. In other words, the host material controls the “turn-on” properties of the ACTFEL device. In addition, the host lattice should have good crystallinity and minimum lattice defects. Post-annealing is widely used to decrease the defect density in the host lattice.
The common host materials are sulfur-based compounds, such as ZnS, CaS, SrS and SrGa2S4 [1, 37]. Of these materials, ZnS is by far the most widely used for ACTFEL device. ZnS has a bandgap of 3.8 eV [38] which makes it transparent to visible light and the dielectric breakdown strength of ZnS is about 1 MeV/cm which allows the electrons to reach high enough energies. A summary of the properties of the sulfides are listed in table 2.2.

2.5.4.2 Luminescent Centers

Since the radiative recombination event in the phosphor is an atomic transition which occurs locally at the luminescent center, many efficient cathodoluminescent (CL) phosphors, such as ZnS:Cu,Al,Ag and ZnS:Ag,Cl, are shown to be poor EL phosphors because donor-acceptor recombination is inefficient under high electric fields. Transition and rare earth elements are most commonly used as luminescent centers.

One important aspect for luminescent centers is the cross section for excitation. To a first approximation, the cross section for excitation is proportional to the geometric diameter of the luminescent center ion. Since the power efficiency of the device is directly proportional to the cross section of the luminescent center by [39]:

$$\eta = \frac{h \nu \sigma N}{eE}$$

where $h \nu$ is the photon energy, $\sigma$ is the excitation cross section, $N$ is the optimum luminescent center concentration, $e$ is the charge of an electron, and $E$ is the electric field, it’s clear that a large cross section is desirable for ACTFEL device.

Another important aspect for luminescent centers is the radiative transition energy which dictates the color of the phosphor. The atomic transition energy is dominated by
the elemental or free ion quantum properties of the luminescent center and the host material has an effect on photon energy of the transition elements [40].

2.6 ZnS:ReF₃ as ACTFEL Phosphor

The electroluminescence studies of zinc sulphide doped with various rare earths were initiated in the late 1960s and several colors were observed from the ZnS:ReF₃ thin films [41,42]. Now, rare-earth activated ZnS phosphors have attracted attention as suitable candidates for full color ac thin film electroluminescent displays [43, 44]. Though a lot of work has been done of understanding visible emission, infrared emission has been ignored. In this research, particular attention will be given to the infrared emission of phosphors because they have wide applications in industry, such as developing night vision equipment like helmet mounted cameras, goggles for friend/foe identification, transmitting signals for fiber optical communication and handheld remote controls, to name just a few.

2.6.1 ZnS

As we have mentioned before, ZnS is by far the most widely studied and also the most commonly used host material for commercial ACTFEL displays due to its unique physical nature and excellent optical properties.

Physically, ZnS lattice sites are tetrahedrally coordinated. Since Laporte-forbidden transitions can be relaxed when the atom or ion is placed in a site without a center of symmetry, the tetrahedral nature of ZnS makes the brightest red and green ACTFEL phosphors efficient.

Optically, ZnS has a bandgap of 3.8 eV. It is therefore transparent throughout the visible range and optical absorption in the phosphor layer is minimal. In addition, high dielectric breakdown strength (1 MeV/cm) allows efficient excitation of the luminescent centers.
The data suggest that ZnS promotes a high density of interface states which are deep enough to result in an exceptional turn-on character [45].

Table 2.1 List of the insulators used in ACTFEL devices with values for the properties of interest.

<table>
<thead>
<tr>
<th>Insulator</th>
<th>Deposition Method</th>
<th>$\varepsilon_r$</th>
<th>$F_{BD}$ (MV/cm)</th>
<th>$\varepsilon_0\varepsilon_r F_{BD}$ (µC/cm²)</th>
<th>Breakdown Mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$</td>
<td>Sputtering</td>
<td>4</td>
<td>6</td>
<td>2</td>
<td>Self Healing</td>
</tr>
<tr>
<td>SiO$_x$N$_y$</td>
<td>Sputtering</td>
<td>6</td>
<td>7</td>
<td>4</td>
<td>Self Healing</td>
</tr>
<tr>
<td>SiO$_x$N$_y$</td>
<td>PCVD</td>
<td>6</td>
<td>7</td>
<td>4</td>
<td>Self Healing</td>
</tr>
<tr>
<td>Si$_3$N$_4$</td>
<td>Sputtering</td>
<td>8</td>
<td>6–8</td>
<td>4–6</td>
<td>Self Healing</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>ALE</td>
<td>8</td>
<td>8</td>
<td>6</td>
<td>Self Healing</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>Sputtering</td>
<td>8</td>
<td>5</td>
<td>3.5</td>
<td>Self Healing</td>
</tr>
<tr>
<td>SiAlON</td>
<td>Sputtering</td>
<td>8</td>
<td>8–9</td>
<td>4–6</td>
<td>Self Healing</td>
</tr>
<tr>
<td>Y$_2$O$_3$</td>
<td>Sputtering</td>
<td>12</td>
<td>3–5</td>
<td>3–5</td>
<td>Self Healing</td>
</tr>
<tr>
<td>Y$_2$O$_3$</td>
<td>EBE</td>
<td>12</td>
<td>3–5</td>
<td>3–5</td>
<td>Self Healing</td>
</tr>
<tr>
<td>BaTiO$_3$</td>
<td>Sputtering</td>
<td>14</td>
<td>3.3</td>
<td>4</td>
<td>Self Healing</td>
</tr>
<tr>
<td>Sm$_2$O$_3$</td>
<td>EBE</td>
<td>15</td>
<td>2–4</td>
<td>3–5</td>
<td>Self Healing</td>
</tr>
<tr>
<td>HfO$_2$</td>
<td>Sputtering</td>
<td>16</td>
<td>0.17–4</td>
<td>0.3–6</td>
<td>Self Healing</td>
</tr>
<tr>
<td>Ta$_2$O$_5$-TiO$_2$</td>
<td>ALE</td>
<td>20</td>
<td>7</td>
<td>12</td>
<td>Self Healing</td>
</tr>
<tr>
<td>BaTa$_2$O$_6$</td>
<td>Sputtering</td>
<td>22</td>
<td>3.5</td>
<td>7</td>
<td>Self Healing</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>ALE</td>
<td>60</td>
<td>0.2</td>
<td>1</td>
<td>Propagating</td>
</tr>
<tr>
<td>Sr(Zr,Ti)O$_3$</td>
<td>Sputtering</td>
<td>100</td>
<td>3</td>
<td>26</td>
<td>Propagating</td>
</tr>
<tr>
<td>SrTiO$_3$</td>
<td>Sputtering</td>
<td>140</td>
<td>1.5–2</td>
<td>19–25</td>
<td>Propagating</td>
</tr>
</tbody>
</table>

*Self healing: when breakdown initiates, the material can inherently isolate the localized breakdown and prevent catastrophic breakdown [36].
Table 2.2 Phosphor materials and properties.

<table>
<thead>
<tr>
<th>Phosphor Material</th>
<th>Emission Color</th>
<th>Luminance (Cd/m²) @60Hz</th>
<th>Luminous Efficiency (lm/W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZnS:Mn</td>
<td>Yellow</td>
<td>300</td>
<td>2~4</td>
</tr>
<tr>
<td>ZnS:Sm,F</td>
<td>Reddish-Orange</td>
<td>8</td>
<td>0.05</td>
</tr>
<tr>
<td>ZnS:Sm,Cl</td>
<td>Red</td>
<td>12</td>
<td>0.08</td>
</tr>
<tr>
<td>ZnS:Mn/Filter</td>
<td>Red</td>
<td>75</td>
<td>0.8</td>
</tr>
<tr>
<td>CaS:Eu</td>
<td>Red</td>
<td>12</td>
<td>0.05</td>
</tr>
<tr>
<td>CaS:Eu,F,Se</td>
<td>Red</td>
<td>_</td>
<td>0.7</td>
</tr>
<tr>
<td>ZnS:Tb,F</td>
<td>Green</td>
<td>125</td>
<td>1.3</td>
</tr>
<tr>
<td>ZnS:Mn/Filter</td>
<td>Yellow-Green</td>
<td>80</td>
<td>_</td>
</tr>
<tr>
<td>CaS:Ce</td>
<td>Green</td>
<td>10</td>
<td>0.1</td>
</tr>
<tr>
<td>ZnS:Tm,F</td>
<td>Blue</td>
<td>&lt;1</td>
<td>&lt;0.1</td>
</tr>
<tr>
<td>SrS:Ce</td>
<td>Blue-Green</td>
<td>65</td>
<td>0.44</td>
</tr>
<tr>
<td>ZnS/SrS:Ce/Filter</td>
<td>Greenish-Blue</td>
<td>14</td>
<td>0.20</td>
</tr>
<tr>
<td>ZnS/SrS:Ce</td>
<td>Bluish-Green</td>
<td>96</td>
<td>1.3</td>
</tr>
<tr>
<td>SrS:Ce,F,Ga</td>
<td>Blue-Green</td>
<td>110</td>
<td>_</td>
</tr>
<tr>
<td>CaGa₂S₄:Ce</td>
<td>Blue</td>
<td>13</td>
<td>_</td>
</tr>
<tr>
<td>SrS:Ce,Eu</td>
<td>Eggshell-White</td>
<td>32</td>
<td>0.4</td>
</tr>
<tr>
<td>SrS:Ce/CaS:Eu</td>
<td>Paper-White</td>
<td>17</td>
<td>_</td>
</tr>
<tr>
<td>ZnS:Mn/SrS:Ce</td>
<td>Yellowish-White</td>
<td>450</td>
<td>1.60</td>
</tr>
</tbody>
</table>

[37]
2.6.2 Rare-Earth Ions

Rare-earth ions, also referred to as the Lanthanides, play an important role in much of modern optical technology as the active constituents of materials. There are an amazing number of applications for these rare-earth-activated optical materials and much of today’s cutting-edge optical technology and future innovations rely on their unique properties.

The lanthanide series have an electron configuration of: 1s\(^2\) 2s\(^2\) 2p\(^6\) 3s\(^2\) 3p\(^6\) 4s\(^2\) 3d\(^{10}\) 4p\(^6\) 5s\(^2\) 4d\(^{10}\) 5p\(^6\) 6s\(^2\) 4f\(^x\). First, it can easily be shown that f shells of rare-earth ions are only partially filled. These partially filled shells of f electrons give rise to spectrally narrow localized electronic transitions that occur at wavelengths ranging from the far-infrared to the vacuum-ultraviolet and provide the basis for optical technology in which light may dynamically interact with a material that contains these ions. The qualitative energy level schemes and emission lines for various rare earth elements, such as Nd, Er and Tm (Tm and Er are used in this research) are shown in Fig 2.10 [50].

Secondly, it can also be shown from the electron configuration that the 4f levels of rare-earth ions are shielded by the 5s and 5p electrons so that the 4f electrons remain highly localized to the ion and their optical transitions maintain much of an atomic-like character. This characteristic of the rare-earth ions arises from the unique situation in which the lowest-energy electrons are not spatially the outermost electrons of the ion, and thus have a limited direct interaction with the ion’s environment. Fig 2.11 shows the radial positions of the outer orbital of the Ce\(^{3+}\) ion. The “shielding” of the 4f electrons from the environment by the outer filled shells of 5s and 5p electrons prevents the 4f electrons from directly participating in bonding and allows them to maintain much of the
character exhibited by a free-ion. These 4f levels are subsequently unaffected by the host lattice.

Chase et al. [47] investigated the EL emission of the device with rare earth fluorides in the phosphor layer. For ZnS:ErF$_3$, the device has a faint green emission which results from the transitions $^2\text{H}_{11/2} \rightarrow ^4\text{I}_{15/2}$ and $^4\text{S}_{3/2} \rightarrow ^4\text{I}_{15/2}$. Fig 2.12 shows the complete spectrum. About 10 states are observed to fluoresce.

For ZnS:TmF$_3$, weak emission in the blue and the red is observed (Fig 2.13). The emissions are believed to originate from the $^1\text{G}_4$ state.

2.6.3 Charge Compensation

Earlier works [46] have noted that samples doped with Re metals show only spotty luminescence, whereas halide coactivation (F$^-$, Cl$^-$) gives uniform emission over the entire surface. Moreover, it’s difficult to introduce rare earth ions substitutionally into ZnS with relatively high concentration because of the mismatch in ion size and charge valence between Zn$^{2+}$ and RE$^{3+}$ ions. Chase et al. [47] proposed a new type of emission center which introduced rare earth fluoride in ZnS. Studies by Jayaraj et al. [48] on the effect of halides (F$^-$, Cl$^-$, Br$^-$) and oxide (O$_2^-$) on the EL emission spectra of ZnS:Pr TFEL devices show that the fluoride dopant produces the maximum brightness. In this study, the ZnS film is doped with fluorine for charge compensation.
Fig 2.10 Energy level scheme for different rare earths [50].
Fig 2.11 Schematic diagram of the Ce$^{3+}$ ion which shows the radial positions of the outer orbitals [1].
Fig 2.12 Spectrum of ZnS:ErF$_3$ over a) the visible, and b) the near-infrared region [47].
Fig 2.13 Spectrum of ZnS:TmF₃ over a) visible and b) near-infrared region [47].
CHAPTER 3
EXPERIMENTAL PROCEDURE

3.1 Introduction

In this chapter, the procedures for fabricating half-stack and full-stack ZnS:REF\textsubscript{3} thin film devices will be described. Characterization methods, including electrical and optical characterization, will also be discussed.

3.2 Device Fabrication

3.2.1 Substrates

The substrates are 7059 glass coated with transparent conductor indium tin oxide (ITO) and insulator aluminum titanium oxide (ATO) deposited using atomic layer deposition (ALD) by Planar Systems, Inc. Before phosphor layer deposition, the substrates were cleaned with methanol followed by 6 minute ozone clean.

3.2.2 Phosphor Film Deposition

The ZnS:REF\textsubscript{3} layers (RE=rare earth, ErF\textsubscript{3}, TmF\textsubscript{3} were used in this study) were deposited by RF magnetron sputtering using independent 2” (5 cm) ZnS and REF\textsubscript{3} sources. The ZnS target was bulk polycrystalline ZnS purchased from Morton Thiokol, Inc., and the REF\textsubscript{3} targets were purchased from Target Materials, Inc. The sputtering system is designed to simultaneously operate 3 RF-sputtering guns which make it possible to co-sputter elements and compounds from separate targets. In this study, the concentration of rare earth was controlled by the power and duty cycle of the ZnS:REF\textsubscript{3} versus pure ZnS source.
The sputter system allowed as many as seven $1\times2$" substrates and one bare glass substrate of the same size to be processed simultaneously. The bare glass substrate was used in each run to measure the film thickness by stylus profilometry so that 1 µm thickness of phosphor film could be maintained. The substrates were preheated by a carbon heater and rotated at 7 rpm by a stepping motor during deposition to enhance uniformity. The load-lock chamber was pumped down to 10 mTorr by a wet rotary piston roughing pump (Leybold Trivac D65B). The base pressure of the system was approximately $10^{-6}$ Torr and it’s achieved by a turbomolecular pump (Leybold Trubovac 450). The working pressure was raised to 20 mTorr by flowing argon, controlled by the gas flow rate and a throttle valve, which is installed between the main chamber and turbomolecular pump. There are also several gauges which are used to measure vacuum pressure. The thermocouple gauges were used to measure the pressure of the load-lock chamber, the ionization gauge was used to measure the base pressure of the main chamber, and the capacitance manometer was used to monitor working gas pressure during deposition.

Performance of EL device is influenced by deposition conditions, including substrate temperature, deposition time, concentration of doping elements, work pressure, etc. Conditions for phosphor deposition were evaluated and optimized by measuring the brightness at 40V above threshold of half-stack devices. In a separate study [49], it was shown (Fig 3.1 and Fig 3.2) that for ErF$_3$ and TmF$_3$, the best processing conditions are as shown in Table 3.1.
Table 3.1 Optimized processing conditions for ZnS:REF$_3$ films.

<table>
<thead>
<tr>
<th>Film</th>
<th>Temperature (°C)</th>
<th>Work Pressure (mTorr)</th>
<th>Power and Duty Cycle (ZnS)</th>
<th>Power and Duty Cycle (ReF$_3$)</th>
<th>Deposition Rate (nm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZnS: ErF$_3$</td>
<td>150</td>
<td>20</td>
<td>120 W 100%</td>
<td>120 W 75%</td>
<td>4.8</td>
</tr>
<tr>
<td>ZnS: TmF$_3$</td>
<td>140</td>
<td>20</td>
<td>120 W 75%</td>
<td>120 W 100%</td>
<td>4.6</td>
</tr>
</tbody>
</table>
3.2.3 BTO Film Deposition

The top dielectric layer was deposited for full-stack device. For half-stack device, this step was omitted. In this study, barium tantalum oxide ($\text{BaTa}_2\text{O}_6$) films were used as the top dielectric layer and deposited on the phosphor layer by RF sputter deposition. A partially masked glass/ITO substrate was included in each run to measure thickness and capacitance. The dielectric constant was calculated by:

$$K = \frac{C \times d}{\varepsilon_0 \times A}$$

where $C$ is the capacitance (F), $d$ is the thickness of the dielectric layer (m), $A$ is the area of the Al contact ($m^2$), and $\varepsilon_0$ is the permittivity of a vacuum, a universal constant having the value of $8.85 \times 10^{-12}$ F/m.

To get a dielectric constant of 21~25, the processing conditions for BTO are shown in Table 3.2.

3.2.4 Al Film Deposition

Al film, as the top electrode, was formed by thermal evaporation through a shadow mask that has opening of 0.25 cm diameter circle. Base pressure for this process was approximately $10^{-5}$ Torr and a typical thickness is 230nm. Contact to the ITO for testing EL emission was achieved by scratching through the BTO, phosphor and ATO films with a diamond scribe and melting indium metal over the scratched area. Molten In wets the ITO layer for electrical contact.

3.2.5 Rapid Thermal Annealing

The purpose of rapid thermal annealing (RTA) in this research is to enhance the EL intensity. It’s known that as-deposited microstructure has a high density of defects and annealing can increase the film crystallinity [50]. In addition, annealing can also activate
Fig 3.1 Infrared brightness dependence on concentration [49].
Fig 3.2 Infrared brightness dependence on temperature [49].
Table 3.2 Deposition conditions for BTO.

<table>
<thead>
<tr>
<th>Base Pressure (Torr)</th>
<th>Work Gas (%/%)</th>
<th>Work Pressure (mTorr)</th>
<th>Power (W)</th>
<th>Deposition Rate (nm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>~10^{-5}</td>
<td>Ar/O (95/5)</td>
<td>50</td>
<td>120</td>
<td>11</td>
</tr>
</tbody>
</table>
the dopants so that infrared intensity will increase. However, as the annealing temperature goes too high, the dopants will start to cluster and hence infrared emission will decrease. In another study [50], it was shown that there is a critical annealing temperature for ZnS:ErF$_3$ film. Below 425 °C, the infrared signal becomes stronger as the temperature increases; for annealing temperature above 425 °C, the infrared signal decreases.

The samples were placed in a covered graphite susceptor, which was then inserted into the central heating zone of the quartz furnace tube. A thermocouple located at about 1 mm from the phosphor film monitored the temperature (Fig 3.4). To avoid contamination from the graphite, the top and bottom of the samples were covered by two pieces of silicon wafer. Before annealing, the quartz tube was purged for 30 minutes by high purity nitrogen gas (99.9999%) to remove residual oxygen and this purging was continued during the annealing. The heating sources for RTA were two sets of halogen lamps, cooled by both water and air on top and bottom of the quartz tube chamber.

The time for temperature ramping was set for 1.5 minutes, and the annealing time was consistently 1 hour in this study. It normally took 25 minutes to cool down to 40°C.

3.3 Device Characterization

3.3.1 EL Measurement

The primary optical properties such as brightness and emission spectrum were measured for all films. A typical a.c. trapezoidal wave was applied at a frequency of 2.5 kHz. The pulse rise time and fall time are each 5 µsec and the pulse duration time is 30 µsec. Using reflective optics, the emitted light was collected, channeled and focused into the acceptance cone of an Oriel MS257™, 0.25m monochromator equipped with appropriate gratings. A chopper is used for improved signal to noise. An Oriel Merlin™
Fig 3.3 Infrared brightness dependence on annealing temperature (Er) [50].
Fig 3.4 Schematic of rapid thermal annealing set up.
radiometry system interfaced the monochromator and detectors with the acquisition
software. This unit also functioned as chopper controller and lock-in amplifier as well. A
thermo-electric cooled, Ge photodiode on the second exit port of the monochromator
detected the near IR spectrum.

3.3.2 Q-V Measurement

The set-up for electrical measurement is shown in Fig. 3.5. A Tektronix TDS 510A
digitizing oscilloscope is used to measure the voltage at three points which are indicated
as \( V_1, V_2, V_3 \) respectively. The voltage drop across the device was calculated as \( V_2 - V_1 \),
and the voltage drop across the resistor was calculated as \( V_1 - V_3 \). The current passing
through the device was calculated by dividing the voltage drop across the resistor by its
resistance, i.e.,

\[
i = \frac{V_1 - V_3}{R}
\]

The external charge was calculated by integrating the current over time, i.e.,

\[
Q = \int idt
\]

The data were collected on the Tektronix 510A oscilloscope from a Si-diode
photomultiplier tube (PMT) manufactured by Oriel with 1k\( \Omega \) bridge resistor.
Fig 3.5 Schematic of electrical measurement set up.
CHAPTER 4
COMPARISON OF INFRARED EMISSION FROM HALF-STACK AND FULL-STACK ZINC SULFIDE RARE EARTH FLUORIDE ACTFEL DEVICES

4.1 Introduction

This chapter describes the electrical and optical property differences between half-stack and full-stack ZnS:REF$_3$ ACTFEL devices. First, further details of experimental procedure are given. This is followed by a summary of the results of electro-optical characterization of the devices. The observed differences are then discussed in the context of the conduction charge of the various devices. The purpose of this study is to investigate the effects on the infrared EL properties of single insulator versus double insulator ACTFEL devices.

4.2 Experimental

ZnS:REF$_3$ phosphor films (more specifically, ErF$_3$ and TmF$_3$ were used in this study) were deposited by RF planar magnetron sputtering system from two separate targets of pure ZnS and ZnS:REF$_3$. The deposition conditions were discussed in detail in Chapter 3. Table 4.1 gives the phosphor film thickness used in this portion of the study. To form a half-stack device, Al contacts were deposited directly on the ZnS:REF$_3$ films by thermal evaporation.

To form a full-stack device, the top insulator layer was deposited by a RF planar diode sputtering system from a single BaTa$_2$O$_6$ (BTO) target. Afterwards, Al contacts were deposited on the BTO film by thermal evaporation. Table 4.2 gives the BTO and Al film thickness, respectively.
Table 4.1 A list of ZnS:REF₃ phosphor film thickness.

<table>
<thead>
<tr>
<th>Phosphor film</th>
<th>ZnS:ErF₃</th>
<th>ZnS:TmF₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (µm)</td>
<td>0.9</td>
<td>0.7</td>
</tr>
</tbody>
</table>
Annealing is used to increase the EL intensity. In this research, ZnS:ErF₃ ACTFEL devices were annealed at 425 °C for 1 hour in nitrogen.

4.3 Results

4.3.1 ZnS:ErF₃ as ACTFEL Phosphor

4.3.1.1 EL properties comparison

4.3.1.1.1 B-V curve comparison

Fig 4.1 shows the brightness vs. voltage characteristics at 1550 nm for the best ZnS:ErF₃ devices, both as-deposited and annealed.

Table 4.3 compares the turn-on voltages for half vs. full stack devices. From the table, it can be seen that the turn-on voltage for full-stack devices is 40V higher than that for half-stack devices for both the as-deposited and annealed conditions. In addition, the turn-on voltage is 30V higher for as-deposited samples as compared to annealed samples.

4.3.1.1.2 Infrared emission comparison

Fig 4.2 compares the infrared spectral distribution of ZnS:ErF₃ (a) half-stack and (b) full-stack devices. The largest infrared signal at ~1550 nm originates from a $^4I_{13/2} \rightarrow ^4I_{15/2}$ transition [47], and the other near-infrared signal at ~985 nm is the result of a $^4F_{7/2} \rightarrow ^4I_{11/2}$ transition [47].

The spectra in Fig 4.2 show some interest characteristics. The most obvious differences between half-stack and full-stack devices are that the 1550nm infrared signal for the half-stack device is stronger than the infrared signal from a full-stack device. The $B_{40}$ values are compared in table 4.4. In addition, a shoulder appears at ~1480nm for full-stack devices.

Fig 4.3 shows the peak height ratio of the 1550nm peak to the 980nm peak at 40V above threshold. In as-deposited condition, the ratio is 1.9 for half-stack device and 0.2
Table 4.2 A list of BTO and Al film thickness.

<table>
<thead>
<tr>
<th>Thickness (nm)</th>
<th>BTO</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>250</td>
<td></td>
<td>230</td>
</tr>
</tbody>
</table>
Fig 4.1 Brightness vs. voltage for ZnS:ErF$_3$ as-deposited and annealed half and full stack devices.
Table 4.3 Turn-on voltage comparison between ZnS:ErF₃ half and full stack devices.

<table>
<thead>
<tr>
<th></th>
<th>As-deposited</th>
<th></th>
<th>Annealed</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Half-stack</td>
<td>Full-stack</td>
</tr>
<tr>
<td>Turn-on voltage</td>
<td>180</td>
<td>220</td>
<td>150</td>
<td>190</td>
</tr>
<tr>
<td>(V)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

for full-stack device. In annealed condition, the ratio is 17 for half-stack device and 1.1 for full-stack device. It can easily be seen that a higher 1550nm/980nm ratio is obtained in half-stack devices.

4.3.1.1.3 Infrared/visible comparison

Fig 4.4 shows the spectral distribution of the visible emission of ZnS:ErF$_3$ half-stack and full-stack devices. The three emission peaks in the spectrum agree with those reported for erbium doped ZnS film [47]. The faint green light emitted by this system is the result of the transitions at ~525 nm ($^2$H$_{11/2} \rightarrow ^4$I$_{15/2}$) and ~550 nm ($^4$S$_{3/2} \rightarrow ^4$I$_{15/2}$) [47]. In addition, there is another transition at ~660 nm ($^4$F$_{9/2} \rightarrow ^4$I$_{15/2}$). In the as-deposited condition, half-stack devices have higher visible emission intensities than full-stack devices. In the annealed condition, the visible 525 nm and 550nm signals from half-stack devices are stronger, while at 660nm the visible intensity of full-stack is stronger.

The peak height ratio of 1550nm peak to 550nm peak at 40V above threshold in half-stack and full-stack devices is given in Fig 4.5. The data shown in Fig 4.5 displays the same trend as the ratio of 1550nm/980nm reported previously. The ratio is higher for half-stack devices compared to full-stack devices in both the as deposited and annealed conditions; however the ratio is 9 before and 25 after annealing.

The effect of annealing at 425oC on infrared intensity can also be seen from the data in Table 4.4. Annealing enhances the infrared intensity in both half-stack devices and full-stack devices. In addition, it can also be seen from Fig 4.3 and Fig 4.5 that annealing at 425°C increased both the 1550nm/980nm ratio and the 1550nm/550nm ratio. Other data suggest that annealing at higher temperatures may decrease the 1550nm/550nm intensity ratio [50].
Table 4.4 B\textsubscript{40} comparison between ZnS:ErF\textsubscript{3} half and full stack devices at 1550 nm.

<table>
<thead>
<tr>
<th>Irradiance (µW/cm\textsuperscript{2}nm)</th>
<th>As-deposited</th>
<th>annealed</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Half-stack</td>
<td>Full-stack</td>
</tr>
<tr>
<td></td>
<td>0.287</td>
<td>0.02</td>
</tr>
</tbody>
</table>
Fig 4.2 Infrared emission spectrum of (a) as-deposited, (b) annealed ZnS:ErF$_3$ half vs. full stack devices
4.3.1.2 Electrical measurements

Charge-voltage data are critical for an understanding of the transport properties of an ACTFEL device. The significance and procedure for measuring electrical properties were discussed in more detail in chapter 2 and chapter 3, respectively.

The Q-V plots in Fig 4.6 show the external charge on the y-axis versus the applied voltage on the x-axis. Data are shown in Fig 4.6 (a) and (b) for as-deposited and annealed samples, respectively, measured at 40V above threshold using a 120 Ω sense element. The external conduction charges are summarized in table 4.5.

As shown in Fig 4.6 and table 4.5, the half-stack devices show 85% higher conduction charge density as compared to the full-stack devices in the as deposited condition and 65% higher conduction charge density in the annealed condition. In comparison to the as-deposited samples, the annealed samples show about 17% higher conduction charge density for half-stack devices and 31% higher conduction charge density for full-stack devices.

The evaluation of internal charge, $Q_{int}$, from a plot of external charge, $Q_{ext}$, versus time is illustrated in Fig 4.7. The average internal charge (for Al+ and Al- polarity) for ZnS:ErF$_3$ half-stack and full-stack devices are listed in table 4.6, and the same trend as for external charge are observed. The half-stack devices show about 86% higher internal charge compared to the full-stack devices in both as deposited and annealed conditions.

Capacitance-voltage (C-V) data were calculated for both half-stack and full-stack devices as shown in Fig 4.8 (a) and (b) for as-deposited and annealed conditions, respectively. Note that the total capacitance above turn-on for the half-stack devices shows a linear upward slope, and that capacitance values above turn-on are about 1~2 times larger for the half-stack devices.
Fig 4.3 Ratio of the 1550nm peak to the 980nm peak (at V40) for ZnS:ErF3 half and full stack devices in the as deposited or annealed conditions.
Fig 4.4 Visible emission spectrum of ZnS:ErF$_3$ as-deposited and annealed half-stack vs. full-stack devices.
Fig 4.5 Ratio of the 1550nm to 550nm peak intensities (at V_{40}) for ZnS:ErF_{3} half and full stack devices in the as deposited or annealed conditions.
4.3.2 ZnS:TmF$_3$ as ACTFEL Phosphor

4.3.2.1 EL properties comparison

Fig 4.9 shows the comparison of brightness vs. voltage characteristics at 792 nm between ZnS:TmF$_3$ as-deposited half-stack and full-stack devices. The turn-on voltage for a half-stack device is 130V, much lower than the 170 V turn-on voltages for a full-stack device.

Fig 4.10 and Fig 4.11 show the spectral distribution of near-infrared emission and visible emission for ZnS:TmF$_3$ as-deposited half-stack and full-stack devices. The signal at ~790 nm originates from a $^3$F$_4 \rightarrow ^3$H$_6$ transition, and the signal at ~480 nm is the result of a $^1$G$_4 \rightarrow ^3$H$_6$ transition [47].

Table 4.7 summarizes the EL property comparison between ZnS:TmF$_3$ as-deposited half-stack and full-stack devices.

Generally, EL properties of ZnS:TmF$_3$ as-deposited half-stack vs. full-stack devices display the same trends as the ZnS:ErF$_3$ devices reported in the previous section. The turn-on voltage is 40V lower for half-stack device compared to full-stack device, while $B_{40}$ at 790nm is 0.006 $\mu$W/cm$^2$nm for half-stack, much larger than 0.0017 $\mu$W/cm$^2$nm for full-stack devices.

4.3.2.2 Electrical measurements

Q-V data from ZnS:TmF$_3$ ACTFEL devices are shown in Fig 4.12. External conduction charge and internal charge are summarized in table 4.8.

Similar to Er doped devices, ZnS:TmF$_3$ half-stack devices show higher external and internal charge as compared to the full-stack device. Additionally, capacitance-voltage (C-V) data (Fig 4.13) for ZnS:TmF$_3$ as-deposited half-stack vs. full-stack devices
Table 4.5 External conduction charge at $V_{40}$ for ZnS:ErF$_3$ half-stack and full-stack devices.

<table>
<thead>
<tr>
<th>Charge density ($\mu$C/cm$^2$)</th>
<th>As-deposited</th>
<th>Annealed</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Half-stack</td>
<td>Full-stack</td>
</tr>
<tr>
<td></td>
<td>2.4</td>
<td>1.3</td>
</tr>
</tbody>
</table>
Fig 4.6 Q-V data at $V_{40}$ from (a) as-deposited, (b) annealed ZnS:ErF$_3$ half-stack vs. full-stack devices.
Fig 4.7 $Q_{\text{ext}}$ vs. time showing the value of $Q_{\text{int}}$ for a half-stack as-deposited ZnS:ErF$_3$ device.
Table 4.6 Internal charge at V_{40} for ZnS:ErF$_3$ half-stack and full-stack devices.

<table>
<thead>
<tr>
<th></th>
<th>As-deposited</th>
<th>Annealed</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Half-stack</td>
<td>Full-stack</td>
</tr>
<tr>
<td>Internal charge</td>
<td>0.09</td>
<td>0.05</td>
</tr>
<tr>
<td>(µC)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Half-stack</td>
<td>Full-stack</td>
</tr>
<tr>
<td></td>
<td>0.13</td>
<td>0.07</td>
</tr>
</tbody>
</table>
Fig 4.8 Capacitance vs. voltage data for (a) as-deposited, (b) annealed ZnS:ErF$_3$ half-stack vs. full-stack devices.
Fig 4.9 Brightness vs. voltage for ZnS:TmF$_3$ as-deposited half and full stack devices.
Table 4.7 EL property comparison between ZnS:TmF$_3$ as-deposited half-stack and full-stack devices.

<table>
<thead>
<tr>
<th></th>
<th>Half-stack</th>
<th>Full-stack</th>
</tr>
</thead>
<tbody>
<tr>
<td>Turn-on voltage (V)</td>
<td>130</td>
<td>170</td>
</tr>
<tr>
<td>790 nm B$_{40}$ (µW/cm$^2$nm)</td>
<td>0.006</td>
<td>0.0017</td>
</tr>
<tr>
<td>480 nm B$_{40}$ (µW/cm$^2$nm)</td>
<td>0.94</td>
<td>0.57</td>
</tr>
<tr>
<td>790nm/480nm ratio</td>
<td>0.006</td>
<td>0.003</td>
</tr>
</tbody>
</table>
Fig 4.10 Visible red and near-infrared emission spectrum of as-deposited ZnS:TmF$_3$ half-stack vs. full-stack devices.
Fig 4.11 Visible blue emission spectrum of as-deposited ZnS:TmF$_3$ half-stack vs. full-stack devices.
displays similar trends as for ZnS:ErF$_3$ devices. The capacitance value above turn-on for ZnS:TmF$_3$ half-stack devices is about 100% larger than for full-stack devices.

4.4 Discussion

In this study, the primary measure of ACTFEL device performance is brightness, especially the infrared emission intensity. The data in Fig 4.1 and 4.2 show that the lack of the top insulator in ZnS:ErF$_3$ devices dramatically decreases the turn-on voltage and improves the infrared EL brightness. In addition, data in Fig 4.3 and Fig 4.5 show that half-stack devices also have higher infrared/near-infrared and infrared/visible peak height ratios than full-stack devices.

As for the effect of annealing at 425°C, the data in Figs 4.2, 4.3 and 4.5 show that post-annealing of ZnS:ErF$_3$ devices enhances the infrared brightness and the infrared/near-infrared and infrared/visible peak height ratios.

One explanation for a brighter device is an increased amount of conduction charge. The external conduction charge is measured by Q-V data, and the internal charge is equal to the stored charge from Q(t) curves, at the point where the applied voltage is 0V due to the rapid discharge of the device capacitance at the end of the voltage pulse. These data are shown in Fig 4.6. For as-deposited half-stack devices, the amount of external conduction and internal charge is 85% and 90%, respectively, more than for full-stack devices. For 425°C annealed half-stack devices, the amount of external conduction and internal charge is 65% and 86% more, respectively, than for full-stack devices. Since the $B_{40}$ of half-stack devices is higher than that of full-stack devices by a factor of 10, the improved brightness most likely results from an increased conduction charge without the top insulator.
Annealing also increases the amount of conduction charge. Fig 4.6 shows that for half-stack device, annealing causes increases of 17% and 45% in the external conduction.

Fig 4.12 Q-V curve at V40 from as-deposited ZnS:TmF3 half-stack vs. full-stack devices.
Table 4.8 External conduction and internal charge for ZnS:TmF₃ as-deposited half-stack vs. full-stack devices.

<table>
<thead>
<tr>
<th></th>
<th>Half-stack</th>
<th>Full-stack</th>
</tr>
</thead>
<tbody>
<tr>
<td>External conduction charge (µC/cm²)</td>
<td>2.7</td>
<td>1.5</td>
</tr>
<tr>
<td>Internal charge (µC)</td>
<td>0.12</td>
<td>0.075</td>
</tr>
</tbody>
</table>
Fig 4.13 Capacitance vs. voltage curves for as-deposited ZnS:TmF3 half-stack vs. full-stack devices.
and internal charge, respectively. For full-stack device, annealing causes increases of 31% and 40% in external conduction and internal charge, respectively. Annealing has been reported to increase the film crystallinity [50] and the infrared intensity. Based on this study, increased crystallinity allows an increased conduction charge and consequently an improved brightness.

Fig 4.8 shows that capacitance above turn-on is about 1~2 times larger for half-stack device than that for full-stack device. The explanation for this is that as the voltage goes above turn on, the phosphor breaks down so that the device becomes one capacitor for half-stack and two capacitors in series for full-stack. It can be seen that the capacitance for one capacitor is larger than that for two capacitors in series, which is the main reason for increased conduction charge in half-stack devices.

ZnS:TmF$_3$ devices were also studied in this research. However, data were collected only from as-deposited films. The data in Fig 4.9 to 4.10 show the same trends as are observed for ZnS:ErF$_3$ devices. Half-stack device exhibits a 40V lower turn-on voltage, a three times higher near-infrared intensity, and a 100% higher near-infrared/visible ratio. For half-stack devices, the external conduction and internal charges are 80% and 60% larger than for full-stack devices, respectively. Fig 4.13 shows that capacitance above turn-on is 100% larger for half-stack device than that for full-stack device, which again would result in increased conduction charge.
CHAPTER 5
CONCLUSION

The effects on RF magnetron sputter deposited ZnS:ErF$_3$ and ZnS:TmF$_3$ alternating current thin film electroluminescent (ACTFEL) devices of a single (half-stack device) versus both a top and bottom dielectric layer (full-stack device) has been investigated. The half-stack device was formed by depositing Al dot contacts directly on the doped ZnS phosphor film. For full-stack devices, a BaTa$_2$O$_6$ (BTO) film was first deposited as a top insulator, followed by deposition of the Al dot contacts. The half-stack device was found to have lower turn-on voltages (180V vs. 220V for full-stack) in as deposited ZnS:ErF$_3$ ACTFEL devices. In addition, the half-stack device was also found to increase the infrared $B_{40}$ brightness by a factor of 10 over full-stack devices. Both external and internal conduction charge were shown to increase for half versus full stack devices, suggesting that this was the origin of the larger brightness. The effects on EL brightness and spectral distribution of annealing for one hour in nitrogen at 425°C was studied for ZnS:ErF$_3$ films. Compared to as-deposited films, annealed devices showed improved infrared intensity by 35% and lower the turn-on voltages by 30V. Again, increased external conduction and internal charge was the main reason for the improved brightness.

Similar parameters were tested for ZnS:TmF$_3$ half and full stack devices prepared in a similar fashion. The trends for both ZnS:TmF$_3$ and ZnS:ErF$_3$ ACTFEL devices were similar. Half-stack devices exhibit a 40V lower turn-on voltage and a 3.5 times higher infrared signal versus full stack devices, consistent with larger external conduction and internal charges in half-stack devices.
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BIOGRAPHICAL SKETCH

Jie Zhou was born on October 19, 1972, in Shanghai, China. In July 1988, she was admitted to the Department of Mechanical Engineering at East China University of Science and Technology. After receiving a B.S degree in July 1992, she worked at the Chemical Planning and Design Institution in China. In July 1996, she joined the graduate school at East China University of Science and Technology and received a M.S degree in April 1999. In December 2000, she received admission from the University of Florida and joined Dr. Holloway’s group in August 2001. In April 2003, she completed her master’s thesis under the supervision of Dr. Holloway.