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THIN FILM ELECTROLUMINESCENCE

By

Ian Mackay

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TABLE OF CONTENTS

1.0 ABSTRACT	1
2.0 BACKGROUND	2
3.0 EL MECHANISMS	4
4.0 CURRENT DEVICE CHARACTERISTICS	6
4.1 Active Layer	7
4.2 Dielectric Layers	7
4.3 Substrate and Electrode Layers	8
4.4 Matrix Addressing	9
5.0 LOW VOLTAGE DEVICES	10
5.1 AC Devices	10
5.2 DC Devices	11
6.0 EXPERIMENTAL	13
6.1 Phase 1	14
6.2 Phase 2	14
6.3 Phase 3	16
7.0 RESULTS AND CONCLUSIONS	19
8.0 REFERENCES	20

FIGURES

1.	Typical AC Thin Film Device	6
2.	Experimental EL Display Cells	13
3.	Experimental Setup for RF Sputtering	15
4.	Pulsed-Mode Test Circuit	17

1.0 ABSTRACT

The history of electroluminescent(EL) device research is reviewed with an emphasis on the polycrystalline materials effort. The mechanism of EL behavior for direct-current(DC) and alternating-current(AC) powder structures as well as thin film devices is summarized. Current AC device characteristics are evaluated and matrix addressing constraints to low power operation are discussed. Alternatives for lower voltage operation including high dielectric strength materials for AC use, and DC device designs are considered. Attempts at the fabrication of a low voltage, pulsed operation, DC device using a sputtered active layer are reported.

Limited results are reported due to difficulties with the RF deposition equipment. The results indicate, however, that low voltage DC devices may be possible with sputtered active layers under the proper deposition conditions. A major problem in our active layer films of 250 nm or less thickness appeared to be pinholes causing shorts.

In support of the fabrication effort, film thickness determination via interference microscopy is discussed, a pulsed-mode device test circuit is reported, and colorimetric methods for film composition analysis reviewed.

2.0 BACKGROUND

Electroluminescence is generally defined as the direct conversion of electrical energy into light as exhibited by a solid state device. A good example of this behavior is the light emitting diode(LED). The phenomena is distinct from incandescence where electrical energy is first converted into heat and then into light.

Citings under the broader category of luminescence date back to the early 17th century¹. The characterization and study of electroluminescent behavior however is much more recent. Two main branches of the field exist, both beginning early in this century. The first, historically, was the study of single crystal electroluminescence. This branch of EL research has focused on semiconductor crystals. The LED is a result of this research. The second branch incorporates electroluminescence in polycrystalline materials, and is where our attention is directed. Research in this field has been primarily concerned with the development of larger area devices, typically for lighting or display applications.

The beginning of this second branch of electroluminescent(EL) study is most often attributed to Georges Destriau whose first reported on the subject in 1936². His paper detailed a device which produced light from a copper activated zinc sulfide(ZnS) powder, suspended in oil. The suspension luminesced under a strong alternating current(AC) when placed between a metal electrode and a mica insulator.

Destriau later refined his cell by replacing the castor oil and mica with a polymer binder. This basic design, referred to as AC powder EL, was utilized in an effort to develop phosphor powder lamps during the early 1950's³. This was the first major research commitment to EL devices.

Some success was realized from this effort, particularly in low-light level devices. Back lighting for today's liquid crystal(LCD) computer displays use this technology. AC powder lamps could not compete for high brightness applications, however. This is due to limitations in efficiency and operating life. The maximum efficiency of AC powder lamps is about 15 lumens/watt compared with about 60 lumens/watt for fluorescent technology^{4,5}. Their operating life is roughly 2,000 hours compared with 10,000 hours for fluorescent lighting.

In the display area, AC powder technology was used to prototype both numeric and matrix-addressed television display panels during the 1950's and 1960's⁶. As in the case of the lamp devices, operating life was a significant limitation. In addition, with respect to television displays, a significant hurdle was the high operating voltage of the panel. This necessitated control circuitry which was prohibitively expensive(at the time) and complex to manufacture. As a result, the devices never reached commercial application.

During the late 1950's research began on three alternative designs to AC powder for electroluminescent displays⁷. One is AC thin film, another is DC thin film, and the third is DC powder. The AC thin film device is different from the AC powder structure because the phosphor layer is sandwiched between two dielectric layers instead of being suspended in a dielectric material. In addition, the activator is no longer copper but manganese(Mn)⁸.

The DC thin film device is the simplest structure. It eliminates the dielectric completely, leaving a copper-activated phosphor film between two conducting surfaces⁹.

The DC powder structure is identical to its thin film counterpart except for the dimensions and microstructure of the phosphor layer¹⁰.

Of these designs, the AC thin film structure has been the most successful and is commercially available today. Although the high voltage characteristic remains, it is less of a restriction due to the development of high voltage IC drivers. In addition, displays with a long operating life are feasible with this technology due to the difference in microstructure between the powder and thin film phosphor layers¹¹.

The DC thin film device remains attractive because it can operate at lower voltages, but efforts to date have not yielded stable films. The primary problem is catastrophic breakdown in the phosphor layer due to overheating.

DC powder efforts are hampered primarily by a short operating life similar to the AC powder devices.

3.0 EL MECHANISMS

The mechanism for luminescent behavior is different for powder and thin film devices. The AC powder structure consists of grains of ZnS imbedded in a dielectric. Each ZnS grain contains copper as an activator and may contain traces of other elements such as Mn or Cl. The copper, as copper sulfide (Cu_xS), is deposited along structural defects in the grain.

When immersed in a strong electric field, these conductive Cu_xS needles serve as a local source for electrons and holes to the surrounding phosphor. The holes are created at a luminescent center adjacent to the interface and trapped by the Cu_xS activating the luminescent center. Field reversal causes recombination and luminescence. During high brightness operation, however, the Cu_xS needles become excessively hot and begin to diffuse into the grain. This weakens the needle structure and lowers the effectiveness of the injection mechanism. Eventually the luminescent behavior ceases¹².

The DC mechanism for powder is somewhat similar in that Cu_xS needles serve to concentrate the field and act as a source of electrons. In the AC case, however, the needles are on the interior of the ZnS grain and trap holes through a double-injection mechanism. The exterior of the grain is copper free to protect against conduction in the layer. In the DC case, the Cu_xS is deposited on the outside of the grains. A constant high field is then applied to induce migration of the Cu_xS toward the interior of the grain. This process, called forming, results in Cu_xS needles directed into the grain. After the forming process, the needle structures can concentrate the applied field sufficiently to inject electrons into the phosphor. The electrons accelerate in the ZnS conduction band and impact excite luminescent centers as they travel through the grain. As the centers relax, they luminesce. However, the forming process which creates the luminescent behavior continues during device operation. Continued Cu_xS diffusion eventually destroys the needles and luminescence ceases. This effect is accelerated by high brightness operation¹³.

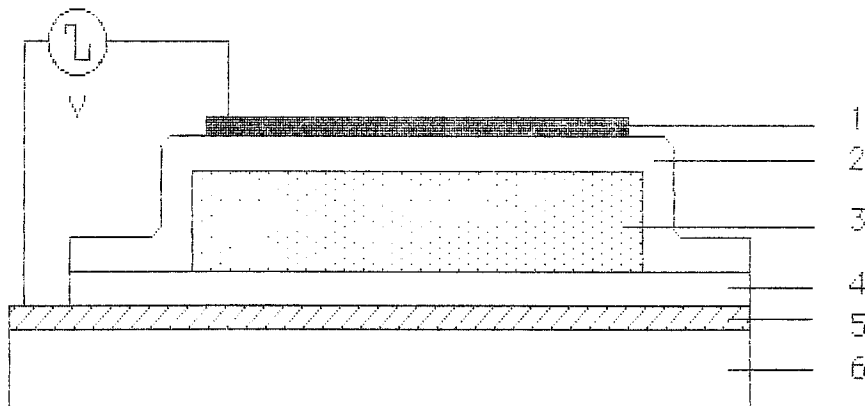
Luminescence in the thin film AC structure operates by a hot electron mechanism similar to the DC powder, but without the Cu_xS dependency. Here the electrons are provided from traps at or near the phosphor/insulator interface. The carriers tunnel into the ZnS conduction

band and are accelerated by the field. They then impact excite the Mn luminescent centers in the film and collect at the opposite insulator interface. The Mn electrons luminesce upon relaxation. At field reversal the trapped electrons at the phosphor/insulator interface repeat their behavior in the opposite direction. In this case, high brightness operation does not affect the stability of the device¹⁴.

4.0 CURRENT DEVICE CHARACTERISTICS

Figure 1. shows a typical AC thin film electroluminescent (ACFEL) display device structure^{15,16,17}. The active layer(3) is sandwiched between identical transparent dielectric layers(2),(4).. The front electrode(5) is also transparent to permit viewing of the cell through the glass substrate(6). The rear electrode(1) is typically evaporated Aluminum (Al). Both electrodes are patterned to provide x-y matrix addressing. This allows subdivision of the active area into picture elements (pixels). The pixels are excited a row at a time and refreshed at 60 Hz. This permits the flicker-free display of raster images.

A typical commercial display device is operated with a square-wave excitation at 200 volts. Pulse width varies from 10 to 50 μsec with a refresh rates of 60 to 500 Hz. Increases in pulse width and refresh rate increase the duty factor and therefore the brightness of the display device¹⁸. The device is usually sealed to provide protection from moisture contamination.



1. Metal Electrode
2. Insulating Layer
3. Active Layer
4. Insulating Layer
5. Transparent Electrode
6. Glass Substrate

Figure 1. Typical AC Thin Film EL Device

4.1 Active Layer

The active layer(3) is typically 500 nm thick and deposited by electron beam evaporation(E-beam) or atomic layer epitaxy(ALE). It consists of ZnS activated with 0.2 weight % Mn(ZnS:Mn). Luminous efficiency peaks at this Mn concentration¹⁹. Lower levels do not provide as large an excitation cross section in the device. Higher concentrations of Mn cause an increase in non-radiative recombination which decreases efficiency.

Variations in thickness result from trade-offs between threshold voltage, and maximum brightness of the cell. The threshold voltage is the minimum voltage required to produce luminescence in the film. It is determined by multiplying the film thickness by the field strength needed to induce breakdown in the film. For ZnS this breakdown value is approximately 1.9×10^6 volts(V)/cm²⁰. Thus a 500 nm film will require a threshold voltage of $(5 \times 10^{-5}\text{cm}) \times (1.9 \times 10^6 \text{ V/cm}) = 95$ volts.

The maximum brightness is determined by the number of luminescent centers in the active layer. This, in turn, is essentially a linear function of film thickness since the density is fixed by the optimum Mn concentration.

The light produced by the ZnS:Mn phosphor is yellow-orange with an emission peak at 585 nm. The strongly non-linear brightness-voltage characteristic saturates within 10% of the threshold voltage, at a value of about 1000 footlamberts(fL) for a 500 nm film. It is the only color available in commercial EL displays. Other phosphors have been used experimentally to generate different colors but with much lower brightness. In particular, terbium fluoride(ZnS:TbF₃) produces green, samarium fluoride(ZnS:SmF₃) produces a red emission. Blue emission has been reported using a strontium sulfide host and cerium fluoride activator(SrS:CeF₃)²¹.

4.2 Dielectric Layers

Several materials have been successfully used in the dielectric layers(2),(4). These include alumina(Al₂O₃), silicon nitride(Si₃N₄), yttrium oxide(Y₂O₃), and amorphous barium titanate(a-BaTiO₃). The films are

typically 200-300nm in thickness and deposited via E-beam, ALE, or sputtering. The primary design considerations for the transparent dielectric are stability and maximum charge density.

The usual reason for device failure is due to the loss of dielectric strength in the insulators. Dielectric layer instability can be due to moisture sensitivity, adhesion problems, chemical reactivity with adjacent layers, or structural changes experienced under operating or preparation conditions²².

The maximum charge density is the product of the low-field dielectric permittivity and the breakdown field constant of the dielectric film. When this value is exceeded, the dielectric breaks down. During device operation below the threshold voltage, the dielectric and active layers act like three capacitors connected in series. Charge is therefore distributed evenly across each layer, and the charge densities are identical. The maximum charge density rating for the dielectric layer must therefore exceed that of the active layer. Otherwise, the dielectric will break down before the active layer does, causing excessive current flow and overheating in the device.

4.3 Substrate and Electrode Layers

The substrate(6) is typically a low-alkali borosilicate glass such as Corning 7059²³. This glass type minimizes the potential of contamination from ion diffusion during operation. It also has sufficient thermal stability to withstand the 500°C annealing cycle sometimes used in display manufacture.

The transparent electrode(5) is usually tin oxide(SnO_2) or indium-tin oxide(ITO) and about 200 nm thick. ITO is preferable due to its lower resistance. The electrode is patterned into 512 columns for a typical display panel. Column length is limited to 10 inches²⁴. Longer lengths cause uneven brightness across the panel due to excess resistance. The back electrode(1) is usually aluminum(Al). It is patterned into 256 rows for a typical panel. The Al rows and ITO columns are orthogonal and define a pixel at each intersection.

4.4 Matrix Addressing

The orthogonal arrangement of the electrodes associates a unique pixel location with each row/column address pair. When the difference in voltage on a row/column pair exceeds the threshold value for luminescence, the pixel is turned on. This addressing arrangement is the simplest for a display device. It requires one control circuit for each row and column.

Multiple pixels can be illuminated simultaneously in a horizontal row to provide line-at-a-time addressing. This is done by placing a voltage below threshold on the row electrode and selectively setting additional voltage on column electrodes. In a typical display, this technique is used to excite each row sequentially at a minimum rate of 60Hz. Since this rate is faster than our ability to detect flicker, the display provides the appearance of a continuous image.

Matrix addressing can only be used for phosphors with a non-linear brightness-voltage response curve. This is due to the sneak circuit formed across non-illuminated pixels by partial row and column voltages. Phosphors with a linear brightness response cause these partially addressed pixels to glow which adversely affects the contrast of the display²⁵.

5.0 LOW VOLTAGE DEVICES

The concept of a low voltage EL panel is attractive because both power consumption and display cost could be reduced significantly. These parameters are particularly important since the main application for EL displays is currently in the portable computer market. Displays manufactured today dissipate about 9 watts at a 25% screen fill factor²⁶. This is substantial considering a typical portable computer equipped with EL requires an average of 12 watts total²⁷.

There are three approaches to lower voltage EL operation. The simplest is to reduce the thickness of the active layer, but this adversely affects brightness. A second concept is to reduce the voltage across the dielectric layers of an AC device. The third approach is to use a DC device.

5.1 AC Devices

In the AC structure prior to breakdown, charge(Q) is distributed evenly across each layer. The voltage drop(V) of the layer is determined by the capacitance(C) according to:

$$V = Q/C \quad (1)$$

The operating voltage of the device is the sum of the voltage drops across each layer. An increase in capacitance of the dielectric layers will reduce the voltage drop across them and the resultant operating voltage of the display.

The capacitance for a dielectric layer is given by:

$$C = k\epsilon A/t \quad (2)$$

where k is the dielectric constant, ϵ is the free space permittivity constant, A is the surface area of the layer, and t is the thickness of the layer. Inspection of the above definition indicates that capacitance can be increased by reducing the thickness of the layers, or by increasing their dielectric constant. Reduction of the thickness below 200 nm is not

practical due to the high density of pinhole defects which effectively short the layer. The use of materials with higher dielectric constants has been demonstrated however^{28,29}.

The voltage across the dielectric layers can be determined from equation (1) once the charge across the layers is established and their capacitance is calculated. The charge can be found using equation (1) for the ZnS active layer if its capacitance is first determined using equation (2). The dielectric constant for ZnS is 8.5 and the permittivity constant is 8.85×10^{-12} farads/meter. A 500 nm ZnS film therefore has a capacitance of 1.5×10^{-4} farads/meter². Using the equation (1) and a 95 volt threshold value, the charge(Q) across the layers is 1.4×10^{-2} coulombs/meter². From equation (2), a 200 nm dielectric layer of Si₃N₄ (dielectric constant of 7) has a capacitance of 3×10^{-4} farads/meter². With the given charge value, the voltage drop across each dielectric layer would therefore be 45 volts, and the total device threshold voltage would be around 185 volts. Changing to a 200 nm dielectric layer of BaTiO₃ (dielectric constant of 14) would cut the voltage drop to 23 volts across each dielectric layer. The threshold voltage for this device would be around 140 volts. The highest reported dielectric constant for an EL insulating layer was 150 for PbTiO₃²⁹. The use of this material in a 200 nm film would result in a device threshold of 100 volts. This material is not commercially acceptable however due to brightness limitations believed to be caused by charge leakage³⁰.

5.2 DC Devices

DC devices operate at a lower voltage than AC displays because they do not require dielectric layers. Threshold voltages as low as 10 V have been reported³¹ although 30 to 45 volts is more common^{32,33}. Some DC devices similar in thickness to the AC structure show lower threshold voltages than the ZnS:Mn breakdown value reported earlier. These devices contain a sputtered active layer or a significant Cu concentration. In the case of the sputtered film, lower threshold values are attributed to improved crystallinity and a longer electron mean free path. In the case of Cu based films, much of the active layer contains low resistance Cu_xS. The driving voltage is mostly across a narrow, luminescing, copper free, "formed" region in the film.

The problem with most DC devices is short life due to overheating

from excess current. Proposed solutions to this problem include adding a fourth resistive layer^{34,35} and using pulsed operation³⁶. The addition of a resistive layer, however, raises the threshold voltage to about 100 volts. Pulsed operation reduces the brightness of the display, but has yielded a stable device.

6.0 EXPERIMENTAL

The research goal was to produce a stable low threshold voltage device through the use of a sputtered active layer and pulsed operation. The experimental cell was modeled after a pulsed-mode DC display cell reported by Abdalla, et al.³⁶. Their original three layer device contained a 10 μm active layer deposited by independent co-evaporation of Zn, S, Mn, and Cu. This was sandwiched between electrode layers of ITO and Al. The device had a 45 volt operating voltage pulsed at 1kHz with a 0.5 percent duty cycle.

The new design substituted a sputtered active layer for the evaporated film reported. The improved crystallinity of the sputtered film was expected to reduce the operating voltage compared with the original cell. The experimental cell, shown in Figure 2, was also a three layer device supported by a glass substrate. The substrate was approximately 9 cm on a side, and came with a thick film coating of SnO_2 . This served as the transparent conductive first layer. The sputtered film formed the middle layer. The top layer consisted of 8 evenly spaced 3.8 x 0.75 cm Al pads vapor deposited through a mask.

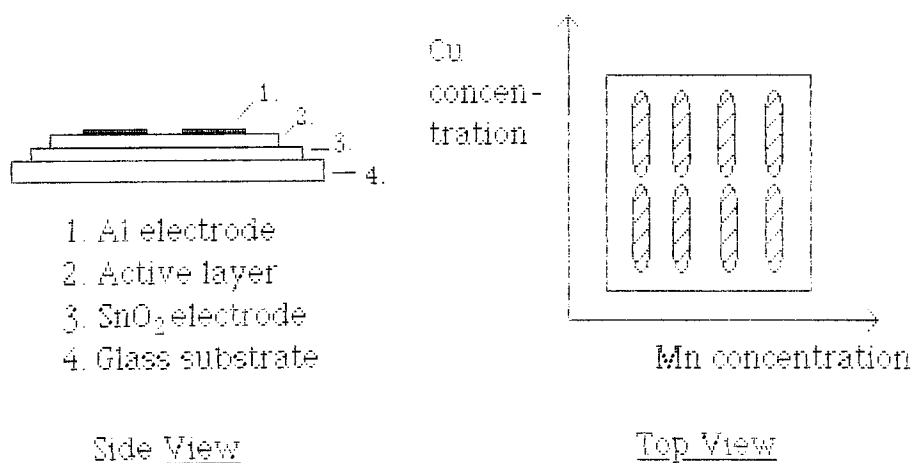


Figure 2. Experimental EL Display Cells

The experimental work was separated into three phases. The purpose of the first phase was to develop basic skills in thin film vapor deposition and design the necessary apparatus to produce the experimental cell. The second phase consisted of trial RF sputter depositions, the determination of appropriate film thickness measurement methods, and the design of test circuitry for the cell. The final phase was devoted to attempts at actual device fabrication and the determination of methods for analysis of the active layer composition.

6.1 Phase 1

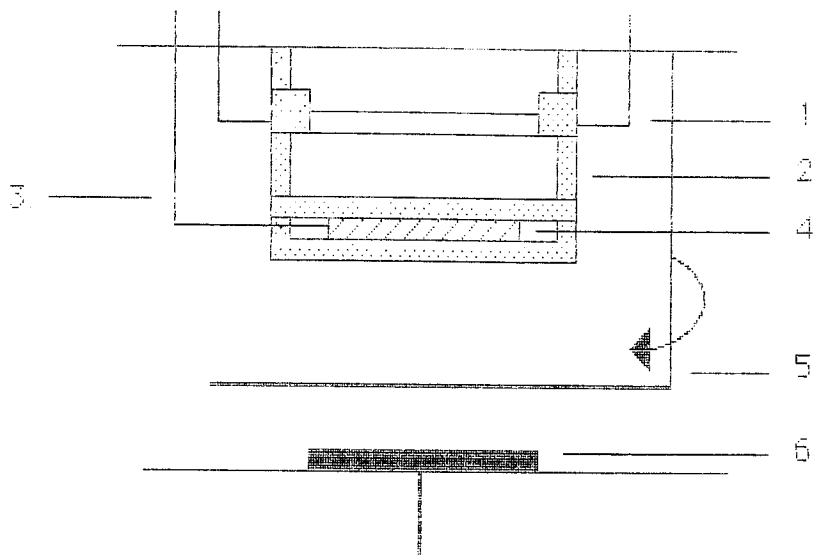
During this phase, a CVC thermal evaporator was used for thin film preparation. In the initial experimental series, Al films on glass slides were deposited to gain familiarity with the equipment. Al pellets heated in a tungsten basket served as the source. Base pressure of this system was typically 2×10^{-5} Torr. Source to substrate distance was about 20 cm.

In the next series an Al-MgF₂-Al capacitor was fabricated to develop some experience in multi-layer masking, and substrate temperature control. A quartz heater and thermocouple was installed in the system and the substrate heated during MgF₂ deposition to improve film density. Phase 1 concluded with the design of a stage, mask and heater for the experimental cell shown in Figure 3.

6.2 Phase 2

Phase 2 began with the installation of the completed stage in the RF sputtering system. The system was a 500W custom design with a 15 cm MRC target. In addition to the custom stage, the chamber was equipped with an Inficon rate deposition monitor. The target substrate distance was 3 cm. A shutter was used to protect the substrate until sputtering began and surface contaminants were removed from the target.

The phase 2 experiments were intended to be simple ZnS depositions on glass. The work was completed, but major difficulties arose at this point.



1. Quartz heater
2. Substrate holder and mask
3. Thermocouple contact
4. Substrate
5. Rotatable shutter
6. RF target

Figure 3. Experimental setup for RF Sputtering

An RF system contains a matching network to adjust the impedance of the chamber to the output of the power supply. Proper matching is necessary to minimize reflected power and deliver the necessary power to the target. However this system could not be properly matched. The result was excess reflected power, fluctuating and often insufficient power to the target, and massive electrical interference. The network was redesigned several times, and finally swapped-out but with only modest success. As a consequence, films produced by this system were generally very thin(250 nm maximum) and of inconsistent quality.

The electrical interference complicated the situation because it rendered the thermocouple pressure and temperature gauges, and the thickness monitor inoperable during sputtering. The loss of the temperature thermocouple forced the abandonment of substrate temperature control.

The loss of the deposition monitor dictated a search for alternative methods to determine film thickness. The solution was found in interference microscopy. A silicon wafer placed near the substrate during deposition could be used as a sample for the thickness determination. The wafer was used because it provided the smooth reflective surface necessary for proper measurement. By masking a portion of the sample during deposition, a sharp transition between the coated and uncoated portions was formed. The interference microscope generates a fringe pattern that shifts according to :

$$d = (\Delta S/S) * (\lambda/2) \quad (3)$$

where d is the optical thickness of the film, ΔS is the shift in the fringe pattern, S is the width of the fringe pattern, and λ is the wavelength of the light source. This method allowed film thickness to be readily determined ± 20 nm.

The last of the phase 2 tasks was the design of a test circuit for pulsed-mode operation of the experimental cell. The circuit was based on a 555 integrated circuit(IC) timer³⁷ and is shown in Figure 4. The variable resistor and capacitor were used to set the pulse width from 4-20 μ s and repetition rate from 30-600 Hz. The output of the 555 IC was inverted to form a positive pulse and fed into the base of an ECG97 power transistor. This in turn modulated DC current provided by a 0-300V DC power supply. The variable supply voltage was used to determine the threshold voltage. The output square wave to the device was monitored with an oscilloscope.

6.3 Phase 3

Efforts in the final phase were directed at producing an experimental cell. The major task was to produce a ZnS:Mn,Cu active layer with appropriate Cu and Mn concentrations.

In order to produce multiple Cu and Mn concentrations across the substrate, the sputter target was set up to generate a concentration gradient of the metals across the film. This was done by adding Cu foil(0.75 cm² area) and a square of Mn filings(2 cm² area) to the 15 cm diameter target of bonded ZnS. The Cu and Mn were aligned at 90 degrees

to each other and placed near the edge of the target. In the completed device, each test cell(defined by an Al pad) contained a unique Mn and Cu concentration.

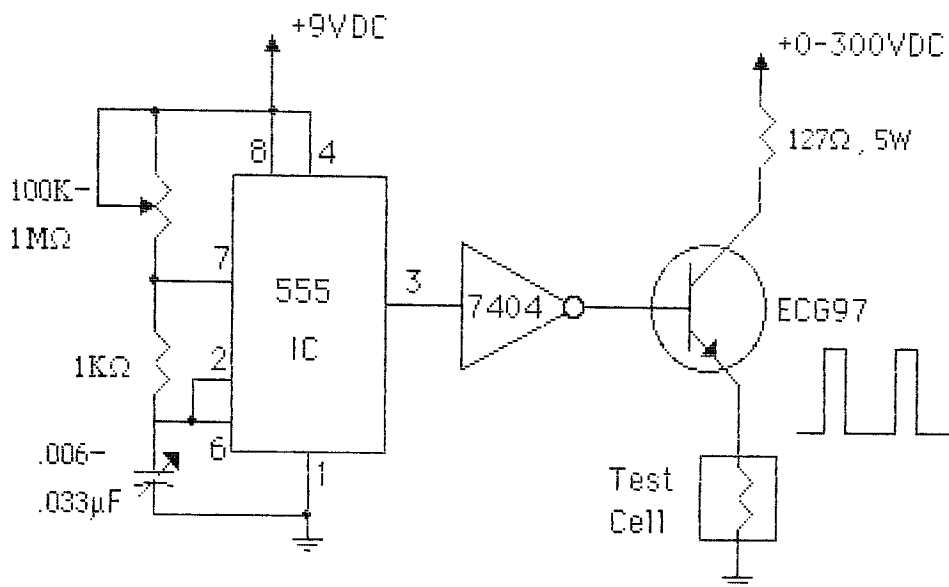


Figure 4. Pulsed-Mode Test Circuit

The sputtered films were then deposited in Ar at a pressure of 40-50 mTorr. Base pressure ranged from $6-9 \times 10^{-6}$ Torr.

The final Al depositions were carried out in the thermal evaporator using an Al mask designed in phase 1.

Sputtered films suffer from a lack of stoichiometric integrity between the composition of the source material and the resulting thin film. In order to determine the actual film composition of the active layer, an analytical laboratory was set-up and the following colorimetric determination methods employed:

Manganese determination was made using the persulfate method³⁸. In this procedure, a standard permanganate solution of known concentration is first made. Then, the unknown manganese sample is

dissolved in a solution of mercuric sulfate, nitric acid, phosphoric acid, and silver nitrate. Ammonium persulfate is then added and the solution boiled to oxidize the manganese to permanganate. After cooling and proper dilution, the optical transmission of the unknown is measured using a spectrophotometer set at a 525 nm wavelength. Comparison with the transmission of the standard yields the unknown Mn concentration.

Zinc determination was made using the dithizone method³⁹. In this method the unknown zinc sample is dissolved in dilute hydrochloric acid. Sodium acetate is added to raise the pH, and sodium thiosulfate is added to complex interfering Cu. The solution is then washed with 0.005 % dithizone in carbon tetrachloride to form zinc dithizonate in the organic layer. This is washed with additional sodium thiosulfate-acetate solutions, diluted and read in the spectrophotometer at 535 nm. Comparison with the transmission of a known Zn concentration completes the analysis.

Sulfur determination was made using the methylene blue method⁴⁰. In this procedure the sulfur unknown is first converted to zinc sulfide using zinc acetate. The sample is then dissolved in 5.5N hydrochloric acid and cooled. Then a n-n-dimethyl-paraphenylene diamine reagent is added and the result oxidized with a ferric chloride solution. After equilibrium is reached, the result is methylene blue which can be read at 670nm.

Colorimetric tests for copper were not evaluated.

7.0 RESULTS AND CONCLUSIONS

The sample devices were tested in a sealed chamber evacuated to 350 mTorr in order to minimize moisture contamination during operation. Results were hampered by difficulties with the custom sputtering apparatus. RF feedback which could not be eliminated caused reduced power throughput, unstable operation, and electrical interference. These problems made it difficult to deposit films and even more difficult to obtain repeatable results.

The sputtered films produced were typically 150 - 250 nm thick. Many of the samples were shorted, probably due to pinhole defects in the active layer. One test cell successfully displayed the yellow-orange luminescence characteristic of ZnS:Mn at a driving voltage of 15 volts, but then quickly destroyed itself due to overheating.

The inability to reproduce working films precluded the testing of pulsed mode operation in any detail, as well as the film composition analysis.

It appears from the limited results however that sputtered ZnS:Mn,Cu films can be driven at lower threshold voltages than their evaporated counterparts if the deposition can be properly controlled. But the pinhole defect density in the active layer was apparently too high to make this method practical for phosphor films of less than 250 nm thickness using the deposition methods we employed. Pinhole defects could potentially be reduced by controlling the substrate temperature during deposition, or performing a post-deposition anneal. A successful reduction in defect density for the active layer could result in an experimental EL device with stable low voltage operation.

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