DISTRIBUTED STRESS SENSING AND NON-DESTRUCTIVE TESTS
USING MECHANOLUMINESCENCE MATERIALS

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DISTRIBUTED STRESS SENSING AND NON-DESTRUCTIVE TESTS
USING MECHANOLUMINESCENCE MATERIALS

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ABSTRACT

Rapid aging of infrastructure systems is currently pervasive in the US and the anticipated cost until 2020 for rehabilitation of aging lifeline will reach 3.6 trillion US dollars (ASCE 2013). Reliable condition or serviceability assessment is critically important in decision-making for economic and timely maintenance of the infrastructure systems. Advanced sensors and nondestructive test (NDT) methods are the key technologies for structural health monitoring (SHM) applications that can provide information on the current state of structures. There are many traditional sensors and NDT methods, for examples, strain gauges, ultrasound, radiography and other X-ray, etc. to detect any defect on the infrastructure. Considering that civil infrastructure is typically large-scale and exhibits complex behavior, estimation of structural conditions by the local sensing and NDT methods is a challenging task. Non-contact and distributed (or full-field) sensing and NDT method are desirable that can provide rich information on the civil infrastructure’s state.

Materials with the ability of emitting light, especially in the visible range, are named as luminescent materials. Mechanoluminescence (ML) phenomenon is the light emission from luminescent materials as a response of an induced mechanical stress. ML materials offer new opportunities for SHM that can directly visualize the stress and crack distributions on the surface of structures through ML light emission.

Although material research for ML phenomena have been made substantially, applications of the ML sensors to full-field stress and crack visualization are still at infant stage and have yet to be full-fledged. Moreover, practical applications of the ML sensors for SHM of civil infrastructure have difficulties since numerous challenging problems (e.g. environmental effect) arise in actual applications. In order to realize a practical SHM system employing ML sensors, more research needs to be conducted, for examples, fundamental understandings of physics of ML phenomenon, method for quantitative stress
measurements, calibration method for ML sensors, improvement of sensitivity, optimal manufacturing and design of ML sensors, environmental effects of ML phenomenon (e.g. temperature), image processing and analysis, etc.

In this research, fundamental ML phenomena of two most promising ML sensing materials were experimentally studied and a methodology for full-field quantitative strain measurements, for the first time, was proposed along with a standardized calibration method. Characteristics and behavior of ML composites and thin films coated on the structure have been studied under various material tests including compression, tension, pure shear, bending, etc. In addition, ML emission sensitivity to the manufacturing parameters and experimental conditions was addressed in order to find optimal design the ML sensor. A phenomenological stress-optics transduction model for predicting the ML light intensity from a thin-film ML coating sensor subjected to in-plane stresses was proposed. A new full-field quantitative strain measuring methodology by ML thin film sensor was developed, for the first time, in order to visualize and measure the strain field. The results from the ML sensor were compared and verified by finite element simulation results. For NDT applications of ML sensors, experimental tests were conducted to visualize the cracks on structural surfaces and detect damages on structural components.

In summary, this research proposes and realizes a new distributed stress sensor and NDT method using ML sensing materials. The proposed method is experimentally validated to be effective for stress measurement and crack visualizations. Successful completion of this research provides a leap toward a commercial light intensity-based optic sensor to be used as a new full-field stress measurement technology and NDT method.
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1-1 Introduction

Several full-field strain measurement methods are available, including DIC [1], photoelastic coatings [2], and Moire and interferometric methods [3]. Among these, DIC techniques have been most widely used in various applications for its non-contact sensing ability and relative ease of implementation and use. In DIC, strains are computed results from measured displacement fields. The resolution of the displacement (strain) from DIC depends on camera resolution, lens optical quality, and the quality and size of the speckles on the specimen surface. Besides these, the strain accuracy is very dependent on the facet sizes and facet steps and they should be adopted with the surface pattern accordingly. Moreover, DIC is very sensitive to fluctuation and nonuniformity of the light intensity and the calculation process is tedious and time consuming. Also, there are many existing commercially available NDT methods such as Ultrasound, Radiography and other x-ray, Visual, optical & photonic, Eddy current, Infrared/thermal imaging, Liquid penetrant, Magnetic particle, and Acoustic emission to detect any defect on structures and can be used in SHM applications. These methods are used for inspection, SHM, and maintenance of structures and industrial machine components following safety-related regulation. All the current NDT methods have advantages and disadvantages depending on materials, defect forms, level of effects from environment, etc. In general, methods providing accurate inspection results (e.g. ultrasound, radiographic method) are very skill and labor intensive and require high-cost equipment. Therefore, for use in routine inspection, it may not be suitable. Hence, a method is needed that is less skill-intensive, less labor and time
consuming, and less affected by environment for the full-field stress measurement, NDT and SHM applications.

ML materials have the capability to emit light when subjected to mechanical stresses. The applied stresses could be caused by tension, pressure, bending, fracture, impact, friction, and so on. By converting the ML light emission to the actual stress (strain values), the ML particles can be employed to detect any cracks or defects or measure the mechanical stresses on the structural surfaces. Recently, the ML sensor has drawn significant attention as a stress sensor for its direct visualization of strains and cracks through ML light intensity. Several attempts to apply film-type ML sensors to the practical monitoring of civil infrastructure have been reported. Various ML materials have been applied to stress sensors[4, 5], impact sensors[6]; damage sensors[7], visualizations of stress distributions of solids[8], monitoring of active cracks[9], stress fields near the tip of a crack[10], quasi-dynamic crack-propagation in solids[11] and internal defects of pipes[12] by their light emission characteristics under elastic/plastic deformations, friction, fractures[13] or impacts. According to the previous research on mechanoluminescent materials[14], SrAl$_2$O$_4$:Eu (SAOE) and SrAl$_2$O$_4$:Eu,Dy (SAOED) are known for their intense light emission under mechanical loadings to the extent that the emission can be seen in broad daylight with the naked eye. These two materials are considered the most promising ML sensing materials for SHM applications [14]. Whereas SAOE is known to be more suitable for dynamic crack visualization because of its high sensitivity, SAOED is suitable for full-field strain visualizations because of its better linearity [15]. Stress-sensitive ML sensors can visualize distributions of stresses and strains and can potentially be used for monitoring structural integrity in large SHM systems.

1-2 Aim of the Thesis

The goal of this work is to obtain more understanding of the performance of Mechanoluminescence materials to develop a calibration model for ML strain sensors which can convert the ML light emission to the real-time full-field strain measurements and to validate the quantitative strain measurement. Moreover, the quantitative strain measurements are compared with the commercially Finite Element (FE) software.
In addition, ML emission sensitivity to the manufacturing parameters and experimental conditions is considered in order to improve the ML sensor performance. These advanced visual sensors will be used as new full-filed strain sensors and a non-destructive method for damage detection and SHM purposes.

1-3 Dissertation Outline

Introduction and the aim of this dissertation are presented in Chapter 1. Chapter 2 covers the literature survey of various mechanoluminescent materials, manufacturing methods, photoluminescence mechanisms, and the exiting methods of measuring strains and the current models of ML phenomena. The other visual sensing methods are also briefly described in Chapter 3. Experimental methods used in this thesis are described in Chapter 3 and experimental setup and procedures are explained in Chapter 4. Experimental results are presented in Chapter 5. A new predictive transduction model and a novel calibration method are described in Chapter 6 and followed by the verification and validation of the results. Visualization of crack propagation by using ML sensor is presented in Chapter 7 and followed by a case study for stress visualization. Finally, conclusion and future works are presented in Chapter 8.
CHAPTER II
LITERATURE REVIEWS

2-1 Overview

In the past decade, attention has been paid to new smart materials such as Piezoelectric, Mechanoluminescence, Magneto-electric materials, and so on [16-19]. These materials are designed in a way that their properties can significantly change by external stimuli such as stress, temperature, magnetic, or electric fields. Among those, ML materials can emit light when they are subjected to dynamic mechanical stresses. The light emission color of the ML materials covers a broad spectral range from ultraviolet to infrared light. The mechanical stimulations can be caused by tension [20-23], compression [24-27], friction [4, 28-32], pressure [12], bending [33], impact [34-36], ultrasonic [37], and so on. Figure 2-1 visually shows the green light emission of a ML composite circular disc shaped specimen under the compression loading. This ML light intensity increases by increasing the applied load.

![Compression test of a light emitting sample of ML materials](image)

Figure 2-1: Compression test of a light emitting sample of ML materials
Over the past century, the ML phenomenon has been investigated spectroscopically and crystallographically [38]. According to the previous studies, 19% of organic, 36% of inorganic, 37% of aromatic compounds, 70% of alkaloid, and around 50% of all crystalline materials show the mechanoluminescence properties [39-42]. An interesting usage of this phenomenon is that it can be employed to visualize mechanical stress (or strain) by converting the visible light intensity into the mechanical energy [43]. The applications of the ML materials include, but not limited to, stress sensors [4, 5], impact sensors [6], damage sensors [7], visualizations of stress distributions of solids [8], monitoring of active cracks [9], calculating the stress intensity factors near the tip of cracks [10], visualization of the crack-propagation in solids [11] and internal defect detection of pipes [12]. However, the ML phenomenon is complex due to many factors involved in transient changes of ML light emission and very few studies have been focused on the quantification strain measurements from the ML sensor.

In this chapter, various types of the ML materials are introduced and different manufacturing methods are described. Mechanoluminescence mechanism is explained and followed by literature survey of previous studies on applications and performances of different ML materials.

2-2 Various Mechanoluminescent Materials

A variety of ML sensing materials —such as SrAl$_2$O$_4$:Eu$^{2+}$ (green) [5], SrAl$_2$O$_4$:Eu$^{2+}$,Dy$^{3+}$ (green) [11, 44], ZnAl$_2$O$_4$:Mn$^{2+}$ (green) [28], ZnS:Mn$^{2+}$ (yellow) [4], SrAl$_2$O$_4$:Ce$^{3+}$ (ultraviolet) [45], SrAl$_2$O$_4$:Ce$^{3+}$,Ho$^{3+}$ (ultraviolet) [45], BaSi$_2$O$_5$N$_2$:Eu$^{2+}$ (bluish-green) [46], CaZnOS:Cu$^{2+}$ (red) [47], CaZnOS:Mn$^{2+}$ (red) [48], CaZnOs:Mn$^{2+}$,Li$^+$ (red) [27], SrMgAl$_6$O$_11$:Eu$^{2+}$ (green) [49], SrCaMgSi$_2$O$_7$:Eu$^{2+}$ (bluish-green) [50], Sr$_2$MgSi$_2$O$_7$:Eu$^{2+}$ (green) [51], Ca$_2$MgSi$_2$O$_7$:Eu$^{2+}$,Dy$^{3+}$ (green) [52], CaYAl$_6$O$_{12}$:Eu$^{2+}$ (green) [53], CaZr(PO$_4$)$_2$:Eu$^{2+}$ (blue) [54], Ca$_2$Al$_2$SiO$_7$:Ce$^{3+}$ (yellow) [55, 56] and so on— have been developed and improved as potential ML stress sensors. As indicated in the parenthesis, the available colors of the ML light emission cover a broad spectral range from ultraviolet to infrared light. Each ML material shows the ML sensitivity to the mechanical loading to varying extents. To be used as stress sensors, they desire to have several critical characteristics, for instance; prompt response, intense ML light intensity, multi stress sensitivity, and wide measurement range for dynamic loading [48].

5
In the past, europium-doped alkaline earth aluminates, mainly SrAl$_2$O$_4$:Eu$^{2+}$ (SAOE), has increasingly received attentions for its luminescence properties [57]. The auxiliary activator role of Dy$^{3+}$ in long lasting afterglow of SrAl$_2$O$_4$:Eu$^{2+}$,Dy$^{3+}$ (SAOED) was discovered by Maruyama et al. [58]. SAOED showed a consistency in brightness and persistent after-glow properties at room temperature for several hours. These materials show a very intense green light emission with a peak wavelength of 520 nm. The light emission is very intense which can be seen even with naked eye. The excellent chemical stability, as well as the non-toxic properties of these phosphorescent materials, has resulted in employing them in various applications as luminescent pigments [59]. ZnS:Cu is the other phosphorescent materials which is substituted by SAOED because of its extremely sensitivity to moisture and chemically instability [60]. Table 2-1 shows the comparison between the most promising ML materials which are suitable for stress sensor applications [14].

<table>
<thead>
<tr>
<th>Crystals</th>
<th>Wavelength peak of the ML light emission (nm)</th>
<th>ML intensity</th>
<th>Suitability for stress sensors</th>
</tr>
</thead>
<tbody>
<tr>
<td>SrMgAl$<em>6$O$</em>{11}$:Eu</td>
<td>512</td>
<td>Very high</td>
<td>No</td>
</tr>
<tr>
<td>Sr$_2$MgSi$_2$O$_7$:Eu</td>
<td>460</td>
<td>High</td>
<td>No</td>
</tr>
<tr>
<td>SrCaMgSi$_2$O$_7$:Eu</td>
<td>490</td>
<td>High</td>
<td>No</td>
</tr>
<tr>
<td>SrBaMgSi$_2$O$_7$:Eu</td>
<td>440</td>
<td>Moderate</td>
<td>Yes</td>
</tr>
<tr>
<td>SrAl$_2$O$_4$:Eu,Dy</td>
<td>520</td>
<td>High</td>
<td>Yes</td>
</tr>
<tr>
<td>SrAl$_2$O$_4$:Eu (compression)</td>
<td>520</td>
<td>Very High</td>
<td>Yes</td>
</tr>
<tr>
<td>SrAl$_2$O$_4$:Eu (tension)</td>
<td>520</td>
<td>High</td>
<td>Yes</td>
</tr>
<tr>
<td>SrAl$_2$O$_4$:Eu (film)</td>
<td>520</td>
<td>High</td>
<td>Yes</td>
</tr>
<tr>
<td>ZnS:Mn</td>
<td>580</td>
<td>Medium</td>
<td>Yes</td>
</tr>
</tbody>
</table>

SrAl$_2$O$_4$:Eu$^{2+}$ and SrAl$_2$O$_4$:Eu$^{2+}$,Dy$^{3+}$ phosphors have been found to be the most suitable elasto-mechanoluminescent materials for stress sensor application [61]. Therefore, their fundamental ML
properties based on experimental test are characterized and their performances in stress sensor application and NDT methods are investigated in this thesis.

2-3 Manufacturing Methods of Mechanoluminescent Materials

Several techniques have been developed for preparation of the mechanoluminescent powder such as: solid state reaction method, chemical synthesis techniques, and laser synthesis. Solid-state method is a conventional synthesis method of the rare earth activated strontium aluminates and is a thermally driven reaction of Al₂O₃ and SrCO₃. In this reaction method, a very high temperature (above 1250º C) is required and also the grain size of the particles is in the order of 10-100 micron and smaller particles must be obtained by grinding method [62]. It can also cause defects to the particles and significantly decreases the efficiency of the luminescence phenomena [63].

Alternatively sol-gel method has been developed to prepare the phosphorescent materials at lower sintering temperatures [64, 65]. The disadvantage of the sol-gel method is its long synthesis time requirement which limits the industrial use [59]. Different synthesis techniques have also been developed such as hydro thermal and combustion methods [62, 66, 67], co-precipitation method [68], sol-gel-microwave processes are [69], and laser synthesis method [59]. In the following, common methods and techniques of preparing the SAOE and SAEOD particles are described in details.

2-3-1 Solid-state reaction method

Solid-state method is one of the conventional and the most widely used methods of manufacturing the phosphorescent materials such as SAOE and SAEOD from a mixture of solid raw materials. Solids do not react together at room temperature. Therefore, a very high temperature, often 1000 to 1500º C, is required in order for the reaction to occur at an appreciable rate. Fu et al. used the following procedure to manufacture the SAOE powder by using solid-state reaction method. In the first step, SrCO₃ and Al₂O₃ are mixed and the host compounds are synthesized by firing the intimate mixtures at high temperature. For the doping ions sources, Eu₂O₃ is used. After grinding the mixture, it is heated at 700º C for two hours. Then they are calcined at 1400º C for five hours in the thermal-carbon reducing atmosphere [70].
Manufacturing of the SAOED powder by the solid-state reaction method is described as follows. In the first step, appropriate mixtures of SrCO₃, Al₂O₃, Eu₂O₃, oxide of lanthanide elements (expect Eu and Pm), and B₂O₃ are calcined as a flux in mildly reducing atmosphere at 1300º C for one hour [58, 71]. In order to have a uniform product, the final mixture is ground and sieved through a 200 mesh screen. The molar ratio of the Dy to Eu is selected as 2 to 1 [58].

Jha and Chandra used a different procedure [71]. Initially, they mixed appropriate quantities of the raw materials SrCO₃, Al₂O₃, Eu₂O₃, and Dy₂O₃ according to the molar ratio and then press them into a pellet. Small amount of boric acid (HBO₃; AR) was used as a flux. The pellet was pre-sintered in a furnace at 1000º C for 5 hours in air. Then the product was ground and sintered at 1350º C for more 5 hours in a reducing atmosphere of 95%Ar + 5%H₂ flow. Many pieces of phosphor particles were obtained in the size range of mm. Suitable size were reached by grinding and polishing.

2-3-2 Combustion synthesis method

The combustion synthesis method is a common chemical synthesis technique for preparing the nanoparticle phosphorescent powders. In the combustion synthesis methods, all the procedures are conducted in liquid phases so each component is precisely controlled and uniformly dispersed in the liquid [70]. Moreover, the process is very safe, facile, instantaneous and energy saving [70]. Figure 2-2 shows the experimental test setup for the combustion synthesis technique from Chander et al. experiment.

Fu et al. proposed the following method to manufacture the nano- crystals SrAl₂O₄:Eu²⁺ by using the combustion synthesis method. First, Sr(NO₃)₃ (0.2 M), Al(NO₃)₃ (0.2 M), Eu(NO₃)₃ (0.1 M), and urea(0.2 M) solutions are prepared [70]. Then, appropriate volumes are mixed in evaporating dish. The molar ratio of urea to NO₃⁻ is selected as 2 to 3 which can controls the grain size of the SAOE particles. The bigger grain sizes can be obtained by higher molar of the urea to NO₃⁻. After fully stirring, the precursor solution is maintained in a muffle furnace with a lid at 600º C. Initially, the solution boiled and underwent dehydration, followed by decomposition with the evolution of large amounts of gases (oxides of carbon, nitrogen and ammonia). The white foamy and voluminous SAOE powder will be obtained by spontaneous combustion with enormous swelling. The total process takes less than five minutes [70].
Chander et al. developed a modified combustion technique for preparing the SAOED phosphors [72]. In their process, a mixture of respective metal nitrates, flux and combustible agent (urea/camphor) were thermally treated with slight modification at 400-600°C for about 5 minutes. The advantage of this method is obtaining the more homogenous incorporation of dopants and large-scale production of the nanophosphor in a short interval time.

![Schematic experimental setup of the combustion synthesis technique](copied from [72])

**2-3-3 Sol-gel synthesis method**

The Sol-gel technique is a method for producing solid materials from small molecules. Conversion of monomers into a colloidal solution (sol) that act as the precursor for an integrated network (gel) of either secrete particles or network polymers are involved in the process [65, 73].

Lu et al. prepared the SAOED phosphor powder by a new sol-gel technique [74]. Aluminum isopropoxide and strontium acetate were used in their study as precursors. Their experiment shows that the SrAl₂O₃ was formed at 900°C, which is 300°C lower than the required temperature for conventional solid-state reaction. Eu₂O₃, Dy₂O₃, Sr(CH₃CO₂)₂·1/2H₂O and Al(i-OC₃H₇)₃ were selected as the starting materials. Aluminum isopropoxide was dissolved in ethylene glycol monoethyl and the oxides of Eu, Dy, and strontium acetate were dissolved in concentrated nitric acid and deionized water respectively. Then,
two solutions were mixed with the molar stoichiometry 0.96:0.04:0.04 corresponding to Sr/Eu/Dy. The precursor solution was stirred at room temperature for one hour and heated at 60º C for 12 hours. After hydrolysis and condensation, the prepared white gels were dried at 180º C for 24 hours, calcinated in a furnace with air atmosphere at temperatures from 900 to 1250º C for 4 hours, respectively. Finally, the powders were heated at 1200º C for 2 hours in a reducing atmosphere to assure complete conversion of Eu³⁺ to Eu²⁺ [74]. Figure 2-3 demonstrates the schematic flow chart diagram for the synthesis of SAOED powder by using the sol-gel synthesis technique.

Figure 2-3: schematic flow chart diagram of the sol-gel synthesis method for preparing the SAOED phosphors powder (redrawn from [74])
Co-precipitation synthesis method

Co-precipitation is a synthesis technique in order to prepare the phosphorescent nanoparticles [68, 75]. The starting materials are Sr(NO$_3$)$_2$, Al(NO$_3$)$_3$.9H$_2$O, (Aldrich, 99.% purity), Eu(NO$_3$)$_3$.5H$_2$O, Dy(NO$_3$)$_3$.5H$_2$O with corresponding molar ratio of 0.97 : 2.0 : 0.01 : 0.02 [75]. Then the stoichiometrically weighed nitrates is dissolved stirred for 20-25 minutes. The solution was then titrated with (NH$_4$)$_2$CO$_3$ to be precipitated. The colloidal precipitates were dried at 100$^\circ$ C for 20 hours in a dry oven and then annealed at 300$^\circ$ C for 5 hours to assure the water and remnant nitrates are removed completely. A small amount of B$_2$O$_3$ (2 wt %) was added to the calcined powders as a flux. Finally, the powders were heated in the temperature range of 800-1400$^\circ$ C for 3 hours in a reducing atmosphere of Ar-5%H$_2$ gas. Figure 2- 4 shows the schematic flow chart of the co-precipitation synthesis method for preparing the SAOED phosphorescent powder.

![Figure 2- 4: Schematic flow chart diagram of the co-precipitation synthesis method for preparation of the SAOED (redrawn from [75])](image-url)
2-3-5 Laser synthesis method

Aroz et al. developed a new laser melting method for the synthesis of SAOED phosphor [59]. They used a high-power density CO₂ laser irradiation to achieve the high temperature to synthesize the SrCO₃, Al₂O₃, Eu₂O₃, and Dy₂O₃. Figure 2-5 demonstrates the schematic experiment setup of the laser synthesis method used by Aroz et al. Their method is fast synthesis, one step and conducted in air at atmospheric pressure. In their method, starting materials were commercial SrCO₃, Al₂O₃, Eu₂O₃, and Dy₂O₃ powders according to the nominal composition of SrAl₂O₄, SrAl₂O₄:Eu₀.₀₁, and SrAl₂O₄:Eu₀.₀₁Dy₀.₀₂. A Retch 2000 Ball Mill has been used to dry and mix the starting materials for one hour at 250 rpm and isopropyl alcohol has been employed as a liquid suspension medium. The precursor powder was placed in a refrigerated aluminum crucible and scanned with a thin-line beam configuration thereafter. They have claimed that the proposed laser synthesis method yields pure monoclinic SrAl₂O₄ without the addition of any flux (i.e. boron oxide) to stabilize the monoclinic polymorph [59].

Figure 2-5: Schematic experimental setup of the laser synthesis apparatus employed in Aroz et al. work, laser source (1), beam steering system (2), laser beam steering zone (3), laser-surface modified aluminum crucible (4) (copied from [59])
2-4 Mechanisms of the Photoluminescence

In this section the light emission mechanism of the ML materials is described. The mechanism of this phenomena is explained separately for three different ML materials: 1) rare earth doped strontium aluminates and other persistent luminescent crystals (i.e. SAOE, SAOED, CaZnOS:Mn$^{2+}$, CaZr(PO$_4$)$_2$:Eu$^{2+}$ and ect.), 2) ZnS:Mn crystals, and 3) X- or $\gamma$-irradiated alkali halide crystals.

2-4-1 Mechanism of the photoluminescence of rare earth doped strontium aluminates and other persistent luminescent materials

The mechanism of the mechanoluminescent behavior of SAOED crystals in the linear elastic range can be described as the following steps on the basis of the previous researches [5, 32, 48, 54, 76-78]:

Step 1: When the SAOED is irradiated by UV light, the electrons of Eu$^{2+}$ are excited from 4f$^7$ ground level to the 4f$^6$5d$^1$ level lying very close to the bottom of the conduction band.

Step 2: The 5d levels of Eu$^{2+}$ are partly in the conduction band of SAOED and some electrons can escape to the conduction band with the aid of the photoexcitation and Eu$^{2+}$ becomes Eu$^{2+}$-h$^+$. 

Step 3: Some of the escaped electrons come back to the 5d levels and then to the 4f$^7$ ground level subsequently and that produces the persistent luminescence (PL) light emission. The rest of the escaped electrons, are trapped from the conduction band to an electron trap at the oxygen vacancy levels located in the vicinity of the photogenerated Eu$^{2+}$-h$^+$. These electron traps can be shallow or deep and they are considered to be stable at room temperature [48, 76].

Step 4: When a stress is applied, the SAOED lattice is deformed, inducing the strain energy and the trapped electron are de-trapped and could be excited to the conduction band. The shallow trap levels could be excited by weak strain energy to the conduction band, while the deep trap levels needs high strain energy to be able to go to the shallow levels then to the conduction band, or directly to the conduction band by tunneling [48].

Step 5: The de-trapped electrons come back to the 5d levels of Eu$^{2+}$-h$^+$ and eventually to 4f ground levels and the inherent green light emission can be observed. This phenomenon is named as mechanoluminescence (ML) [76].
Figure 2-6 schematically demonstrates the mechanism of the photoluminescence of the rare earth doped strontium aluminates and other persistent luminescent materials.

![Mechanism of ML phenomena of SAOED based sensing films](image)

Figure 2-6: Mechanism of ML phenomena of SAOED based sensing films (ET: energy transfer; kT: thermal energy; Vo: oxygen vacancy; Vsr: strontium vacancy)[79]

2.4.2 Mechanism of the photoluminescent of ZnS:Mn crystals

The step by step mechanism of the ML phenomena in ZnS:Mn crystals can be describe as follow [76, 80]:

Step 1: The piezoelectric field is formed because the crystal structure of ZnS is non-centrosymmetric

Step 2: The electrons from filled-electron traps are detrapped to the conduction band due to the band bending caused by the piezoelectric field

Step 3: The non-radioactive energy is released during the electron-hole recombination

Step 4: The Mn$^{2+}$ ions are excited by the released energy caused by electron-hole recombination

Step 5: The excited Mn$^{2+}$ ions are relaxed and the light emission is produced.
2-4-3 Mechanism of the photoluminescence of X- or γ-irradiated alkali halide crystals

The step involved in the elastico ML emission of X- or γ-irradiated alkali halide crystals are as follows [76, 81, 82]:

Step 1: The induced stress causes bending of the dislocation segments.

Step 2: The electrons are captured from the interacting F-centers lying in the expansion region of the dislocation segments.

Step 3: The captured electrons move with the dislocation segments and slide through the axes of dislocation.

Step 4: The recombination of dislocation-captured electrons with holes lying in the dislocation donor bands produces the light emission of the halides ions in V2 centers or other hole centers [76].

2-5 Different Technique for Applying the Stress on Mechanoluminescence Crystals

Different loading techniques have been used to induce the stress on ML particles. These loading techniques consist of: compression, bending, tension, torsion, impact or impulsive, cleaving and cutting, laser, ultrasonic, cyclic, air blast, grinding and milling, hydraulic pressure, and tribo or rubbing techniques [25, 76, 83].

2-5-1 Compression loading technique

A compression technique can be used to simultaneously measure the stress-strain curves by using universal testing machine (Instron). The samples can be made in a circular form disc shape [27, 46, 84, 85]. Stress is measured by using a load cell and strain is measured by using linear variable differential transducer (LVDT) [24-27]. Figure 2- 7 shows a red light emission of the CaZnOS:Mn^{2+} disc specimen under compression loading.
2-5-2 Tension loading technique

Many researchers have performed tension test of ML samples. Those samples could be bulk ML materials or ML thin film attached to the host materials [20-23]. Universal testing machine is usually used to control the maximum loading and load rate.

2-5-3 Bending loading technique

Li et al. carried out a four-point bending test to predict fracture in a reinforced concrete structure coated with a SAOE thin film [33]. Dickens et al. used ZnS:Mn bulk rectangular specimen and performed a three-points bending test in accordance with the ASTM D790 standards in order to visualize the crack initiation and propagation through polymer doped matrices [87]. Crack propagation has been observed in unmanipulated concrete using mechanoluminescence paint and by conducting the three-points bending test [88].

2-5-4 Friction loading technique

Many researchers have carried out the tribo of friction techniques to apply the mechanical stress to the mechanoluminescence materials [4, 28-32]. Yamada et al. used a handmade testing machine to apply
the mechanical friction to a crystalline SAOE film. The friction was applied by a friction rod made of nylon. The rotational speed of the rod and the amplitude of the load on the film were adjustable. However, the actual friction force on the ML film was not measured.

![Friction Rod](image)

Figure 2-8: Light emission of the ML disc shaped specimen induced by mechanical friction, a) before friction, b) during friction and under dark environment (copied from [86])

2-5-5 Cyclic loading technique

Someya et al. focused on the rise time and decay times of SAOE materials during cyclic elastic deformation [26]. A commercial SAOE powder was mixed with optical epoxy resin and used to form disks. A compression test was repeated five times at intervals of three seconds. The study showed that the ML response did not significantly change from the 2nd to the 5th cycle [26]. Sohn et al. studied the harmonic response of the SAOED sensors under cyclic loading. In addition, hysteresis behavior was observed in the ML and was examined by comparison with the hysteresis that is typical in piezoelectricity [89].

Recently Kim and Kim investigated the ML response under the cyclic dynamic torsional loading [83, 90]. A rotational shaft, an AC servomotor, a power brake, and an in-line torque transducer were used to appropriately apply the cyclic torsional load to a ML rod specimen [11]. The experimental set up of the ML response to the dynamic torsional loading demonstrates in Figure 2-9.
Hydraulic pressure technique

Ono et al. applied a hydraulic pressure to a defected pipe coated with ML thin film in order to detect, localize, and quantify the internal defect in the pressurized pipe [12]. A high-performance liquid chromatography (HPLC) pump was used to increase the pipe hydraulic pressure and the internal pressure.
was measured by the pressure sensor. The experimental setup of the Ono et al. research is shown in Figure 2-10.

![Experimental setup of the ML response to the hydraulic pressure loading](image)

**Figure 2-10**: Experimental setup of the ML response to the hydraulic pressure loading (copied from [12])

### 2-5-7 Impact or impulsive loading technique

An impact technique can be used to excite the ML crystal [34-36]. Chandra and Zink utilized a piston impact technique [24, 25, 91]. An impulsive excitation test of ML was performed by using dropped ball through a guided cylindrical pipe [92].

### 2-5-8 Ultrasonic loading technique

Zhan et al. used an ultrasonic power to observe the light intensity change of the ML materials [37]. The ultrasonic power distribution on the surface of the transducer was visualized by using a thin ML film and a CCD camera.
Different kind of devices can be utilized in order to measure the light intensity of the mechanoluminescent materials, including: photomultiplier tubes (PMT), semiconductor diodes, Charge-coupled device (CCD) cameras, and spectrometers (spectroscope). Although PMTs and CCD cameras provide information regarding the light intensity, they do not give any information regarding the spectral content (i.e. wavelength) of the ML emission [76]. In this thesis, spectrometer, CCD camera, and photomultiplier tube are mainly utilized to measure the ML light emission. Thus, they are briefly described in the following manuscript.

2-6-1 Spectrometer

A spectrometer (spectroscope) is a device which can used to measure the properties of the light over a specific portion of the electromagnetic spectrum. Figure 2-11 shows a commercial spectrometer with an optic cable. These optical instruments can measure the light intensity as well as independent variables such as wavelength, wavenumber, electron volts and so on. The provided wavelength information from the spectrometer can be used to compute sample absorbance, transmittance, reflectance, and emittance.

![Figure 2-11: Commercial Spectrometer used to measure the light intensity characteristics](image)
2-6-2 CCD camera

A charge coupled device (CCD) is an integrated circuit etched onto a silicon surface forming light sensitive elements called pixels. A CCD camera is an analog electronic instrument that can be used as the image sensor which is a substitution of film in an electronic camera. The photon will be converted into the electron by CCDs and then stored in each pixel. The maximum number of electrons, which can be hold by each pixel, varies from 35,000 to as many as 500,000 depending on the model of the CCD. Photon striking silicon surfaces while the image is being integrated. The number of electrons stored in each pixel is proportional to the number of photons that struck that pixel. After completion of the exposure time, the electrons for each pixel are shifted out of the CCD and converted to a number, indicating how dark or light each particular pixel should be, and stored in the image file. The first CCD camera was invented in 1969 at Bell Labs by Willard Boyle and George Smith [93]. Figure 2-12 depicts two commercial high speed CCD cameras. They can capture up to 5000 frames per second.

The resolution of the camera is dictated by the resolution of image sensor. However, there are some interpolation software which can produce increased pixel counts by using multi-CCD sensors. The sensitivity to light of the sensor controls the ISO range (Gain level) that the camera can emulate.

Figure 2-12: Commercial CCD cameras used for measuring the ML light emission,

a) AVT Bigeye G-283B from Allied Vision, b) image pro X from LAVISION

One of the most important specifications of the CCD camera is the physical size of the sensor. Generally, larger sensors perform better at lower light levels. Also, they can receive more photons at any
given light level which allow us to capture better images in lower-light with reducing in light intensity noise. Similar to larger film formats, larger sensors require longer focal length and hence larger lenses, thus dictating the size (and weight) of the camera. Larger lenses may also increase the cost of the camera and use more power which yields to increasing battery requirements [94].

2-6-3 Photomultiplier tube

Photomultiplier tubes (PMT) are extremely sensitive light intensity detector in the ultraviolet, visible, and near-infrared spectral ranges. They are superior in response speed and sensitivity and widely used in analytical instruments and industrial measurement systems. Figure 2-13 shows an amplified photomultiplier from THORLABS.

![Photomultiplier tube](image)

Figure 2-13: An amplified photomultiplier from THORLABS, model: PMM01

A PMT is a vacuum tube consisting of an input window, a photocathode, focusing electrodes, an electron multiplier and an anode usually sealed into an evacuated glass tube [95]. Schematic construction of a photomultiplier is shown in Figure 2-14. In addition to their very high levels of gain, photomultipliers also exhibit a low noise level, high frequency response and a large collection area.
Figure 2- 14: Construction of a photomultiplier tube (copied from [95])

A PMT detects the light intensity and provides an output voltage through the following steps:

1) Light passes through the input window;
2) Light excites the electrons in the photocathode so that photoelectrons are emitted into the vacuum;
3) Photoelectrons are accelerated and focused by the focusing electrode onto the first dynodes where they are multiplied by means of secondary electron emission. This secondary emission is repeated at each of the successive dynodes;
4) The multiplied secondary electrons emitted from the last dynode are finally collected by the anodes.

2-7 Stress Visualization by Using ML Materials

Different techniques exist to visualize the stress distribution on structural surfaces including Digital Image Correlation (DIC) [1], photoelastic coatings [2], and Moire and interferometric methods [3]. ML materials can be used as an alternative non-contact sensing technology, for a direct view of stress distribution [5]. Xu et al. studied the stress distribution of a disc under a compression load by using SAOE materials. They used an ICCD (intensified couple-charged device) to capture images of the ML light emission. The comparison of the light intensity with a simulation result confirmed that such a ML image successfully reflects the stress distribution [5]. The measured ML intensity, gave a direct view of stress distribution even with naked eyes (see Figure 2-15). The visualization of stress distribution due to that
intense visible light emission was obtained by the application of only a single compression in atmosphere and has an advantage against the thermography which needs repetitive cycles of stress and thus is limited to evaluate stress in a fatigue process [96].

![Stress distribution of a bulk SAOE disc shaped specimen under a compression load](image)

Figure 2-15: Stress distribution of a bulk SAOE disc shaped specimen under a compression load (copied from [5])

If ML materials are coated on the surfaces of the host solid, each particle can act as a small sensor while the whole surface can reflect the stress distribution of the solid surface. The technique can be used to visualize the stress distribution of the solid when the stress is applied [22]. Figure 2-16 is demonstrates a comparison between the ML light emission of an open-hole specimen made by SAOE and Von-Mises stress distribution calculated from a FEM analysis.
Porotvin-Le Chatelier (PLC) effect was also visualized by using the ML technique [22]. The SAOE ML-sensing film was coated on aluminum alloy and a uniaxial tensile test was performed. The position, slope angle of the band front, and propagation velocity of the PLC bands were observed and measured by using a high-speed camera [22]. The effect of the PLC corresponding to the strain-stress curve can be seen in Figure 2-17.
Timilsina et al. used mechanoluminescent materials to determine mode I stress intensity factor (SIF) of a Compact Tension (CT) specimen according to the ASTM specification [97]. A mixture of the SAOE powder and acrylic resin with a weight ratio of 30% SAOE/resin was prepared and hot pressed by using a conventional specimen mounting machine. A sharp blade was used in order to provide a crack initiation point. A universal testing machine was utilized to apply the stress to the CT specimen. Figure 2-18 illustrates a series of images of the ML light emission from a SAOED in the vicinity of the crack tip of the CT specimen [97]. The load was applied at a constant loading rate of 80 Ns\(^{-1}\) and the images were captured by using a high speed camera.
Timilsina et al. accumulated the ML intensities of the images to evaluate the stress field in the vicinity of the crack tip. Sequential images of the accumulated ML emission can be shown in Figure 2-19 [97].

They also performed a uniaxial tensile test to study the effect of the stress rate on a bulk SAOE specimen. The effective stresses corresponding to the cumulative ML intensities in the compact tension test were extracted from the uniaxial tension test results. As expected the change of the ML light emission increases by increasing the applied stress rate (or load rate) [97]. Finally they provide the SIF from the ML
intensity based on deviator stress and hydrostatic stress. They claimed that the SIF or fracture toughness in mode I can be characterized without the fully understanding of the ML mechanism if the correct criterion is selected [97]. Sohn et al. coated a compact tension test specimen with a thin SAOED film by using pulsed laser deposition [98]. The crack on the CT specimen from the ML film was captured and visualized in their study.

Zhan et al. used ML technique to directly visualize the distribution of ultrasonic power [37]. They found a linear relationship between the ML light intensity of the SAOE thin film and the ultrasonic power. Figure 2- 20 demonstrates the cross-sectional view of the transducer, the images of the ML sensing film by using a CCD camera, and the distribution of the ML intensity along the X-axis [37].

![Figure 2-20](image)

Figure 2- 20: (a) Cross-sectional view of transducer (outer area: epoxy resin; central area: piezoelectric ceramic patch). (b) ML image recorded during ultrasonic vibration. (c) Distribution of ML intensity along X-axis (copied from [37])

Generally, the ultrasonic power exhibits an uneven distribution on the surface of a piezoelectric transducer. Acoustic radiation force produced by ultrasonic waves was calculated and compared with ML light emission. A visualized three dimensional distribution of the ultrasonic power output measured by ML sensing technique is illustrated in Figure 2- 21 [37]. Their study showed that ML technique has the potential to be performed in ultrasonic power measurements and other ultrasonic applications [37].
2-8 Visualization of the Crack Propagation by Using ML Materials

There are numerous efforts to observe and visualize the crack propagation by using the ML materials [9, 23, 33, 87, 88, 98-101]. Sohn et al. investigated the crack tip stress field of a CT specimen using SrAl₂O₄:Eu²⁺,Dy³⁺,Nd³⁺ materials [99]. An acute notch tip was introduced into the CT specimen using a very sharp blade so as to provide a crack initiation point. The load was applied by a load cell with the maximum load of 20 kgf. In their study, the crack tip stress field was shown in terms of ML at a certain stress intensity factor [99].

Kim et al. visualized the crack propagation and bridging stress distribution by using the ML paint and examine the mechanism of fracture in alumina ceramics [101]. A CT test was performed and a very high speed camera (15,000 frames per second) was employed to capture the micro scale images of the crack propagation. Sequence of high-speed images with the corresponding load values is depicted in Figure 2-22. The figure also demonstrates the corresponding bridging stress distribution converted from the ML light emission [101]. According to their research, it appears that the initial crack propagation arises in a noticeably short time which is less than 66 μs.
Lee et al. was able to visualize the crack propagation in a concrete beam with a notch by using the ML thin film [88]. A three point bending test was setup and the images were captured by a high speed camera.
camera. In addition to the ML paint, a crack-mouth-opening-displacement (CMOD) gauge was installed in order to compare the ML images with the crack opening displacement.

![Figure 2-23: Three point bending test setup of the ML paint in a notch concert beam (copied from [88])](image)

Crack length and crack growth speed was measured by using the ML images. According to their observation, the rate of crack propagation was much faster than the rate of unloading [88]. Dickens et al. made a small beam by using ZnS:Mn ML material and conducted a three bending point test to visualize the crack propagation [87].

The ML film was firstly applied onsite to a building as a non-destructive evaluation method by Terasaki et al. [9]. They investigated the ML application in construction field and monitored the influence of the drilling construction on a building. Two different types of ML as sheet-type and spray-film-type was placed on the crack position of a wall. A high speed-CCD camera was captured the ML images and the monitoring site was covered with a black curtain to provide the dark condition and prevent environmental light effects [9]. The performance of the ML sensor was also evaluated on daily deformation and crack mouth opening displacement in their study [9]. The results showed that ML sensing technique is a promising candidate for non-destructive evaluation.

The fracture can be predicted in a reinforced concrete by using ML sensor [33]. A four-point bending test was carried out and intense ML light emission of the SAOE film coated on the concrete
surface was found before the deep cracks propagated (see Figure 2-24) [33]. The process and direction of
 crack propagation were investigated and measured directly by using ML sensing film.

Figure 2-24: Real-time ML images of the coated reinforced concrete beam with four-point bending test
(copied from [33])

Terasaki et al. studied the application of the ML sensor on visualization of the active crack on an
old bridge for the first time [100]. The ML sheet type was placed on the main girder and the images were
acquired from the dynamic loading of the general traffic vehicles by using a CCD camera under the dark
condition. The results from that study showed that ML sensor can be applied not only to the visible crack
but also to invisible crack. The durability of the ML sensor was also investigated and the degradation of the
ML sensor was negligible during the application period for over 2 months [100].

Historical log of the fatigue crack opening and growth was recorded by using ML sensors [23].
Ueno et al. applied the ML sensor to a carbon fiber reinforced plastic (CFRD) composite pressure vessel
surface to visualize the fatigue cracks [102]. The vessels were stressed by periodical internal pressure until
a fatigue crack appears on the wall of a vessel. Difficulty of fatigue cracks detection in composite vessels
compared to the steel ones is that the fatigue crack first appears in the internal aluminum liner while being
covered with the outside CFRP layer [102]. If ML materials applied to the surface of the composite vessel, any internal defects or cracks can make stress concentration on the surface and will be visualized and detected from the observed ML images. Figure 2-25 exhibits the internal fatigue crack detection by using the ML sensors. In order to reduce the noises of the images acquired from the CCD camera, principal component analysis (PCA) method was utilized of the ML images for each fatigue examination cycle [102, 103]. PCA is an effective method to extract the pure ML pattern from the ML time series images [102].

![ML Image](image)

**Figure 2-25:** Detecting the internal fatigue crack on the CFRD composite pressure vessel by using the ML sensor (copped from [102])

2-9 Existing Models for Photoluminescence and Mechanoluminescence

Stress-sensitive ML sensors can visualize distributions of stresses and strains and can potentially be used for monitoring structural integrity in large structural health monitoring (SHM) systems. Several attempts have been reported to apply film-type ML sensors to the practical monitoring of civil
infrastructure [9, 100]. Following these practical applications of ML sensors, some efforts were made that focused on developing predictive models for converting the mechanical stress to the ML light emission values [26].

Although the luminescing mechanisms of the persistent luminescence (PL) and ML phenomenon are identical (i.e. recombination of detrapped carriers with Eu$^{2+}$ luminescence centers and their subsequent decay to the ground state), the PL and ML phenomena have different causes. PL is caused by thermal relaxation of the excited Eu$^{2+}$ ions and going back of electron from conduction band to valance band while ML is caused by band-bending/thermal de-trapping. Thus, the long lasting PL effects should be separately taken into account and characterized when proposing a predictive model.

2-9-1 Natural persistent luminescent models

As it is investigated in the literature, both SAOE and SAOED have a peak light intensity equal to wavelength 520 nm which is related to the green light [85, 104]. Figure 2-26 illustrates two phosphorescence spectra from SAOE and SAOED of the disc specimens under the non-loading condition. As it can be seen, SAOED shows a higher light emission than the SAOE. The peaks of spectra will be diminished by time.

![Figure 2-26: The phosphorescence spectra of the disc specimen of ML materials](image-url)
In the past, researchers studied the physical long-lasting PL decay phenomenon [105], and several models for predicting the afterglow of different phosphorescent materials were found to be available [72, 77, 106-108]. Figure 2-27 shows the experimental data of the decay behavior of the ML materials. According to the figure, the light intensity decreases exponentially as the time goes.

![Figure 2-27: Natural decay curve of the persistent luminescence (PL) of the phosphorescence materials](image)

Katsumata et al. proposed a predictive equation decay curve of SAOED phosphorescent materials by using Lorentzian fit [107]. The equation can be expressed as follow:

$$I = I_0 + A_1 \exp\left(-\frac{t}{\tau_1}\right) + A_2 \exp\left(-\frac{t}{\tau_2}\right)$$  \hspace{1cm} \text{Eq. 2-1}

where $I$ presents the phosphorescence intensity, $I_0$, $A_1$, and $A_2$ are the constants, $t$ is the time, and $\tau_1$, $\tau_2$ are the decay constants of the phosphor. It can be presumed from the above equation that $\tau_1$ and $\tau_2$ are representing two different trap depths of the electrons. The higher value of the two decay time constants ($\tau_1$ and $\tau_2$) is attributed to the higher density of deep traps and results in longer decay times, whereas the lower value is attributed to the higher density of shallow traps and results in shorter decay times [72].
Many researchers have investigated the ML phenomenon and developed the mathematical [6, 24, 35, 80-82, 91, 92, 109] or empirical [12, 20, 26, 83, 90, 104, 110] models. These models provide a promising interpretation of the ML and can yield to a calibration model which converts the ML light emission to the mechanical stress (or strain).

According to the past experimental researches, what triggers the ML is the loading rate (instantaneous load change), rather than the static load [4, 31, 32, 85] and more load rate (or strain rate) yields to more light intensity of ML materials (see Figure 2-28). This observation motivated Kim et al. to propose a rate-equation model. Their predictive model is a loading-rate-dependent equation [20]. In their study, a mixture of the epoxy resin and SAOED powder was hot presses using a conventional specimen-mounting machine and a typical tensile specimen was prepared. They used a CCD camera to capture the ML images during the loading. Their results showed that the relationship between the loading rate and the ML intensity was not simply linear.

![Figure 2-28: ML intensity versus applied load of a tensile test (copied from [20])](image-url)
The mathematical rate equation of the ML process proposed by Kim et al. can be expressed as follows [20]:

\[
\begin{align*}
\frac{dN_{Eu^{2+}}}{dt} &= K_T \frac{dP}{dt} N_{Eu^{2+}} N_{Dy^{3+}} - K_P N_{Eu^{2+}}, \\
\frac{dN_{Eu^{3+}}}{dt} &= K_P N_{Eu^{2+}}, \\
\frac{dN_{Eu^{+}}}{dt} &= \frac{dN_{Dy^{3+}}}{dt} - \frac{dN_{Dy^{4+}}}{dt} = -K_T \frac{dP}{dt} N_{Eu^{+}} N_{Dy^{4+}}
\end{align*}
\]

where \( N_{Eu^{2+}} \) and \( N_{Eu^{+}} \) are the number densities of divalent europium activators in the excited and the ground states. \( N_{Eu^{+}} \) is the number density of univalent europium activators. \( N_{Dy^{3+}} \) and \( N_{Dy^{4+}} \) are the number densities of trivalent and tetravalent dysprosium ions. \( P \) is the instantaneous load. As previously mentioned, the load \( P \) could be approximated to be proportional to strain (or stress), since the ML took place in the elastic regime. \( K_T \) is the constant for the interaction (or recombination) term. \( K_P \) is the long phosphorescence rate constant, which was obtained from long phosphorescence measurement without loading. The instantaneous loading rate \( (dP/dt) \) was obtained by numerically differentiating the measured load profile with respect to time. The nonlinear term stands for the interaction between the trap-capturing hole (tetravalent dysprosium ions) and the ionized activators (univalent europium ions). Their results showed that the predicted rate equation matched well with experimental data [20].

Xu derived the following equation which reflects the relation between the luminescence intensity, strain, and strain rates [104].

\[
S_{ML} = C_0 \varepsilon \frac{d\varepsilon}{dt}
\]

where \( S_{ML} \) is the ML intensity, \( \varepsilon \) is the strain, \( t \) is the time, and \( C_0 \) is the normalization factor. Kawai et al. have used the above equation and proposed a method of the Split Hopkinson Pressure Bar (SHPB) experiments to measure the local strain-rate distributions by using mechanoluminescence materials, combined with a high-speed camera and image intensifier [110]. The effect of strain and strain rate are
considered in Eq. 2-3, however, the linear relation between the ML and the strain and strain rate may not be ideal. Ono et al. integrated the Eq. 2-3 with respect to time and derived the following equation [12].

\[ A_{ML} = C_1e^{-2} = \int S_{ML} dt \]  

Eq. 2-4

According to the above equation, \( A_{ML} \) is proportional to the square of the strain and the strain distribution can be obtained without considering the strain rate. The hoop strain distribution on the outer surface of a defective pipe was acquired by using ML film and performing the Eq. 2-3. The experimental results from the proposed model were compared and verified with those from strain gage and calculated results from commercially finite element software [12].

The ML light intensity increases by increasing the applied stress or loading rate and decreases with repetitive loading. However, it can be recovered after being irradiated with UV light at room temperature [26]. Someya et al. proposed the lifetime-based measurement of stress using ML materials. They used the rise time of ML, \( \tau_{up} \), and the decay time, \( \tau_{down} \), which were individually calculated using the least square method [26]. The proposed model can be expressed as:

\[ I(t) = Ae^{-t/\tau} + C(\tau_{up} - \tau_{down} : \tau) \]  

Eq. 2-5

where \( I(t) \) is the ML light intensity, \( t \) is time, and \( A \) and \( C \) are the calibrated constants. They claimed that lifetime-based measurements can decrease by time and are not dependent of the concentrations and excitation power [26]. They also found the harmonic response of the ML light emission under the cyclic loading (see Figure 2-29) [89].
Figure 2-29: Harmonic response of the ML light intensity (red line) to the applied cyclic load (black line) at a frequency of 1 Hz (copied from [89])

They also performed the fast Fourier transform analysis (FFT) of the ML light emission response. The FFT of the ML response showed a series of prominent peaks around the frequency of applied load and their integral multiples, as shown in Figure 2-30.
The following equation was introduced by Sohn et al. which predicts the harmonic behavior of the ML under cyclic loading [89].

\[
ML(t) = \left[\alpha P(t) + \beta P(t)^2\right] D(t),
\]

\[P(t) = e^{i\omega t},\]

\[D(t) = [\eta e^{-t/\tau_1} + (1 - \eta) e^{-t/\tau_2}],\]

\[\alpha = \alpha_e - i\alpha_o, \quad \delta_1 = \tan^{-1}(\alpha_e / \alpha_o),\]

\[\beta = \beta_e - i\beta_o, \quad \delta_2 = \tan^{-1}(\beta_e / \beta_o),\]

where \(P(t)\) is the normalized applied load and \(D(t)\) is the decay function consisting of two exponential decay components. \(\tau_1\) and \(\tau_2\) are the decay times for each of the exponential decay components. \(\omega\) is the angular frequency of the applied load. \(\delta_1\) and \(\delta_2\) are the phases for \(ML_1\) and \(ML_2\) with respect to the applied
load, and \( \eta \) is a simple amplitude weight constant. \( \alpha \) and \( \beta \) are complex constants for \( ML_1 \) (the first deconvoluted component) and \( ML_2 \) (the second deconvoluted component) relating to \( \delta_1 \) and \( \delta_2 \), respectively. The actual load was a tensile type cyclic load such that the ML response was always positive and the minimum load was approximately zero. They have also interpreted the ML loss in terms of hysteresis behavior [89].

Kim and Kim introduced a new simple empirical equation explaining the ML light intensity as a function of the applied torque load as follows [83]

\[
ML = \alpha T + \beta \dot{T} + \gamma (T\ddot{T})
\]

Eq. 2-7

where \( \alpha, \beta, \) and \( \gamma \) are the regressed coefficients, \( T \) is the applied torque, and \( \dot{T} \) is the rate of the applied torque. In their study, genetic algorithm (GA) was employed to optimized the constant coefficients by using root mean square error (RMSE) [83]. In their recent study, they investigated the harmonic response of the ZnS:Cu under torsional cyclic loading and provided a predictive empirical equation between the torque input \( T(t) \) and PMT output (ML intensity) \( M(t) \) as follows [90].

\[
\dot{M} = \alpha |\dot{T}| (f(T) - M) + \dot{T}g(T),
\]

Eq. 2-8

where \( f(T) \) and \( g(T) \) are functions that can determine the shape of the hysteresis loop, and these functions can be chosen as

\[
f(T) = aT, \quad g(T) = b,
\]

Eq. 2-9

where \( a \) and \( b \) are constants. Figure 2-31 shows the ML light emission response to the applied sinusoidal input torque and also the ML intensity-torque hysteresis loop. As it can be seen in the figure, the ML intensity responded with a phase delay.
They evaluated the measured torque results from their predictive model and claimed that their model can accurately estimate the torque values from the output voltage of the PMT sensor generated by ML light emission. Although they were able to measure the torque applied to the rotational shaft, the ML particle light emission is dependent on the applied stress. In a rotational shaft, the shear stress on the shaft’s surface is relative to the radius of the shaft, the applied torque and the torsional rigidity. Hence, their model should be modified in order to measure the shear stresses on the shaft’s surface.

2-10 Exiting Techniques for Full-field Strain Measurements

There are several full-field strain measurement methods including Digital Image Correlation (DIC) [1], photoelastic coatings [2], and Moire interferometric methods [3]. In this section, common full-
field strain measurement techniques are briefly described and their advantages and disadvantages are
highlighted.

2-10-1 Digital Image Correlation method

Digital image correlation (DIC) is an optical method that can be used to measure the displacement
of a structure in response to loading conditions in order to perform deformation analysis. DIC involves
tracking of points on the surface of a specimen based on the gray-scale pixel values in images taken of the
specimen before and after the load is applied. The tracking is done by identifying an area of an image
around a surface point of interest, and then finding the area in a second image, taken under load, that most
closely correlates with the first. Full-field in-plane displacement of a structure can be measured by using
DIC to measure displacements at nodal points of an arbitrarily dense mesh. Figure 2-32 schematically
shows the DIC working system which consists of the specimen with the speckles on, CCD cameras, and the
correlation software [111].

![Figure 2-32: Schematic DIC working principle system (copied from [111])](image_url)
DIC techniques have been most widely used in various applications for its non-contact sensing ability and relative ease of implementation and use. DIC can also provide the 3D displacement and velocities as well as all the surface strain values and it is the ideal solution for determination of material properties, component analysis and verification of finite element analysis.

In DIC, strains are computed results from measured displacement fields. The resolution of the displacement (strain) from DIC depends on camera resolution, lens optical quality, and the quality and size of the speckles on the specimen surface. Besides these, the strain accuracy is very dependent on the facet sizes and facet steps and they should be adopted with the surface pattern accordingly. Failure to assign the appropriate facet parameters yields to inaccurate strain results. By increasing the facet size, the accuracy of the resulting measuring points improves, but the computation requires more time. Moreover, the local effects within the facet size cannot be captured and less strain can be calculated in the marginal area and adjacent points around the boundary. Furthermore, DIC is very sensitive to fluctuation and nonuniformity of the light intensity and the calculation process is tedious and time consuming.

2-10-2  Photoelastic coating method

Photoelastic coating method is a widely used full-field technique for accurately measuring surface strains to determine the stresses in a part or structure during static or dynamic testing. A flat or curved surface of specimen will be coated with a thin layer of birefringent material (usually a polymer) for doing the stress analysis. Then, as the load is applied to the specimen, the coating is illuminated by polarized light from a reflection polariscope. When viewed through a white light reflection polariscope, the strained coating exhibits black isoclinic and colored isochromatic fringes. Full field isochromatic isoclinic patterns are directly and instantly visible and can be photographed, processed and analyzed by using a digital compensator to determine the fringe order value with appropriate software to translate the data into the stress and strain levels. Figure 2-33 schematically demonstrates the polariscope reflection in photoelastic method. Sequential images of a loaded specimen using the photoelastic method can be seen in Figure 2-34. A software analyzer needs to be used in order to convert the illumination the actual stress or strain levels.
The photoelastic coating method has many advantages compared to other methods of experimental stress analysis. It provides a full field quantitative data, which enables to determine the complete distribution of surface strains and their directions, and directly highlighting severely strained areas.
Moire interferometry is a very versatile and effective method for determining in-plane and out-of-plane displacement and strain fields [114, 115]. The term Moire is a French word referring to an irregular wavy finish usually produced on a fabric by pressing between engraved rollers. In optics it refers to a beat pattern produced between two gratings of approximately equal spacing. [116]. Moire fringes are formed by the superposition of two gratings, for instance, two arrays of uniformly spaced lines. These fringes are contour maps of the difference between the gratings and can be used in determining of the surface deformations. Moire measurements are performed routinely in the interferometric domain with fringes representing subwavelength displacements per contour. Since Moire responds only to geometric changes, it is equally effective for elastic, viscoelastic, and plastic deformations, for isotropic, orthotropic and anisotropic materials, and for mechanical, thermal, and dynamic loadings. Figure 2-35 shows the moire pattern (or beat pattern) produced by two identical straight-line gratings rotated by a small angle relative to each other. A dark fringe is produced where the dark lines are out of step one-half period, and a bright fringe is produced where the dark lines for one grating fall on top of the corresponding dark lines for the second grating. If the angle between the two gratings is increased, the separation between the bright and dark fringes decreases.

Figure 2-35: (a) Straight-line grating. (b) Moire between two straight-line grating of the same pitch at an angle $\alpha$ with respect to one another (copied from [116])
CHAPTER III
EXPERIMENTAL METHODS

3-1 Overview

In this chapter the experimental methods of preparing the ML composite and ML sensing thin film is explained. The instruments for measuring the light emission from the ML materials are described. Different mechanical testing methods are explained and illustrated on ML composite and aluminum specimens coated with ML film.

3-2 Manufacturing of ML Ceramic Powders

In this section, the manufacturing process of two different ML particles SrAl₂O₄:Eu²⁺ and SrAl₂O₄:Eu²⁺,Dy³⁺ is described.

3-2-1 SrAl₂O₄:Eu²⁺

The manufacturing process of SrAl₂O₄:Eu²⁺ (SAOE) particle is shown in Figure 3-1. SrAl₂O₄:Eu (SAOE) was manufactured by the solid-state reaction method as follows. First, high purity raw materials SrCO₃ (99.995%), Al₂O₃(>99.99%) Eu₂O₃ (>99.99%) were mixed with Ethanol (CH₃CH₂OH) and 1% weight Boric Acid (H₃BO₃) flux and went through ball milling. In order to evaporate water, the mixture was dried for one hour at 50º C. After that, the dried mixture was calcinated at 900º C for one hour within open furnace.
Heating and cooling speed is set to 5°C/min. Once it is cooled down to room temperature, fill the furnace with Ar+5%H₂ mixed gas with one end of the open furnace closed. Perform sintering keeping at 1300°C for four hours. Heating and cooling speed is kept at 5°C/min as previously. During sintering, Ar+5%H₂ mixed gas needs to be flowed into the furnace at a constant speed. Generally, the flow speed of the mixed gas is set to 200-500 ml/min. The reason for continuously flowing gas is for removing decomposed materials and at the same time helping with sintering at a consistent air condition. After sintering and cooling down to the room temperature, crush up the solidified sample with mortar and pestle and by using the rotavapor-drying method, perform ball milling for 24 hours not separating the materials. Finally, in order to evaporate the remaining ethanol solvent, perform vacuum drying at 100°C. Figure 3-2 shows the SAOE powder used in this thesis. This SAOE powder shows a very faint light emission under non-loading condition in the dark environment.
3-2-2 $\text{SrAl}_2\text{O}_4: \text{Eu}^{2+}, \text{Dy}^{3+}$

$\text{SrAl}_2\text{O}_4: \text{Eu}^{2+}, \text{Dy}^{3+}$ (SAOED) powder used in this study was commercial phosphorescent material named LumiNova which was invented and developed by NEMOTO & CO. Ltd., of Tokyo, Japan and imported and distributed in United States by United Mineral & Chemical Corporation (UMC). This SAOED phosphor shows very strong and long-lasting photoluminescence afterglow. The body color of the particles is light yellowish green with the average size of 10-40 $\mu$m. The excitation source wavelength should be between 200 and 450 nm and the peak emission wavelength is around 520 nm. The ML particles have a very good chemical stability except against the water. Detailed molar ratios of the rare earth doped elements europium ($\text{Eu}^{2+}$) and dysprosium ($\text{Dy}^{3+}$) are not available from the manufacture. Figure 3-3 depicts the commercial SAOED powder used in this thesis under the room light and dark environment conditions. It is clear that the powder shows a strong photoluminescence afterglow in dark environment.
3-3 Manufacturing of ML Sensing Films

In this section, the manufacturing process of making the ML sensing film by using the doctor blade method is described.

3-3-1 Polymer matrix for binding ML particles

In order to make a ML sensing film, ML powder should be mixed in an optical epoxy. The selection of the suitable polymer resin is essential in ML sensor fabrication. In selecting the appropriate polymer as the matrix of the composite to embed the ML particles, some important factors are taken into account such as 1) high transmittance in UV(for excitation) and visible (for emission) region, 2) adequate impact strength, 3) stiffness for effective transferring of mechanical stress to ML particles, and 4) easiness for solution casting. The transmittance of the polymer directly affects the absorption and emission of the composites. Three different commercial optical epoxies have been selected and tested under tension loading and the sensitivity and change of the ML light emission of each sample is evaluated under the same loading condition. These three epoxies are: 1) 105 epoxy resin and 206 hardener (West System), 2) High performance fiberglass (FiberGlass Coating, Inc.), and 3) High Gloss Epoxy resin (Glaze Coat). In the first step, three aluminum dog-bone specimens were coated with the three different epoxies mixed with SAOED powder by the mass concentration ratio of 1:1. Standard tensile tests with a fix loading rate have been
conducted to see the change of the ML light emission from each specimen. Among the three epoxies, the high performance fiberglass did not show any change of light intensity under the loading in the linear-elastic range of the aluminum. Hence, for further investigation of the performances of the epoxies, tensile bare film tests have been conducted only on the epoxies from West System and Glaze Coat. Two different ML films with different epoxy matrixes can be seen in Figure 3-4. The mass concentration ratio of the ML particles to the epoxies is selected 1 to 1.

Figure 3-4: tensile test of the bare ML filme test with different epoxies (left specimen: ML particles mixed with Glaze coat, right specime: ML particles mixed with West System)

Figure 3-5 demonstrates the light emission performances of the ML particles mixed into the two different epoxy matrixes. The loading rate is fixed at 0.5 mm/s for each test. As it can be seen in the figure, the change of the ML light intensity from the specimen with West System epoxy is more than the one from Glaze Coat.
After evaluating the performances of the ML particles in different epoxy matrixes, the epoxy from West System has been selected as final choice because of a higher stiffness and higher ML sensitivity. The weight ratio of resin to hardener is 5:1. The mass ratio of the epoxy to the ML powder is different from 1:5 to 1:1, and the powder is dispersed uniformly in the epoxy by using a magnetic stirrer.

3-3-2 Doctor Blade Method

Doctor blade method is one of the widely used techniques for producing thin films on a substrate. This method was originally developed during the 1940’s as a method of forming thin sheets of piezoelectric materials and capacitors [117] and is now an accepted precision coating method. Figure 3- 6 schematically descripts the doctor blade method of manufacturing the thin film.

To make a film, an applicator with a blade is placed at the left side of the plastic substrate. The mixed epoxy solution with the ML powder is deposited on the substrate directly in front of the knife-blade. Then, the applicator is dragged on the surface toward the right side of the substrate at an approximate uniform rate of 10 to 12 inches (250- 300mm) per second and the coating solution is spread on the substrate. The epoxy film will be cured and dried for 24 hours in the room temperature.

Figure 3- 5: Stress-Ligh intenisty curves for the epoxies from West Sytem and Glae Coat.
A finely machined stainless steel knife-blade fitted into slots in the end section of applicator that allows vertical adjustment of the blade. Rare earth magnets hold the stainless steel blade firmly in place and two finely-made micrometer barrels can be easily set so that gate clearances can be adjusted from 0 to 0.250" in increments of 1 mil (.001") with resolution to 1/2 mil (.0005"). Figure 3-7 demonstrates the Doctor Blade applicator (from Montipower Test Equipment & Specialty Tool) and a thin ML film manufactured by this method.

Figure 3-7: a) Doctor blade applicator, b) manufactured ML thin film by using doctor blade method
3-4 Scanning Electron Microscopy (SEM) Characterization

X-ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and provides information on unit cell dimensions. Figure 3-8 shows a XRD pattern of SAOE powder. It confirms the desired composition and structure of the SAOE samples. Comparing with XRD pattern from other literatures [32], the current sample has less left-over Al₂O₃ in SAOE samples. The comparison results are considered to be an advantage since it means that less left-over un-reacted Al₂O₃ is presented in the sample. According to the inspections of SEM images of SAOE particles (See Figure 3-9), the size of most particles ranges from 1 μm to 20 μm. Figure 3-10 shows the SEM imaged of the SAOED particles. According to SEM analyses, the size of SAOED particles ranged from 2 μm to 60 μm. The SEM images of the dispersed SAOE and SAOED particles in epoxy matrix from the West System can be seen in Figure 3-11 and Figure 3-12, respectively. The mixing mass ratio of the ML powder to epoxy is selected as 1 to 3. According to the figures, both SAO and SAOED particles were observed to be well dispersed in the epoxy resin.

![XRD pattern of SAOE after sintering and PDF plots of SrAl₂O₄ (00-034-0375) and Al₂O₃ (00-046-1212) from ICDD cards [15]](image)

Figure 3-8: XRD pattern of SAOE after sintering and PDF plots of SrAl₂O₄ (00-034-0375) and Al₂O₃ (00-046-1212) from ICDD cards [15]
Figure 3-9: SEM images of the SAOE particles
Figure 3- 10: SEM images of the SAOED particles
Figure 3- 11: SEM images of the SAOE particles dispersed in epoxy
Figure 3-12: SEM images of the SAOED particles dispersed in epoxy
Material Tests for Characterization of ML Specimens

Characteristics and behavior of ML composite or thin ML film coated on substrate have been studied under various materials testing. These different mechanical testing methods are described in the following manuscripts. In all methods, the max load is selected in a way that materials remain in a linear-elastic range.

3-5-1 Tension test

A standard test method for testing aluminum alloy is performed, following the ASTM B557-10, to study the behavior of the ML thin film under uniaxial tensile loading (see Figure 3-13). A universal UTM-Hydraulic testing machine (Instron-1000HDX) is used to conduct the tension test. The Instron machine, hydraulic pomp and controller box depicts in Figure 3-14. Data obtained from the ML response by this method are used to train the Genetic programming (GP) in order to develop the predictive ML transduction and calibration models.

Figure 3-13: Tension test of aluminum specimen coated with ML thin film (dimensions are in inches)
3-5-2 Compression test

In order to study the performance of ML particles under compression loading, compression tests are performed on ML composite specimens. A different testing machine (Instron-5582) was used to perform the compression tests (see Figure 3- 16). All samples were made in a circular disk form having the diameter of 33 mm and the thickness of 10 mm (see Figure 3- 15). Data obtained from the ML response by this method is used to study the loading rate effect on ML materials and also to compare the SAOE and SAOED performances.
Figure 3-15: Compression test on a ML composite disc shaped specimen

Figure 3-16: a) The universal testing machine (Instron 5582) used in this thesis to conduct the tensile tests

b) Compression test platens

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3-5-3 Pure shear test

A standard test method for testing aluminum alloy is performed, following the ASTM B31-05, to study the behavior of the ML thin film under shear stresses (see Figure 3-17). Slotted single shear test fixture is used to mount the specimen and an Instron testing machine is used to conduct the test. Data obtained by this test method are used to verify and validate the proposed calibration model.

![Figure 3-17: Standard shear test of aluminum specimen coated with ML thin film (dimensions are in inches)](image)

3-5-4 Stress concentration test

An open-hole aluminum alloy specimen with coated with ML thin film is tested under tension loading to study the stress concentration effect on ML performance. Figure 3-18 shows the aluminum open-hole specimen coated with SAOE film. An Instron testing machine is used to apply the tension load to
the specimen. Data obtained by this test method are used to verify and validate the proposed calibration model.

Figure 3-18: Stress concentration of aluminum open-hole specimen coated with ML thin film (dimensions are in inches)

3-6 Measurements of ML Light Intensity

Different kinds of devices can be utilized in order to measure the light intensity of the ML sensors, including: photomultiplier tubes (PMT), semiconductor diodes, charge-coupled device (CCD) cameras, and spectrometers (spectroscopes). In this thesis, a spectrometer and a high speed CCD camera were mainly utilized to measure the ML light emission.

3-6-1 Spectroscope

The spectroscope (spectrometer) from StellaNet (BLK-CXR) used to measure the change of the light intensity from the ML particles is depicted in Figure 3-19. A cosine receptor (CR2) is also connected to the spectrometer by an armored fiber optic cable. Data from spectroscope is transferred to the laptop.
through a USB 2 interface connection. Spectral analyzer software (SpectraWiz) was performed for data analysis of the light intensity. A screenshot of the software used in this thesis for the spectral analysis is shown in Figure 3-20.

Figure 3-19: Spectrometer used for measuring the light emission properties from the ML materials

Figure 3-20: Screenshot of the SpectroWiz software for spectral analysis
3-6-2 High speed CCD camera

A high speed CCD camera (AVT Manta G-033B) from Allied Vision Technology is used with a consistent gain level setting. The ICX414 CCD sensor is placed behind the optical lens. All the images are captured in a gray level at frame speed up to 88 fps. The data from CCD camera is transferred to the computer via an Ethernet cable (see Figure 3- 21). The camera can receive an external trigger event though the pin connector. Using an Instron digital input/output board, the CCD camera and controller of the Instron testing machine are synchronized to achieve a perfect coincidence between the captured images and the load step data. The camera operating temperature is between 5 and 45 °C. Figure 3- 22 reflects the detail specifications of the high speed CCD camera used in this thesis.

Figure 3- 21: A commercial CCD camera from Allied Vision Technology used for measuring the ML light emission
<table>
<thead>
<tr>
<th><strong>Manta</strong></th>
<th><strong>G-033</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Interface</strong></td>
<td>IEEE 802.3 1000BASE-T, IEEE 802.3af (PoE) optional</td>
</tr>
<tr>
<td><strong>Resolution</strong></td>
<td>656 x 492</td>
</tr>
<tr>
<td><strong>Sensor</strong></td>
<td>Sony ICX414</td>
</tr>
<tr>
<td><strong>Sensor type</strong></td>
<td>CCD Progressive</td>
</tr>
<tr>
<td><strong>Sensor size</strong></td>
<td>Type 1/2</td>
</tr>
<tr>
<td><strong>Cell size</strong></td>
<td>9.9 μm</td>
</tr>
<tr>
<td><strong>Lens mount</strong></td>
<td>C/CS-Mount</td>
</tr>
<tr>
<td><strong>Max frame rate at full resolution</strong></td>
<td>88 fps</td>
</tr>
<tr>
<td><strong>A/D</strong></td>
<td>14 bit</td>
</tr>
<tr>
<td><strong>On-board FIFO</strong></td>
<td>32 MB</td>
</tr>
<tr>
<td><strong>Output</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Bit depth</strong></td>
<td>8-12 bit</td>
</tr>
<tr>
<td><strong>Mono modes</strong></td>
<td>Mono8, Mono12, Mono12Packed</td>
</tr>
<tr>
<td><strong>Color modes YUV</strong></td>
<td>YUV411, YUV422, YUV444</td>
</tr>
<tr>
<td><strong>Color modes RGB</strong></td>
<td>RGB24, BGR24</td>
</tr>
<tr>
<td><strong>Raw modes</strong></td>
<td>BayerRG5, BayerGR12, BayerRG12Packed</td>
</tr>
<tr>
<td><strong>General purpose inputs/outputs (GPIOs)</strong></td>
<td>2 inputs, 2 outputs</td>
</tr>
<tr>
<td><strong>Opto-coupled I/Os</strong></td>
<td>2 inputs, 2 outputs</td>
</tr>
<tr>
<td><strong>RS-232</strong></td>
<td>1</td>
</tr>
<tr>
<td><strong>Operating conditions/Dimensions</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Operating temperature</strong></td>
<td>+5 °C ... +45 °C</td>
</tr>
<tr>
<td><strong>Power requirements (DC)</strong></td>
<td>8 V - 30 V</td>
</tr>
<tr>
<td><strong>Power consumption (12 V)</strong></td>
<td>&lt;3.6 W</td>
</tr>
<tr>
<td><strong>Mass</strong></td>
<td>&lt;200 g</td>
</tr>
<tr>
<td><strong>Body Dimensions (L x W x H in mm)</strong></td>
<td>86.4 x 44 x 29 mm incl. connectors</td>
</tr>
<tr>
<td><strong>Regulations</strong></td>
<td>CE, FCC Class B, RoHS</td>
</tr>
</tbody>
</table>

Figure 3-22: Detail specification of the Manta G-033 CCD camera from Allied Vision Technology [119]
CHAPTER IV
EXPERIMENTAL SETUP AND PROCEDURES

4-1 Overview

In this chapter the experimental setup and procedures of composite disc shape ML samples and thin ML sensing film coated on aluminum host specimens are described. Samples preparation and test matrix are presented in details. Image processing of ML thin film is introduced and the possible source of experimental errors is explained.

4-2 Disc Specimens

Two different types of composite circular disk-shaped specimen are manufactured using SAOE and SAOED ML particles and the behavior and performance of these two materials are studied under compression.

4-2-1 Samples preparation

SAOE and SAOED powders were mixed with a commercial optical epoxy resin (West System 105 epoxy resin and West System 206 hardener). The mass ratio of the epoxy resin to the ML powder is 3:1, and the powder is dispersed uniformly in the epoxy using a magnetic stirrer. Circular disk-shaped specimens are cured in a mold until they are fully hardened. The diameter of the circular disk specimens is 33 mm and thickness is 10 mm.

4-2-2 Test setup

Experimental test setup is depicted in Figure 4-1. An Instron tester machine is used for compression tests. It is connected to a control computer where users can select various test parameters
related to the test modes (displacement or force-controlled), the upper loading or displacement limit, the loading profile, strain rates, etc. Force-displacement data are also measured to compare with light intensity versus time in the control computer. To measure the light intensity, a spectrometer from StellaNet (BLK-CXR) was used. A cosine receptor (CR2) is also connected to the spectrometer by an armored fiber optic cable. Spectral analyzer software (SpectraWiz) is used in a laptop computer for data analysis of the light intensity. As shown in Figure 4-1, the cosine receptor is attached to the upper loading block at a constant distance from the sample face in order to measure light intensity of samples. During the loading period, changes of the light intensity are continuously measured at a constant integration time 500 ms for all loading rates.

![Figure 4-1: Test configurations of ML effect of SAOE and SAOED composite specimen](image)

### 4-2-3 Experimental test matrices

Two test matrices are created for testing and comparison of SAOE and SAOED samples. In the first set of experiments, we investigate the repeatability, linearity and sensitivity in the low loading rates. In this test matrix, two specimens (i.e. one for each of SAOE and SAOED) are tested. As shown in Table 4-1, “SAOE-SP1-x” means xth test of SAOE specimen 1. The number of tests per each loading rate is summarized in Table 4-1. Tests at the lowest loading rate are conducted to evaluate the minimum loading
rate for ML phenomena. Only one specimen is repeatedly used, mainly in order to check the repeatability of ML phenomena. The upper loading limit is set to 3000 N.

Table 4-1: Test matrix I

<table>
<thead>
<tr>
<th>Loading rate</th>
<th>0.5 mm/min</th>
<th>1 mm/min</th>
<th>2 mm/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAOE-SP1-x</td>
<td>Three tests: (x=1,2)</td>
<td>Three tests: (x=1,2)</td>
<td>Two tests: (x=1,2)</td>
</tr>
<tr>
<td>SAOED-SP1-x</td>
<td>Two tests: (x=1,2)</td>
<td>Three tests: (x=1,2)</td>
<td>Three tests: (x=1,2)</td>
</tr>
</tbody>
</table>

In the second test matrix, five SAOE and five SAOED samples are tested at different loading rates (2, 4, 6, 8, 10 mm/min). Table 4-2 shows the second test matrix for the disc specimens. These loads are used as the high loading rate to evaluate the samples. For each sample, three tests are conducted to check the accuracy and also repeatability for each SAOE/SAOED sample. Due to unavoidable manufacturing errors, the dimension of samples could slightly vary from sample to sample. The upper loading limit is also set to 3000 N.

Table 4-2: Test matrix II

<table>
<thead>
<tr>
<th>Loading rate</th>
<th>2 mm/min</th>
<th>4 mm/min</th>
<th>6 mm/min</th>
<th>8 mm/min</th>
<th>10 mm/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAOE-SP(_z) (z=1,2,3,4,5)</td>
<td>(z=1,2,3,4,5)</td>
<td>(z=1,2,3,4,5)</td>
<td>(z=1,2,3,4,5)</td>
<td>(z=1,2,3,4,5)</td>
<td></td>
</tr>
<tr>
<td>SAOED-SP(_z) (z=1,2,3,4,5)</td>
<td>(z=1,2,3,4,5)</td>
<td>(z=1,2,3,4,5)</td>
<td>(z=1,2,3,4,5)</td>
<td>(z=1,2,3,4,5)</td>
<td></td>
</tr>
</tbody>
</table>

4-3 Thin Film Specimens

Thin ML sensing film manufactured by using doctor blade method is bound on different host specimens. Different type of aluminum samples (standard dog-bone specimen, open-hole specimen, shear test specimen, etc.) are prepared and tested for different purposes. Tensile uniaxial tests are performed to apply stress on aluminum test. The strains on host aluminum samples are equal to the strains on ML sensing film.
4-3-1 Sample preparation

Commercial SAOED powder materials (LumiNova G-300M, United Mineral & Chemical Corporation) are used to manufacture thin ML sensing film. The SAOED powder is mixed with a commercial optical epoxy resin (West System 105 epoxy resin and West System 206 hardener) and thin ML sensing film with different thicknesses is manufactured by the doctor blade method. The mass ratio of the epoxy resin to the SAOED powder varies from 1:5 to 1:1. The powder is dispersed uniformly in the epoxy using a magnetic stirrer. Different aluminum specimens are coated with the cut ML sensing film by using a commercial adhesive (M-Bond 200 from Micro-Measurements).

4-3-2 Test setup

During tension tests of the coated aluminum specimens, images are obtained at a fixed frame per second by using a charge-coupled device (CCD) camera (AVT Manta G-033B) with a consistent gain level setting, and Vision Builder AI software (National Instruments) is used for capturing the images. The camera is positioned approximately 26 cm from the specimens with an exposure time of 0.1 ms and 10 fps. Using an Instron digital input/output board, the CCD camera and controller of the Instron testing machine (220 kips) are synchronized to achieve a perfect coincidence between the captured images and the load step data. Figure 4-1 shows the schematic experimental test setup and procedures.
The ambient light from the environment is completely blocked to minimize errors and inconsistencies of the ML light intensity during the test. The ML sensing film is consistently excited with a 40-watt commercial lamp; two minutes was enough for acquiring fully photoexcited ML light intensity. However, even if it is not fully photoexcited (i.e. charged), the ML sensing film can still emit light under mechanical stresses because the mechanical stress is one of the excitations that generate separated charges (i.e. electrons-holes) like photoexcitation. If it is not fully photoexcited, changes of the ML light intensity may not be consistent due to different initial ML light intensity and effects of stress-free PL decay. Therefore, in our test method, before any tests are carried out, the ML sensor is fully photoexcited. The wavelength of the excitation light source is between 400 and 1000 nm and the wavelength peak is around 600 nm. After full excitation, stress-free persistent luminescence (PL) light is emitted that displays a naturally decaying intensity with respect to time. The maximum load is limited to 15 kN (230 MPa on an
aluminum specimen) so that the aluminum specimen is within the linear elastic range. The maximum load is applied at different strain rates and different stress-free PL decay time intervals. For example, after full photoexcitation of the ML sensing film, the different time intervals are elapsed until the onset of the loadings.

For practical application of ML sensors to the measurement of full-field strains, a dark chamber box equipped with a photoexcitation lamp, controller and CCD camera can effectively block environmental light and is able to measure ML light intensity in consistent conditions.

4-3-3 **Image processing**

The images from the ML thin film are captured at a fixed frame rate by the CCD camera and commercially software for acquiring images (Vision Builder for Automated Inspection, National Instruments). Every single pixel from the ML image acts as a small strain sensor. In order to convert the light emission to the actual strain values, MATLAB software is used to carry out the image processing.

In the first step of the image processing, all images are sorted by time. The gray level images are then read and converted to pixel values. To develop the predictive and calibration, an area on the ML film image is selected, and the average value of light intensities in the area is used for all calculations. The integrated area of the gray level value on the specimen divided by the region of interest yields an average value of the ML light intensity corresponding to the stresses acting on the ML film in case of uniaxial tension test. Since the frame rate is fixed at 10 fps, light intensity versus time can be drawn for each time step (0.1 s). This data is synchronized with the load step data to obtain the light intensity versus load values (i.e. force, stress, or strain).

4-4 **Sources of Errors in Experimental Testing of ML Sensors**

For each kind of sensors, there are possible sources of errors according to the nature of sensor and effects of environment on it. The ML stress sensors are almost new sensors and they have not been widely used in practice. Therefore, we have listed possible error sources for the sensors in the following.
4.4.1 Environmental errors

One of the influential parameters on the performance of the ML sensors is the effect of environmental light condition on the sensors. In all test matrixes, while capturing images, ambient light is completely blocked to minimize the environment contribution to the ML light intensity and we use the same darkness condition for all specimens to decrease this source of errors.

4.4.2 Manufacturing errors

Another source of errors in ML sensor test can be from imperfection of the manufacturing the samples. Since ML powder is mixed with an optical epoxy, any non-uniform distribution of the ML particles in the epoxy matrix can yield to the inconsistency of the ML light emission and the region which has more ML particles can emit more light. To avoid this error, the powder is dispersed in the epoxy using a magnetic stirrer for appropriate time duration.

In the disc-shaped specimens, some errors can be from the geometrical parameters. In the tests, not only should the specimens have the same diameter and thickness, but also the specimen surface along the thickness should be very smooth. If the surface is not smooth enough, it causes stress concentrations in some parts of the specimen and directly affects the light intensity measured by the cosine receptor. This error can be reduced by using the suitable mold and smooth and flat contact surface with loading plates. This issue is more important for the specimen side that faces to the receptor.

In the film tests, having the non-uniform film thickness can make different in the light intensity from the film and make errors. To produce a film with a uniform thickness, we use doctor blade method with a finely machined stainless steel blade. The thickness error of the film is around 0.5 mil.

4.4.3 Measuring device errors

The other source of errors in ML sensor test can be from the devices which measure the light intensity such as CCD cameras or spectrometers. For spectrometers fail in calibrating the wavelength of the spectrometer can produce errors in measuring the wavelength and the light intensity of the ML emissions. In an ideal CCD camera, every photon striking a pixel would be converted into exactly one electron. Then the number of electrons would be precisely counted and converted to a number telling the photographer
exactly how much light struck each pixel. Unfortunately, the process of converting light to pixel values in a CCD image is governed by some fundamental physical laws and other factors that introduce unwanted noise into an image. Some major sources of noise in CCD images can be listed as: dark current, pixel non-uniformity, shot noise, CCD read noise on chip, and electronic interference. To minimize the experimental errors in ML sensor tests, each test is repeated multiple times and in some cases average light intensity is used in calculation to decrease the errors as much as possible.
5-1 Overview

In this chapter the experimental test results of the ML specimens are presented. The results of ML composite disc-shaped compression tests and thin ML sensing film tension tests are presented separately.

5-2 Preliminary Experimental Study of Disc Specimens Under Compression Loading

In this section, the preliminary experimental study on disc-shaped composite ML samples under compression loading is presented. Before describing test results, we present some schematic test results and the way how we use to obtain the results. Figure 5-1 illustrates two phosphorescence spectra from SAOED, that is, under non-loading and loading conditions and measured by spectrometer. As it is shown in the figure, the spectra peak value is around 520 nm which is related to green light and in good agreement with the reported value [85, 104]. After applying 3 kN compressive loading at a load rate of 2 mm per min, the spectra peak value increase. Fitted polynomial curves are used to investigate the spectra peak changes through the test.
Figure 5-1: The original phosphorescence spectra of a ML sensor (SAOED)

Figure 5-2 shows a series of photos of a ML sensor (SAOE) under compression test during the loading history. Figure 5-2 is intended only to visually show ML phenomenon. Following presented results include detail information. Because the ML sensors we have used are the stress sensors, the light clearly shows the stress distribution in the sample which is very well matched with the expected distribution.

The repeatability is very important for long-term monitoring. In addition to general comparisons of the SAOE and SAOED sensors in the first test set, its results will verify the repeatability of the sensors because each specimen was tested six times without changing in the results.
5-2-1 Sensitivity characteristics and strain rate effects of SAOE and SAOED

For the first test matrix, tests were conducted for each sample to investigate the light intensity change by applying the loads. Figure 5-3 shows light intensity changes for both SAOE and SAOED in three different strain rates and different time intervals. As it can be seen in these figures, by increasing the force, the spectra peak values are increased initially and decreased eventually. Causes of this decrease...
are the decaying effect of afterglow and decreasing of the strain rate by the tester machine as the load approaches to the upper limit (3000 N). In the other words, there are two parameters which affect the light intensity; 1: compressive stress which increases the light intensity and 2: the exponentially decaying afterglow which decreases the light intensity. From these figures, it is also clear that the SAOED emits more light intensity in comparisons with SAOE.

Figure 5- 4 illustrates the maximum peak light intensity changes for the SAOE and SAOED in three different strain rates. As it can be seen in the figure, the change in the maximum light intensity for SAOED is significantly more than that for SAOE. From this figure, it is clear that the strain rate increases sensitivity (i.e. light intensity change per unit force) of SAOED much more than it does that of SAOE. The first test results generally indicate that SAOED material is better than SAOE material in terms of the sensitivity because SAOED emits higher light intensity and more sensitive to the strain rate than SAOE.
Figure 5-3: Changes of the phosphorescence spectra of SAOE and SAOED for three loading rates in different time intervals
For the second test matrix, five new specimens are made from the same SAOE and SAOED material batch as that used in test matrix I. The samples are tested under higher strain rates for comparison with the first test set. As we have proven the repeatability of the ML sensors in the first test set, we have used each specimen five times under five different strain rates.

Figure 5- 5 illustrates light intensity changes for SAOE and SAOED versus time for different wavelengths by following the second test matrix. According to Figure 5- 5, both materials show peaks at wavelength 520 nm. The more strain rate is, the more changes in light intensity are exhibited. The presented figures are well-matched with the results of the first test matrix and show that SAOE and SAOED have the same trend for the higher strain rates as the first test matrix.
Figure 5- 5: Changes of the phosphorescence spectra of SAOE and SAOED for five strain rates in different time intervals

5-2-2 Repeatability and linearity of the SAOE and SAOED specimens

Figure 5- 6 illustrates the peak light intensity changes for SAOE and SAOED versus time for wavelength 520 nm. It should be noted that as the tests are conducted under displacement control, we can replace time axis with displacement axis without any change in the graphs. From well-known strain equation
(Δ=PL/EAg), as geometrical and mechanical properties are same in each specimen, the displacement (or time) is linearly proportional to the force. From these figures, because the peak light intensity-time curves are close to each other, the repeadability of SAOE and SAOED specimens are concluded. As it is also expected, strain rate does not have effect on repeadability of SAOE and SAOED materials. Regarding the linearity between the light intensity and the force, SAOED also shows better linearity than SAOE.

Figure 5-6: Peak light intensity versus time curves for two specimens (one is SAOE and one is SAOED) under different strain rates.
5-2-3 Effect of loading rate on light intensity

Figure 5-7 illustrates the maximum peak light intensity changes for the SAOE and SAOED at five different strain rates (2, 4, 6, 8, and 10 mm/min). The loading rates range from a strain rate used for standard static tests to an intermediate strain rate. As shown in Table 5-1, mean values of the maximum light intensity changes of SAOED are greater than those of SAOE in the defined displacement range.

Table 5-1: Comparisons of mean values of max light intensity changes (scope) for SAOE and SAOED at different loading rates

<table>
<thead>
<tr>
<th></th>
<th>2 mm/min</th>
<th>4 mm/min</th>
<th>6 mm/min</th>
<th>8 mm/min</th>
<th>10 mm/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAOE</td>
<td>65</td>
<td>110</td>
<td>159</td>
<td>195</td>
<td>222</td>
</tr>
<tr>
<td>SAOED</td>
<td>94</td>
<td>153</td>
<td>169</td>
<td>197</td>
<td>352</td>
</tr>
</tbody>
</table>

As it can be seen from Figure 5-7, it is also clear that higher strain rate shows higher sensitivity for both SAOE and SAOED. From a rate of 10 mm/min, the sensitivity of SAOED was observed to drastically increase. However, in order to draw general conclusions about loading rate effects, more tests in an extended range of loading rates are needed.
Figure 5-7: Changes of maximum peak light intensity for all SAOE and SAOED specimens under five different strain rates
5-2-4 Saturation of the SAOE and SAOED materials

Figure 5-9 shows the relative light intensity at wavelength 520 nm versus force, averaged for all the specimens, in the five different strain rates. It should be noted that the loading profile of SAOE and SAEOD samples for each loading rate has a linear relation with displacement (or time), which is also shown in the results of the first test matrix. At first we can see that the increasement of light intensity for SAOED is more than that for SAOE. From the figures, the fitted curves are initially linear and then go to nonlinear part for both materials. However, linearity in the SAOED material is better than SAOE. Another point is that, although SAOE specimens are saturated during the loading history for different strain rates, the SAOED are not saturated for the loading profile defined here. This clearly shows that the light intensity of SAOE saturates sooner than that of SAOED which is another advantage of SAOED over SAOE in terms of stress sensing.

Considering the strain rate effects in Figure 5-8 and Figure 5-9, it can be seen that SAOE saturates faster than SAOED does in higher strain rates. It should be noticed that when the strain rate increases, the test duration decreases, therefore we capture fewer data points.
Figure 5-8: Relative peak light intensity and Force versus time for SAOE and SAOED for five strain rates - part 1
Figure 5-9: Relative peak light intensity and Force versus time for SAOE and SAOED for five strain rates - part 2

Table 5-2 shows initial slopes and correlation coefficients calculated until 300 N. Higher correlation coefficients more than 0.95 for both SAOE and SAOED prove ML light emission sensitive to mechanical stresses. However, from the initial slopes of the plots in Figure 5-9, SAOE showed higher initial sensitivity than SAOED. Therefore, in terms of active crack monitoring under dynamic loading, SAOE could be better than SAOED.
Table 5-2: Comparisons of initial slopes and correlation coefficient (force and relative light intensity until 300 N) for SAOE and SAOED

<table>
<thead>
<tr>
<th>Loading rate</th>
<th>Initial Slope</th>
<th>Correlation Coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>SAOE</td>
<td>SAOED</td>
</tr>
<tr>
<td>2 mm/min</td>
<td>0.163</td>
<td>0.12</td>
</tr>
<tr>
<td>4 mm/min</td>
<td>0.223</td>
<td>0.17</td>
</tr>
<tr>
<td>6 mm/min</td>
<td>0.2</td>
<td>0.08</td>
</tr>
<tr>
<td>8 mm/min</td>
<td>0.26</td>
<td>0.15</td>
</tr>
<tr>
<td>10 mm/min</td>
<td>0.305</td>
<td>0.17</td>
</tr>
</tbody>
</table>

5-3 Experimental Observations of Persistent Luminescence and ML Phenomena of a Thin Film

As reported in the reference [120], the ML phenomenon is complex due to many factors involved in transient changes of ML light emission. In the following, the effect of stress, strain rate, stress-free PL decay time interval, stress-state PL decay, and photoexcitation time on thin ML sensing film are considered and investigated.

5-3-1 Evidence of Persistent Luminescence Effects on ML Phenomenon

Figure 5-10 shows the ML light intensity versus time (i.e. stress) curves obtained for each combination of four stress-free PL decay time intervals (1, 2, 3, and 4 min) and four strain rates (0.1, 0.2, 0.3, and 0.4 mm/s). In the figure, the vertical axis values indicate the measured absolute ML light intensities (L.I.). In each experiment, the SAOED film sample is photoexcited for 2 minutes, decayed in dark conditions for different PL decay time intervals under stress-free states, and then loaded to the same maximum force (15 kN). As expected, a nonlinear relationship between the ML light intensity and stresses (i.e. time) is observed. As shown in Figure 5-10, the ML light intensity is found to increase with an increase in strain rates. These stress and strain rate effects for ML sensing materials are well known. The
experimental results also indicate that as the strain rate increases, the initial nonlinearity appears to be gradually diminished. However, the extent of the nonlinearity does not significantly change as the stress-free PL decay time interval increases. Noting that the differences (=max – min) between the max and min values of the y-axes are the same in all graphs, the relative changes of ML light intensity appeared to be dependent on PL decay time intervals at the same strain rate. Figure 5-10 provides evidence that the observed ML light intensity is comprised of both PL decay and ML light emission contributions simultaneously. It was demonstrated that by increasing the stress-free PL decay time intervals, the slope of the PL decay curves is reduced, and thus the PL decay effect is diminished. Although the PL data appear to be linear in the region plotted in Figure 5-10, the PL decay curves of these materials are known to decay exponentially.

Figure 5-10: Effects of stress-free PL decay time interval and strain rate on ML intensity (redrawn from [120])
In order to further investigate the PL decay effect on ML phenomenon, additional tests are conducted under extended PL decay time intervals (1, 2, 3, 5, and 7 min) and different strain rates (0.1, 0.2, 0.3 and 0.4 mm/s). For each combination, three tests were repeated to minimize experimental errors and to evaluate the statistical variance and mean value. Figure 5-11 depicts the effects of the strain rate and the stress-free PL decay time intervals on the relative ML light intensity \( I_r(t) = I(t)_{\text{peak}} - I_0(t_d) \), where \( I(t)_{\text{peak}} \) is the peak light intensity at the load 15 kN and \( I_0(t_d) \) is the light intensity at the onset of loading for each combination. The error bar indicates the standard error of the samples. At the highest strain rate (0.4 mm/s), the ML sensitivity tends to decrease as the stress-free PL decay time interval increases, whereas at the lowest strain rate (0.1 mm/s) the ML sensitivity tends to increase as the stress-free PL decay time interval increases.

![Figure 5-11: Change in relative ML light intensity for stress-free PL decay time intervals and strain rates](image)

Examination of \( I(t)_{\text{exp}} \) and the comparison of experimental test results plotted in Figure 5-11 indicate that the rate of increase of the ML light intensity due to the increase of strain rates is dependent on
the stress-free PL decay time intervals. This implies intrinsic mechanisms whereby the emission of photons by ML and PL decay mechanisms is occurring simultaneously.

5-3-2 Instantaneous PL Decay Rate under Stress States

In previous sections, contributions of stress-free natural PL decay effects on ML phenomena were considered. However, our experimental test results showed that PL decay rates under static stresses are significantly different from the stress-free PL decay rates. Because stresses instantaneously change while loading increases, it is named as instantaneous PL decay. Therefore, in order to investigate effects of stress, strain rates and stress-free PL decay time interval on the instantaneous PL decay rate, a series of tests were conducted at specific decay times (1, 2, 3, and 5 min) with a fixed strain rate of 0.3 mm/s. Loads were linearly applied up to different peak values (3, 6, 9, 12, and 15 kN) and maintained at the peak loads thereafter. Instantaneous PL decay trends are shown in Figure 5-12 a-d for decay times of 1, 2, 3, and 5 min, respectively, at a strain rate of 0.3 mm/s.
Figure 5-12: Instantaneous PL decay under different static stresses at strain rate 0.3 mm/s (a) 1 min decay time; (b) 2 min decay time; (c) 3 min decay time and (d) 5 min decay time where the dashed blue line indicates stress-free PL decay [121]

There are several important observations that can be made from the data. The PL decay rate following the rise to the peak intensity appears to be different from the stress-free PL decay curve corresponding to the same light intensity. Specifically, the PL decay becomes faster as stress increases. As decay times increase, the PL decay rate becomes slower. These observations are consistent with the view of increasing stresses and strain rates reducing the trap depth of oxygen vacancies and Dy³⁺ ions, resulting in an increase of carriers escaping to the conduction band that then flood into the Eu²⁺ luminescence centers.
and decay to the ground state through the emission of a photon. The presence of piezoelectric fields in SAOED results in faster decay rates than PL arising solely from thermal detrapping.

5.3.3 Effects of Photoexcitation Time on Stress-Free PL Decay

From the previous experimental observations related to the PL decay effects on the ML sensitivity, it is recognized that the proper characterization of the PL decay is an important factor for developing the predictive models. According to our experimental results, the stress-free PL decay depends on photoexcitation time (i.e. power). For example, while Figure 5-13(a) illustrates the stress-free PL decay pattern for four different photoexcitation times, Figure 5-13(b) shows the area under the decay curves (See solid circle mark) and the amount of light intensity drop (see open rectangle mark) until 300 seconds versus the photoexcitation time duration. Due to the finite number of photons, both the areas under the PL decay curves and the PL intensity drop appear to asymptotically converge to constant values. This finding is attributed to the finite number of photons and finite energy. In view of the PL model proposed by Aitasalo et al. [122], since the PL intensity must scale with the number of electrons decaying from the $4f^6\, 5d^1$ excited state of the Eu ion to the $4f^7$ ground state, the increase in the overall intensity of the PL decay curves with photoexcitation time is consistent with increasing the population of electrons in the excited state [120].

![Figure 5-13: Stress-free PL decay of SAOED depending on photoexcitation times][120]
5-3-4 ML sensitivity with different SAOED/resin ratio

In this section, the effect of different concentration ratio of the SAOED/resin is studied on the ML light emission performance. Four different ML thin films are prepared with mass concentration ratios of SAOED/resin 1:8, 1:5, 1:3, and 1:1. The thickness of all SAOED films is selected as 0.02 in. Standard dogbone shaped aluminum specimens are coated with the cut ML films by using a commercial adhesive. In every experiment, the SAOED film is photoexcited for 2 minutes for being fully excited. Figure 5-14 shows the stress-free natural PL decay curves of the ML thin film with different mass concentration ratios. As it is clearly seen in the figure, the light intensity of the films with lower mass ratio SAOED/resin drops quickly after the photo excitation power is removed. Also, the film with higher ratio has the longer saturation time. For instance, on the film with concentration ratio 1:1, if we apply the load within the first 60 s, there is no change in light intensity, because the light intensity is saturated and it is constant at 254. It can be also seen that as the concentration ratio of SAOED to resin increases, the rate of the decay curves drop becomes slower. This implies the significant effect of the concentration ratio of SAOED/resin on natural PL decay curves.

Figure 5-14: Stress-free natural PL decay curve of the ML films with different mass ratio resin/SAOED powder

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In order to investigate the effect of concentration ratio of SAOED/resin on the ML phenomenon, a series of tensile tests are conducted on standard dog-bone aluminum specimen under different PL decay time intervals (1, 3, and 5 min) and different displacement loading rates (0.1, 0.2, and 0.3 mm/s). The specimen is remained in linear-elastic range during the test and the maximum load is set at 15 kN. Figure 5-15 depicts the load versus ML relative light intensity with different mass concentration ratios, decay times, and displacement rates. According to the Figure 5-14, the ML light intensity of the film with 1:1 concentration ratio is saturated up to around 80’s second, therefore there is no change in light intensity for 1 min decay of the film with mass ratio 1:1. It should also be noted that if the tests start at a constant decay time with different ML film concentration ratio, each test starts at different initial light intensity. The light intensity from the film with higher concentration ratio starts at higher initial light intensity. According to the Figure 5-14, for 1 min decay time and 0.1 mm/s displacement rates, although we apply stress on the film, the ML light intensity decreases. This implies that the drop of the ML film light intensity due to the natural PL effect is more than increase of light intensity due to ML effect and also the light intensity of the ML thin film will decreases more if the load is not applied.

Another viewpoint of the effects of mass concentration ratio of SAOED/resin on the ability to use the ML phenomena of SAOED in a sensor is shown in Figure 5-16 where the max change of the ML light intensity, $I_{\text{peak}} - I(t'=0)$, where $I_{\text{peak}}$ is the peak light intensity at the load 15 kN and $I(t'=0)$ is the light intensity at the time of loading for each combination of decay times (1, 3, and 5 min) and displacement rates (0.1, 0.2, and 0.3 mm/s). Whereas the data in Figure 5-16 show that the higher mass ratio of SAOED/resin increase the max change of the ML light intensity and this trend is rapidly increase with the mass ratio 1:1.

Figure 5-17 demonstrates the effect of the mass concentration ratio SAOED/resin and decay times on the ML performance with different displacement rates. As expected, the max change of the ML light intensity increases by increasing the displacement rates. The figures also show that the max change of the ML light intensity for each concentration ratio increases until reaches a maximum ML light intensity at a specific decay time and then decreases. As a general trend, it can be concluded that the sensitivity of the ML light intensity increases with higher mass concentration ratio of SAOED/resin. It should also be noted
that the ML film becomes more brittle by increasing the ratio of SAOED/resin, so there is a limitation of the maximum mass concentration ratio of ML powder to Epoxy resin.

Figure 5-15: Effect of mass concentration ratio of SAOED/resin on the real time ML performance
Figure 5-16: Effect of the mass concentration ratio of SAOED/resin on the max change of the ML light intensity with different decay times and displacement rates.
Figure 5-17: Effect of the mass concentration ratio of SAOED/resin and decay time on the ML performance with different displacement rates.
5-3-5 ML sensitivity with different thicknesses

In this section, the performance of the ML light emission is studied with different film thicknesses. Four ML thin films are prepared with different thicknesses of 0.005, 0.01, 0.02, and 0.03 inch. The mass concentration ratio of SAOED to resin is 1 to 3 for all films. The films are coated on standard dog-bone shaped aluminum specimens. In every experiment, the film is photoexcited for 2 minutes before conducting the test. Figure 5-18 shows the stress-free natural PL decay curves of the ML thin film with a fixed mass ratio and different thicknesses. According to the figure, the duration of the film initial saturation time increases by increasing the ML film thickness. The film with the thickness of 0.03 inch does not show any change in PL light emission for more than two minutes. Also, the rate of the decrease of PL light intensity for thinner films is slightly higher than thicker films.

![Figure 5-18: Stress-free natural PL decay curve of the SAOED films with different thickness](image)

Figure 5-18 shows the real time and Figure 5-20 shows the max change of the ML light emission performance of thin film with different thicknesses, decay times, and displacement rates under tensile loading test. The test is limited in linear-elastic range of aluminum material and the max load is set to 15 kN. As a general trend, the ML light intensity change increases by increasing the thickness of the film which...
means higher thickness produces more change of ML light intensity. However, in some cases (0.1, 0.2, and 0.3 mm/s, 3 min decay time), the max change of the light intensity decreases by increasing the ML film thickness from 0.02 to 0.03 inch. Figure 5-21 demonstrates the force versus ML light intensity of two films with thickness of 0.2 and 0.03 inch. As it can be seen in figure, the initial light intensity of film with the thickness of 0.03 is around 243 whereas the ML light intensity of the film with the thickness of 0.02 inch starts at 146 (see Figure 5-18 for 3 min decay time). The maximum change of the light intensity which can be measured by the CCD camera for specific gain level and exposure time is 255. According to the Figure 5-21, the rate of the ML light intensity for thicker film is higher than thinner film in the beginning of the test, however, the light intensity from the film with the thickness of 0.03 inch starts being saturated after 6000 N force and as the results the max change of the ML light intensity of the film with thickness of 0.03 inch is lower than the film with thickness of 0.02 inch. As the general trend, it can be concluded that the film with higher thickness is more sensitive to the mechanical stress and produces more change of the light intensity. However, sufficient decay time period should be given to the film with higher thickness in order to avoid the saturation of the ML light intensity caused by the camera.
Figure 5-19: Effect of the ML film thickness on the real time ML performance
Figure 5-20: Effect of the ML film thickness on max change of the ML light emission with different decay times and displacement rates
Figure 5-21: Force versus light intensity of ML thin films with thicknesses 0.02 and 0.03 inch at 3 min decay time with 0.3 mm/s displacement rate.
6-1 Overview

In this chapter the total light intensity from ML materials is introduced. A predictive mechanoluminescence transduction model for the thin film of SAOED particles is proposed by using genetic programming technique. Sensitivity analysis on contribution of each term in predictive model is performed. A new relationship between stress and total light intensity is explained and a novel calibration method for ML strain sensing film described subsequently. The calibration model is verified and validated by the results from a finite element model.

6-2 Predictive Mechanoluminescence Transduction Model

On the basis of our experimental observations, it is hypothesized that the instantaneous increase in the ML emission and the instantaneous decay in the PL emission can be modeled separately when they are subjected to transient changes of stress and strain rates. Therefore, they will be combined into the total ML+PL light intensity. Moreover, the total ML+PL light intensity will be proportional to the applied mechanical strain energies.

Combining the PL decaying phenomena with the ML light emission effects, the mechanical energy applied to the ML sensing film is transduced to an incremental change of the total ML+PL light emission ($\Delta I$), which can be divided into three components: 1) net ML emission ($\Delta I_{ml}$), 2) stress-free PL decay ($\Delta I_{pl}$), and 3) additional stress-induced PL decay ($\Delta I_{ml}'$). With the exception of the stress-free PL decay, which is only dependent on instantaneous light intensity ($I(t)$, See Figure 6-1), the light intensity changes are correlated to the stress level ($\sigma$) (i.e. time ($t$) in case of fixed strain rate), the strain rate ($\dot{\varepsilon}$) and
the initial light intensity \((I_0)\). The initial light intensity \((I_0)\) can be uniquely determined as a function of the decay time interval \((t_d)\) as long as the film is photoexcited consistently with a standard light source and exposure time. Figure 6-1 depicts the schematic contributions of the ML emission, the stress-free PL, and the stress-state PL light decay effects.

As demonstrated in Figure 6-1, the total ML+PL light intensity can be expressed as:

\[
I_t(I_0, \sigma, \dot{\varepsilon})_n = I_0 + \sum_{i=1}^{n} [\Delta I_{ml}(I_0, \sigma, \dot{\varepsilon})]_i + \sum_{i=1}^{n} [\Delta I_{pl}(I(t))]_i + \sum_{i=1}^{n} [\Delta I'_{ml}(I_0, \sigma, \dot{\varepsilon})]_i
\]

Eq. 6-1

where \(I_0\) is the initial light intensity at the onset of stressing; \(\sigma\) is the stress; \(\dot{\varepsilon}\) is the strain rate; \(n\) is the total number of incremental time steps, and \(I_t\) is the total ML+PL light intensity. Therefore, the net ML light intensity...
intensity can be obtained by incrementally subtracting stress-free PL and additional stress-induced PL
decays from the total light intensity. Thus it is expressed as follows.

\[ I_{ml}(I_0, \sigma, \dot{\varepsilon})_n = I_0 + \sum_{i=1}^{n} [\Delta I_{ml}(I_0, \sigma, \dot{\varepsilon})]_i \]

Eq. 6-2

Eq. 6-2 indicates that the ML light intensity is a function of the stress, strain rate and initial light intensity.

By substituting \( \Delta I \) with \( \dot{I} \Delta t \), in which \( \dot{I} \) is the first derivative of light intensity with respect to time and \( \Delta t \) is a time step, Eq. 6-2 can be recast into the following format.

\[ I_{i}(I_0, \sigma, \dot{\varepsilon})_n = I_0 + \sum_{i=1}^{n} (\Delta I_{pl})_i + \sum_{i=1}^{n} (\dot{I}_{pl})_i \Delta t + \sum_{i=1}^{n} (\dot{I}_{ml})_i \Delta t \]

Eq. 6-3

where \( \tau = t - t_d \) is the timeframe relative to the onset of loading and \( t_d \) is the decay time interval or elapsed time of the loading onset. The integrands on the right hand side are all derivatives of the light intensity with respect to time. In addition to the proposed modeling framework, three different component predictive models are developed in the following subsections.

6-2-1 Predictive Model for Stress-free PL Decay

In this section, predictive models for \( I_{pl} \) and \( \dot{I}_{pl} \) in Eq. 6-2 and Eq. 6-3 are developed. It has been shown that the ML light emission of SAOED is strongly related to the PL decay characteristics. Therefore, the long-lasting PL decay needs to be characterized. A new recent genetic programming technique named multi-gene genetic programming (MGGP) [123] is used to develop the model equation. More information about the MGGP method is found in Appendix A. In the past, researchers studied the physical long-lasting PL decay phenomena [105], and several models for predicting the afterglow of different phosphors were found to be available [72, 106-108]. Notwithstanding the availability of existing models, a new predictive model is developed herein. The new model equation is expressed as a function of time as follows.
\[ I_{pl}(t) = B_0 - B_1 \exp \left( -\frac{C_1}{t} \right) - B_2 \exp \left( -\frac{C_2}{t} \right) \]  

Eq. 6-4

where \( I_{pl}(t) \) presents the stress-free PL decay; \( B_0, B_1, \) and \( B_2 \) are constants; \( t \) is time; and \( C_1 \) and \( C_2 \) are the decay time constants. For generating the experimental data, an SAOED film with a thickness of 0.02 inches was photoexcited for two minutes, and the PL afterglow was measured by the method described in chapter 5. Based on the experimental data shown in Figure 6-2, these constants are calibrated as \( B_0 = 752.2, B_1 = 241.6, B_2 = 34.88, C_1 = 10.04 \) and \( C_2 = 58.06 \). Figure 6-2 shows a comparison between the experiment and the predicted results by the calibrated model. Clearly, the predictive model showed a highly accurate match with the experimental PL decay curve. The model calibration is dependent on the relative differences in densities of shallow and deep traps. The higher value of the two decay time constants (\( C_1 \) and \( C_2 \)) is attributed to the higher density of deep traps and results in longer decay times, whereas the lower value is attributed to the higher density of shallow traps and results in shorter decay times.

The rate equation for the \( I_{pl}(t) \) in Eq. 6-3 is also developed in order to predict the instantaneous changes of the stress-free PL decay. This model equation must be a function of the current light intensity.
(\(I(t)\)) since the stress-free PL decay only depends on the instantaneous light intensity. By the MGGP technique, the rate equation, which is the slope of the PL decay curve at \(I(t)\), is expressed as

\[
\dot{I}_{pl}(I(t)) = \frac{I(t)^2}{D_1 I(t)^3 - D_2}
\]

where \(D_1\) and \(D_2\) are calibrated to \(D_1 = 0.00029\) and \(D_2 = 12388\) based on our experimental data. The rate equation will be used in Eq. 6-3 to predict the change of stress-free PL decay (\(\Delta I_{pl}\)).

6-2-2 Predictive Model for Net Mechanoluminescence Emission

In order to create predictive models that can simulate changes of \(I_{ml}\) and \(\dot{I}_{ml}\), a comprehensive experimental test database was prepared under various conditions by varying the strain rates (0.1, 0.2, 0.3, and 0.4 mm/s) and the decay time intervals (1, 2, 3, 5, and 7 min). The effects of the initial light intensity (\(I_0\)), strain rates (\(\dot{\varepsilon}\)), and stress level (\(\sigma\)) are incorporated into the predictive model. Eighty percent of all test data are used for the learning process in MGGP, and the remaining twenty percent are used to test the derived model for validation purposes. The validation test data are labeled as “Tested Data” in Figure 6-4 and Figure 6-8. The testing data set is not used in deriving the model; it is only used for validation. Eq. 6-6 shows the proposed predictive model equation for the net ML emission in terms of initial light intensity (\(I_0\)), stress (\(\sigma\)) and strain rate (\(\dot{\varepsilon}\)).

\[
I_{ml}(I_0, \sigma, \dot{\varepsilon}) = I_0 + A_1 \dot{\varepsilon} + A_2 I_0 \sigma + A_3 I_0^2 \dot{\varepsilon} + A_4 I_0 \dot{\varepsilon} \sigma + A_5 \dot{\varepsilon} \sigma^2
\]

Eq. 6-6

Based on the experimental data, the model constants are calibrated as \(A_1 = 0.7434\), \(A_2 = 2.043 \times 10^{-10}\), \(A_3 = 7.948 \times 10^{-8}\), \(A_4 = 1.033 \times 10^{-11}\), and \(A_5 = 3.358 \times 10^{-16}\). By subtracting the initial light intensity from the above equation, the relative net ML emission can be obtained as

\[
I_{R,ml}(I_0, \sigma, \dot{\varepsilon}) = A_1 \dot{\varepsilon} + A_2 I_0 \sigma + A_3 I_0^2 \dot{\varepsilon} + A_4 I_0 \dot{\varepsilon} \sigma + A_5 \dot{\varepsilon} \sigma^2
\]

Eq. 6-7

A sensitivity analysis is conducted to study the contribution of each term in Eq. 6-7. Figure 6-3 plots their contributions to net ML intensity under four different test conditions. As shown in Figure 6-3, the second (\(A_2 I_0 \sigma\)) and fifth (\(A_5 \dot{\varepsilon} \sigma^2\)) terms have primary contributions to the increases of the net
relative ML emission. As the decay time interval increases, the contribution of $A_2 I_0 \sigma$ notably decreases due to the fact that the initial light intensity exponentially decays as time increases. The term $A_2 I_0 \sigma$ is indicative of stress-free PL decay effect on net ML light intensity, whereas $A_2 \dot{\sigma} \sigma^2$ represents the effects of the stress and strain rate.

Figure 6-3: Sensitivity analysis on the contribution of each part to the net ML intensity in Eq. 6-7
The first \( (A_i \dot{e}) \) and third terms \( (A_i I_{i0} \dot{e}) \) in Eq. 6-7 are not significant contributors to the net ML light intensity. However, they include strain rates and initial light intensity, which are effects of the PL decay time intervals. Figure 6-4 compares the experimental measurements of the ML light intensity with the predictions by Eq. 6-6 as stresses increase at different strain rates and PL decay time intervals. The proposed model for net ML light intensity was found to predict the experimental results accurately. The experimental data with the “Tested Data” label were not used in deriving the predictive model by MGGP training. Even in the validation cases, the proposed model could accurately predict test data, partly showing its generality. This implies that Eq. 6-6 can properly predict the net ML light intensity for different strain rates and stress levels at different PL decay time intervals.

![Figure 6-4: Comparisons of experimental ML light intensity and predictions by the proposed model ("Tested Data" indicates validation results of the proposed model)](image)

To our surprise, the proposed model turned out to be coincident with an existing model. For example, Chandra and Chandra [14] derived an expression for the rise in ML intensity \( I(t')_{ML} \) of materials
like SAOED in their elastic region as a function of applied stress ($\sigma$) and strain rate ($\dot{\varepsilon}$), where $t'$ is the timeframe measured relative to the onset of loading.

$$I(t')_{ML} = C_1 \dot{\sigma}(\sigma - C_2 \sigma^2) = C_1 E \dot{\varepsilon}(\sigma - C_2 \sigma^2)$$  \hspace{1cm} \text{Eq. 6-8}

where $C_1$ and $C_2$ are constants related to materials parameters and $E$ is the Young's modulus. It is worth noting that the last two terms in Eq. 6-6 match well with Eq. 6-7 for a fixed initial ML light intensity. However, the initial light intensity (i.e. PL decay effect) is not considered in Chandra and Chandra’s model, whereas it is included in the present model. Thus, the present model has the advantage of leading to better prediction of the ML light intensity regardless of the PL decay time intervals. Figure 6-4 presents evidence of the aforementioned advantage. However, the initial light intensity must be available in order to use the proposed predictive model.

### 6-2-3 Predictive Model for Additional Stress-Induced PL Decay Rate

In previous sections, stress-free PL decay and net ML emission were modeled separately. In addition, our experimental test results showed that PL decay rates under static stresses are significantly different from the stress-free PL decay rates. This motivates the development of the third part of the predictive model, which can predict the stress-state PL decay rate ($\Delta I'_{ML}$, see Figure 6-1). Since stress causes an additional decay effect on ML, we referred to it as additional stress-induced PL decay. Therefore, in order to investigate effects of the stress, strain rates, and stress-free PL decay time interval on the additional stress-induced PL decay rate, a series of tests were conducted at specific decay time intervals (1, 2, 3, and 5 min), and strain rates (0.1, 0.2 and 0.3 mm/s). Loads were linearly applied up to different peak values (3, 6, 9, 12, and 15 kN) and were maintained at the peak loads thereafter. Instantaneous PL decay trends are re-shown in Figure 6-5 for decay time intervals of 1, 2, 3, and 5 min, respectively, only at a strain rate of 0.3 mm/s.
Figure 6-5: Stress-state PL decay curves under different static stresses at strain rate 0.3 mm/s (a) 1 min decay time (b) 2 min decay time (c) 3 min decay time and (d) 5 min decay time. The dashed blue line indicates stress-free PL decay.

Differences between the stress-free PL decay rate in Eq. 6-5 and the initial rate of the instantaneous stress-state PL decay trends at peaks load values (3, 6, 9, 12, and 15 kN) will be predicted by
the proposed model as a function of stress, strain rates, and initial light intensity. Figure 6-6 shows $\dot{i}_{ml}'$, which is clearly defined as the difference between stress-state PL decay rate ($=(I_{t+\Delta t}-I_t)/\Delta t$) and stress-free PL decay rate ($\dot{I}_{pl}$, Eq. 6-5) with a time step size ($\Delta t$) of 0.2 sec under different test conditions. The definition is expressed as

$$\dot{i}_{ml}' = \dot{I} - \dot{I}_{pl}$$

Eq. 6-9

where $\dot{I}$ is the stress-state PL decay rate and $\dot{I}_{pl}$ is defined in Eq. 6-5. The positive y-axis values in Figure 6-6 imply that additional stress-induced PL decay is always faster than stress-free PL decay. This occurs because the static stress states will reduce the trap-depth of activator ions by a piezoelectric effect.

For the same reason, as stresses increase, $\dot{i}_{ml}'$ increases. As the PL decay time interval decreases, $\dot{i}_{ml}'$ also increases due to the increased concentration of excited carriers.
According to the experimental observations, a predictive model for $I_{ml}^{'}$ as a function of the initial light intensity, stress, and strain rate are proposed as follows

$$I_{ml}^{'}(I_0, \sigma, \dot{\varepsilon}) = K_1 \dot{\varepsilon} \sigma^2 (1 - \dot{\varepsilon}) + K_2 \sigma + K_3 I_0 \dot{\varepsilon} \sigma.$$ \hspace{1cm} Eq. 6-10

where calibrated coefficients are $K_1=3.149 \times 10^{-16}$, $K_2=-4.845 \times 10^{-9}$ and $K_3=1.449 \times 10^{-9}$ in our model. A sensitivity analysis is also performed to determine the contribution of each term in Eq. 6-10. Figure 6-7 shows the contribution under four different test conditions. According to Figure 6-7 and Eq. 6-10, the third term plays a primary role in the prediction model in lower decay time intervals whereas the contribution of the third term increases by increasing the decay time intervals. The second term is dependent only on stress levels.
Figure 6-7: Sensitivity analysis on the contribution of each term of the additional stress-induced PL decay rate model

Figure 6-8 illustrates the actual and predicted values of the $\dot{I}_{ml}'$ for different strain rates (0.1, 0.2 and 0.3 mm/s) and at different PL decay time intervals (1, 2, 3 and 5 min). According to Figure 6-8, not only can the present model predict the experimental data, but it is validated with the “Tested Data” with a high degree of accuracy.
Insights on Stress-Total Light Intensity Relationship

As previously hypothesized, by inducing stresses to ML sensing film, mechanical energy is transduced to an incremental change of the total ML+PL light intensity. This total ML+PL light emission is combined as a sum of the net ML emission, stress-free PL decay, and additional stress-induced PL decay. By using Eq. 6-3, Eq. 6-5, Eq. 6-7, and Eq. 6-10, stress-free and stress-state PL decay can be calculated and added to the net ML light intensity. Therefore, the total ML+PL light intensity can be expressed in terms of stress ($\sigma$), initial light intensity ($I_0$), and strain rates ($\dot{\varepsilon}$). Figure 6-9 depicts the relative ML light intensity from the experiment and the relative total ML+PL light intensity from the proposed prediction models for two different test conditions. As expected, the relationship between $I_{ml}$ and stress was nonlinear. The difference between the total ML+PL light intensity and the net ML light intensity represents the effects of PL decay.
Figure 6-9: Comparison between the relative $I_{ml}$ from the experiment and the relative total ML+PL light intensity (LI) from the prediction models.

Figure 6-10 (a) shows the relative total ML+PL light intensity versus stress for two test conditions (0.2 mm/s - 5 min decay time and 0.3 mm/s - 3 min decay time). The square marks represent the results from the model, and the cross marks represent the results from the experimental tests. The results show a good agreement between the model and the experimental results. Figure 6-10 (b) demonstrates the relative total ML+PL light intensity versus the square of stress. It appears that the relative total ML+PL light intensity clearly has a linear relationship with the square of stress. Since the deformation energy has a quadratic relationship with stress, it can be concluded that the total ML+PL light intensity also has a linear relationship with the mechanical deformation energy.

$$E_{\text{deformation}} = \frac{1}{2} \frac{\sigma^2}{E} \propto I_t$$

Eq. 6-11
6-4 calibration model for the full-field ML sensing films

There are several full-field strain measurement methods including Digital Image Correlation (DIC) [1], photoelastic coatings [2], and Moire and interferometric methods [3]. Among these, digital image correlation techniques have been most widely used in various applications for its non-contact sensing ability and relative ease of implementation and use. As an alternative, mechanoluminescent thin sensing films can be used as new non-contact full-field strain sensors. In this section, a new quantitative stress measurements calibration model is proposed by using SAOED ML thin film subjected to in-plane stresses.

6-4-1 data preparation and model development

There are many parameters involved in transient changes of the ML emission of a thin ML sensing film. These parameters can be classified into two categories; 1) manufacturing parameters (i.e. materials concentration ratio, ML film thickness, solid epoxy matrix, etc.) and 2) experimental parameters (i.e.
effects of persistent luminescence (PL), effect of stress rate, and effect of photoexcitation). In order to provide a calibration model, the manufacturing parameters are limited by selecting a fixed mass concentration ratio of epoxy to ML powder (1:1) and fixed ML film thickness (0.02 inch). The choice of these parameters is based on the sensitivity analysis of the ML sensing film with different thicknesses and mass concentration ratios. The ML sensing film is consistently photo-excited by a 40 watt broadband light source (having wavelength range of 400-1000 nm) for 2 min before conducting the tension test to assure it was fully saturated with the same photoexcitation powder. After full excitation, stress-free PL light is emitted which displays a naturally decaying intensity with respect to time. To avoid the effect of stress-free persisted luminescence (PL), all the specimens are aged in the dark place for exactly 7 min before the onset of the loading, so the initial light intensity of the films will be constant.

In order to create ML calibration model that can predict the fill-field strain values, a comprehensive experimental tension test are conducted on a standard aluminum dog-bone specimen. The database is prepared under various conditions by varying the strain rates (0.1, 0.2, 0.3, and 0.4 mm/s) at the decay time of 7 min. & min is long enough not to reach the maximum light intensity during the loading. The ML thin film is attached on a standard aluminum dog-bone shape specimen following ASTM B557-10. Loads are linearly applied up to 15 kN and each test is repeated three times to minimize the experimental error. During the loading period, images are captured by a high speed CCD camera at a frame rate of 10 fps. After carrying out the image processing, an average value of ML film light intensity is calculated for each frame fps specific region on the film. Having a fixed frame rate at 10 fps, light intensity versus time can be drawn for each time step (0.1 s).

The Incremental values of step time ($\Delta t$), change of the ML light intensity ($\Delta LI$), ($\Delta \eta$), and effective strain from previous step are incorporated into the MGGP model to predict a calibration equation of the change of effective strain ($\varepsilon_e$) in each time step. The predictive calibration model can be shown as follows.

$$
\Delta \varepsilon_{e(n)} = f(\Delta t_{(n)}, \Delta LI_{(n)}, \Delta \eta_{(n)}, \varepsilon_{e(n-1)})
$$

Eq. 6-12
Which $\Delta e_{e(n)}$ is the change of the effective strain at step $n$, $\Delta t_{(n)}$ is the time increment at step $n$, $\Delta LI_{(n)}$ is the change of the ML light intensity at step $n$, $\Delta \eta_{n} = e_{e(n-1)} \Delta LI_{(n)}$, and $e_{e(n-1)}$ is the effective strain at step $(n-1)$. The effective strain is a scalar value and is defined as follow [124].

$$e_{e} = \frac{2}{3} \left[ \frac{1}{2} \left( e_{11} - e_{22} \right)^2 + \left( e_{22} - e_{33} \right)^2 + \left( e_{33} - e_{11} \right)^2 + \frac{3}{2} \left( \gamma_{12}^2 + \gamma_{23}^2 + \gamma_{31}^2 \right) \right]^{\frac{1}{2}}$$  
Eq. 6-13

which $e_{e}$ is the effective strain, $e_{11}$, $e_{22}$, and $e_{33}$ are the normal strain components, and $\gamma_{12}$, $\gamma_{23}$, and $\gamma_{31}$ are the shear strain components. $\Delta \eta$ is used in the calibration model to improve the accuracy of the model [125]. For each loading case, the incorporated input data are prepared corresponding to five different step time increments, $\Delta t=0.1$, 0.2, 0.3, 0.4, and 0.5 s.

The final empirical equation of the change of effective strain at step $n$, by using the genetic programming, can be expressed as follow.

$$\Delta e_{e(n)} = C_1 \left( \Delta t_{(n)} + \log|\Delta LI_{(n)}| + e_{e(n-1)} \right) - \log|e_{e(n-1)}| 
+ C_2 \left( \Delta LI_{(n)} + \log|\Delta t_{n}| \right) + C_3 \left( e_{e(n-1)} - \Delta \eta_{n} - \log|\Delta \eta_{n}| \right) + C_4$$  
Eq. 6-14

which $C_1$, $C_2$, $C_3$, and $C_4$ are the model constants and calibrated as $C_1=0.1$, $C_2=0.859$, $C_3=0.01408$, and $C_4=0.1719$. The effective strain in each step time can be obtained by accumulation of the changes of the effective strain values of the previous step times. It should be noted that the above calibration equation is based on the SAOED film, with the thickness of 0.02 inch, concentration ratio of SAOED/Epoxy 1:1, and 7 min decay time. If any of these parameters changes, the model constants need to be re-calibrated again.

6-4-2 Verification of the proposed calibration model

In order to verify the proposed calibration model, all the experimental and calibrated effective strains are presented in Figure 6-11 in terms of step time increments. The “Test No.” label represents the number of inputs incorporated into the genetic programming for each test. According to the Figure 6-11, the calibrated data are in a good agreement with experimental data.
Figure 6-11: Calibrated effective strain results versus experimental strain results for different step time increments and loading rates

Figure 6-12 shows the experimental and calibrated effective strain versus relative ML light intensity for different loading rates. The results are presented in five different step time increments for each test. These figures demonstrate the calibration model accuracy. They also show that the model accuracy is not very sensitive to different step time increments.
Figure 6-12: Experimental and calibrated effective strain versus relative ML light intensity for Δt=0.1, 0.2, 0.3, 0.4, and 0.5 s under four different loading rates.

Figure 6-12 visually demonstrates the comparison of all experimental effective strain values and calibrated data, for each input set and for all different tests, in a single figure. The correlation between the calibrated and experimental data is calculated as 0.996. It implies that the calibration model effectively used for predicting the effective strain values.
6-5 Validation of proposed calibration method

In the previous section, the calibrated results were verified by the experimental data. In this section, the performance of the proposed calibration model is validated as a full-field strain measurement method. In order to do so, thin ML sensing film is applied on three different aluminum specimens including: open-hole dog-bone specimen, pure shear specimen, and I-beam specimen. The effective strains from the ML sensor are calculated by the proposed calibration method and validated with FE simulation results. The thickness and concentration ratio of the ML film is exactly similar to the film which was used to develop the calibration model and equal to 30 mil and 1:1. The film is excited for exactly 2 min and aged in the dark for 7 min. The camera setting is similar to the previous experiments and the frame rate is 10 fps.

6-5-1 Tensile test on aluminum dog-bone specimen with an open-hole

A tensile test is conducted on a dog-bone aluminum specimen with an open-hole coated with ML thin film (see Figure 6-14). The tensile load is applied linearly up to 45 kN with the loading rate=0.4 mm/s and the aluminum is remained in the linear-elastic range. During the loading test, images are captured by
the high speed CCD camera at frame rate 10 fps. Load-displacement curve of the aluminum dog-bone specimen with an open-hole is plotted in Figure 6-15.

![Figure 6-14: Aluminum dog-bone aluminum specimen with an open-hole coated with ML thin film](image)

![Figure 6-15: Load-displacement curve of aluminum dog-bone specimen with an open-hole](image)

Figure 6-16 shows the black and white raw images and the distribution of the change of the light intensity from the ML sensor at different loading levels. The time increment of the ML images is equal to 0.4 s. As it can be clearly seen in the figure, more light intensity can be observed around the hole which shows higher stress concentration.

The time increments and the change of the light intensity for each step time are plugged into the Eq. 6-14 to calculate the effective strain for each single pixel. The effective strain for each pixel in each
step time can be obtained by accumulation of the changes of the effective strain values of the previous step times. Figure 6-17 demonstrates the distribution of the full-field effective strain values at three different loading levels (13, 26, and 39 kN) calculated form the calibration model. Also, the effective strain values calculated from the FE simulation (ABQUS 6.13-2) results are presented in Figure 6-17. According to the figure, the present calibration model predicted the effective strain accurately and the results are validated with acceptable accuracy.

As it can be seen in Figure 6-17, the distribution of the effective strain from ML sensor is not perfectly symmetric and the area below the hole shows higher strain values. This is due to the imperfection in specimen set-up in-between the instron machine’s grips. According to the figure, it seems that the specimen was not perfectly placed vertically between the grips and that causes producing more shear strain on the specimen and yields to non-symmetric distribution of the effective strain values on the specimen. The real-life distribution of effective strain values is a considerable advantage of the ML sensor over the finite element analysis since the uncertainty in boundary conditions, material properties, and manufacturing imperfection are the main concerns in finite element simulations.
Figure 6-16: Raw images of ML sensor coated on aluminum dog-bone specimen with open-hole and distributed change of the ML light intensity at different loading levels
Figure 6-17: Comparison of the ML effective strain (left images) and FE effective strain values (right images) at different loading levels.
The distributed effective strain results from the ML calibration model and FE simulation in the vicinity of the open-hole with error percentage distribution at different loading levels is presented in Figure 6-18. The error percentage of the calibration model is obtained by simply subtracting the pixel-by-pixel ML effective strains from FE effective strains divided by FE effective strains. The error equation can be shown as follow.

\[
err = 100 \times \left( \frac{\varepsilon_{e,ML} - \varepsilon_{e,FE}}{\varepsilon_{e,FE}} \right)
\]

Eq. 6-15

which \(err\) is the error percentage, \(\varepsilon_{e,ML}\) is the effective strain from ML sensor, and \(\varepsilon_{e,FE}\) is the effective strain from FE simulation. The effective strain values from FE simulation is interpolated for each pixel following the pixel coordinate of the ML results. According to the error percentage figures, the error of the ML sensor decreases by increasing the loading levels. However, it is worth noting that FE results are assumed to be true reference values.

Figure 6-19 demonstrates the pixel-by-pixel comparison of the FE effective strain and ML effective strain values at load level 39 kN for the selected region of interest. The vertical axes on the right side of the figures can be used to find the corresponding X and Y coordinates to the effective strain values to locate the corresponding pixel on the images. The figure implies that the ML effective strain values are in a good agreement with those from FE simulation.
Figure 6-18: Effective strain results of the ML calibration model and FE simulation in the vicinity of the open-hole with error percentage distribution at different loading levels.
Figure 6-19: Pixel by pixel comparison of the effective strain from FE and effective strain from ML sensor at load 39 kN
6-5-2 Pure shear test

In order to do more validation tests of the ML sensor, a standard pure shear test is performed on aluminum alloy, following the ASTM B31-05 (see Figure 3-17). Slotted single shear test fixture is used to mount the specimen and an instron testing machine is used to conduct the test. Load is applied linearly up to 600 N with the loading rate of 0.3 mm/s. Figure 6-20 shows the load-displacement curve for the aluminum pure shear specimen under the tension loading. Images of the ML sensor are captured at 10 fps with a high speed CCD camera and the calibration model is used to calculate the distributed effective strain at three different loading levels (200, 400, and 600 N). Also, a FE model of the specimen is developed by using commercial software (ABQUS 6.13-2) to numerically calculate the effective strain values. Figure 6-21 shows the black and white raw images with distributed effective strains from FE simulation and ML sensor. The step time between the raw images is equal to 0.3s. As expected, the high stress concentration accrues around the notch and the effective strains increase by increasing the loading level. The lower and higher bound of the color bar is fixed in the figure in order to have a better comparison of the FE simulation and ML sensor results.

Figure 6-20: Load-displacement of the aluminum pure shear specimen
Figure 6-22 displays the distribution of the effective strain results from the ML calibration model and FE simulation of pure shear test in the region of interest around the notch. The error percentage of the ML sensor at different loading levels is also depicted in Figure 6-22 for the cropped area. Eq. 6-15 is used to calculate the error percentage for each pixel at each step. According to the figure, the calibration model can acceptably predict the effective strain for the region with the highest stress concentration; however, there might be significant errors for the area with lower stress concentration. These high errors can be caused by miss-matching of the tracked pixel.

Figure 6-23 shows the effective strain values of the cropped area from the ML sensor versus FE simulation at different loading levels. As it can be seen in the figure, the results shows better correlation by increasing the loading levels from 200 N to 600 N. Figure 6-24 demonstrates the pixel by pixel comparison of the effective strain from FE and effective strain from ML sensor of a pure shear test in the region of interest and at different loading levels. The error percentage of each pixel is also shown on the right axis. It can be clearly seen that as the applied load increases the overall error percentages decrease.
Figure 6-21: Effective strain distribution from FE simulation and ML sensor on the aluminum pure shear specimen at different loading levels.
Figure 6-22: Distribution of the effective strain results from the ML calibration model and FE simulation of pure shear test in the region of interest with error percentage at different loading levels.
Figure 6-23: ML versus FE effective strain of a pure shear specimen in the region of interest at different loading levels.
Figure 6-24: Pixel by pixel comparison of the effective strain from FE and effective strain from ML sensor of a pure shear test in the region of interest and at different loading levels.
The applied load on the aluminum pure shear specimen versus average values of the effective strain for the region of interest from the ML sensor and FE simulation is displayed in Figure 6-25. The figure shows that the average value of the effective strain from ML sensor and FE simulation increase linearly by increasing the load level and the difference between them decrease at higher stress levels.

![Figure 6-25: The average effective strain from ML sensor and FE simulation results in the region of interest versus applied load](image)
6-5-3 Four-point bending test

An I-beam aluminum specimen coated with a thin ML sensing film is prepared for a four-point bending test to see the performance of the ML sensor under the stress due to the bending moments and validate the proposed calibration model. A 4 in width and 3 in height ML sensing film is exactly attached to the middle of the beam’s web. FE software (ABQUS 6.13-2) is used to develop the I-beam model and numerically calculate the effective strain values on the beam’s web. Strain gauges are also installed on the top of the upper flange and on the bottom of the lower flanges in order to verify the FE simulation results. Figure 6-26 displays the experimental test set-up. Load is applied linearly up to 180 kN and the aluminum is remained in linear-elastic range with this maximum load (see Figure 6-27). Images of the ML film are captured at speed 10 fps by using CCD camera.

Figure 6-28 displays the dimensions of the aluminum I-beam specimen which have been used in FE model. Figure 6-29 shows the von-Mises stress distribution of the aluminum I-beam specimen from the FE simulation results for the four-point bending test at load 180 kN. The FE stress distribution of the web’s beam for the area coated with ML sensor is also shown in Figure 6-29. According to the FE results, the neutral axis of the beam is slightly moved to the bottom of the beam and the stress levels at the top of the ML film area are higher than the bottom.
Figure 6-26: Test setup of the four-point bending test on aluminum I-beam specimen coated with ML sensor
Figure 6-27: Load-displacement curve of the aluminum I-beam specimen under four-point bending test

Figure 6-28: Dimensions of the aluminum I-beam for four-point bending test (dimensions are in inches)

To verify the FE results, the longitudinal strains at the top of the upper flange and at the bottom of lower flange are measured by the strain gauge at load 180 kN and compared with those from FE simulation. The results are shown in Table 6-1. The results demonstrate that the longitudinal strain values calculated by FE model match very well with measured values. Therefore, FE results are verified.
Table 6-1: Comparison of the longitudinal strain from FE simulation and strain gauges for the aluminum I-beam specimen under four-point bending test

<table>
<thead>
<tr>
<th></th>
<th>FE simulation</th>
<th>Strain gauge</th>
</tr>
</thead>
<tbody>
<tr>
<td>longitudinal strain at the top of the upper flange</td>
<td>0.002421</td>
<td>0.002365</td>
</tr>
<tr>
<td>longitudinal strain at the bottom of the lower flange</td>
<td>0.002897</td>
<td>0.002869</td>
</tr>
</tbody>
</table>

Figure 6-30 displays the black and white raw images, light intensity images from the raw images and change of the light intensity distribution images from the ML film coated on aluminum I-beam specimen at four different loading levels 63, 90, 126, 153 kN. The step time between the images is 0.4 s. It is obvious from the figures that the change of the light intensity increases by increasing the applied load. As expected from the FE simulation results, the change of the light intensity from the top of the film is higher than the bottom. The distributed effective strain from the FE simulation and ML sensor using the calibration model are presented in Figure 6-31. The results are shown at different loading levels. Also, the error percentages of the results are shown in Figure 6-31 at different loading levels. According to the results, it can be seen that the errors reduce by increasing the loading levels. It can also be concluded that the calibration model can predict the effective strain at higher strain levels with less error. To increase the accuracy of the calibration model by using ML sensor, a film with higher sensitivity to the lower strain levels needs to be developed.

Figure 6-32 demonstrates the correlation between the effective strains from the ML sensor and the effective strains from the FE simulation results of the aluminum I-beam specimen at three different loading levels. It should be noted that only the results from the bottom of the film (0<y<1) and from the top of the film (2<y<3) are presented in the figure because the maximum effective strains exist in those area. As it can be seen in the figures, the correlation will close to one as the loading levels increases and also the accuracy of the ML sensor improves by increasing applied load.
Figure 6-29: The von-mises stress distribution of the aluminum I-beam specimen under four-point bending test at load 180 kN ($\varepsilon_{11} = \text{longitudinal strain}$)
<table>
<thead>
<tr>
<th>Black and white raw images</th>
<th>Light intensity from the raw images</th>
<th>Change of the light intensity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Non-loaded</td>
<td><img src="image1" alt="Non-loaded" /></td>
<td><img src="image2" alt="Non-loaded" /></td>
</tr>
<tr>
<td>63 kN</td>
<td><img src="image3" alt="63 kN" /></td>
<td><img src="image4" alt="63 kN" /></td>
</tr>
<tr>
<td>90 kN</td>
<td><img src="image5" alt="90 kN" /></td>
<td><img src="image6" alt="90 kN" /></td>
</tr>
<tr>
<td>126 kN</td>
<td><img src="image7" alt="126 kN" /></td>
<td><img src="image8" alt="126 kN" /></td>
</tr>
<tr>
<td>153 kN</td>
<td><img src="image9" alt="153 kN" /></td>
<td><img src="image10" alt="153 kN" /></td>
</tr>
</tbody>
</table>

Figure 6-30: Black and white raw images, light intensity images and change of the light intensity distribution images from the ML film coated on aluminum I-beam specimen at different loading levels.
Figure 6-31: The effective strain distribution from FE simulation and ML sensor on I-beam aluminum specimen at different loading levels
Figure 6-32: The effective strain from the FE simulation versus the effective strain from ML sensor of I-beam aluminum specimen at different loadin levels.
CHAPTER VII

VISUALIZATION OF CRACK PROPAGATION AND STRESS DISTRIBUTION

7-1 Overview

In this chapter ML sensor is employed to visualize the crack propagation on two different fiber reinforced prismatic concrete specimens. The ML materials applied to the concrete surface in form of the paint and thin film. Also, the application of the ML sensor in a prototype design aspect is studied on an industrial prototype product and the stress distribution from FE simulation is compared with ML emission distribution.

7-2 Visualization of the Crack Propagation on a Concrete Specimens by using ML Sensor

ML materials can be used as a new non-contact sensor to detect and visualize the active crack and fracture on structural surfaces. Instantaneous results, simple to use and direct visual observation of the real time active crack propagation are the most interesting advantages of using ML sensor as a new fracture prediction technique. Therefore, in this thesis, two different tests are performed on concrete specimens in order to visualize the crack propagation on structural surfaces.

In the first test, SAOED particles are mixed into the optical epoxy (west system, 105 epoxy resin and 206 hardener) with concentration ratio of 1 to 3 and sprayed onto the surface of the prismatic fiber reinforced concrete specimen. The dimensions of the concrete specimen are 4x4x14 in\(^3\). Figure 7-1 shows the experimental test set-up for the first test. Four point bending test is performed on the specimen to generate the cracks. The specimen is photoexcited for two min, the aged in the dark for 5 min to allow the PL light intensity reaches to the reasonable level before conducting the test. All the images are captured at speed 20 fps by using the CCD camera during applying the load. Load is applied linearly proportional to
the extension up to 2.55 mm with the fixed cross-head speed of 0.05 mm/s. Figure 7-2 demonstrates the extension-load curve for the concrete specimen.

The sequential original black and white images are shown in Figure 7-3 and Figure 7-4 with the total number of 36 images. Only the images from the extension 2.46 mm to 2.55 mm are displayed in the figures and the time interval between the images is 0.05 s. In order to have a better visualization of the crack propagation on the concrete specimen, Matlab image processing tool is used and the distributed change of the ML light emission is presented in Figure 7-5 and Figure 7-6. According to the figures, it appears that the initial crack propagation arises in the 12th image which is corresponded to the cross-head displacement 2.488 mm. The corresponding load to this extension is 26.98 kN which is less than the maximum load (27.18 kN). Thus the first crack is generated after the ultimate load and continues to propagate thereafter.
Figure 7-1: The test setup of the prismatic fiber reinforced concrete specimen sprayed with ML sensor under the four-point bending test.

Figure 7-2: Load-displacement curves of a prismatic fiber reinforced concrete specimen.
Figure 7-3: Black and white raw images of crack propagation on a short concrete beam at different loading levels by using the ML sensor- part 1
<table>
<thead>
<tr>
<th>Displacement = 2.506-2.529 mm</th>
<th>Displacement = 2.529-2.553 mm</th>
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<tbody>
<tr>
<td><img src="image1.png" alt="Image" /></td>
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</tr>
<tr>
<td><img src="image19.png" alt="Image" /></td>
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</table>

Figure 7-4: Black and white raw images of crack propagation on a short concrete beam at different loading levels by using the ML sensor - part 2
Figure 7-5: Visualization of crack propagation on a short concrete beam by using the ML sensor after image processing- part 1
Displacement = 2.51 - 2.53 mm

Figure 7-6: Visualization of crack propagation on a short concrete beam by using the ML sensor after image processing - part 2
The second test is performed on a cylindrical fiber reinforced concrete with 4 in diameter and 8 in height. SAOED powder is mixed with optical epoxy from west system with concentration ratio 1:3. Unlike the first test, in this experiment a thin ML film is fabricated by Doctor Blade method with the thickness of 20 mil and attached to the circular face of the concrete specimen. Figure 7-7 demonstrates the experimental test set-up.

Figure 7-7: Experimental test setup of crack propagation on the fiber reinforced concrete cylinder coated with ML sensing film
The specimen is photoexcited with a commercial light for two min, then aged in the dark for 5 min before doing the test. The compression load is applied linearly proportional to the cross-head movement at speed of 0.05 mm/s. All the images are captured with a high speed CCD camera at speed 20 fps. Figure 7-8 shows the extension versus compression load curve for the cylindrical concrete specimen.

![Load-extension curves of the cylindrical fiber reinforced concrete specimen](image)

The sequential black and white images are shown in Figure 7-9, Figure 7-10, and Figure 7-11 with the total number of 36 images. Only the images from the extension 3.664 mm to 3.734 mm are displayed in the figures and the time interval between the images is 0.05 s. The distributed change of the ML light emission for the sequential images are presented in Figure 7-12, Figure 7-13, and Figure 7-14. According to the figures, it appears that the initial crack propagation arises in the 3rd image which is corresponded to the extension 3.668 mm. The corresponding load to this extension is 147.6 kN which is less than the maximum load (151.9 kN). Similar to the first test, the initial crack propagation in this test is generated after the ultimate load too. The results shows the ML materials can be used as a new non-contact sensor to visualize the crack propagation and successfully demonstrates the capability and potency of ML materials.
Figure 7-9: Black and white raw images of crack propagation on a concrete cylinder at different loading levels by using the ML sensing film- part 1
<table>
<thead>
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<th>Displacement = 3.699-3.711 mm</th>
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</tr>
<tr>
<td><img src="image11" alt="Image" /></td>
<td><img src="image12" alt="Image" /></td>
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</table>

Figure 7-10: Black and white raw images of crack propagation on a concrete cylinder at different loading levels by using the ML sensing film- part 2
Figure 7-11: Black and white raw images of crack propagation on a concrete cylinder at different loading levels by using the ML sensing film - part 3.
Figure 7-12: Visualization of crack propagation on a concrete cylinder by using the ML sensing film after image processing- part 1
Figure 7-13: Visualization of crack propagation on a concrete cylinder by using the ML sensing film after image processing- part 2
Figure 7-14: Visualization of crack propagation on a concrete cylinder by using the ML sensing film after image processing- part 3
7-3 Case Study

In order to validate the ML sensor performance on complex geometry samples, the ML sensor is applied on a new design of an industrial product. Figure 7-15 displays the prototype shower faucet which is used for visualization of stress by using ML sensor.

Figure 7-15: Prototype shower faucet sample, MOEN Inc.
Figure 7-16 shows non-coated and coated with ML sensor specimens. The specimen is fixed by means of an especial fixture designed by MOEN and mechanical load is applied linearly up to 200 N with a rigid tip. Speed of the instron machine cross-head is selected as 0.5 mm/s. Figure 7-17 demonstrates the fixture and experimental test setup. The aim of this test is to visualize the stress distribution on the prototype product and find the location of the highest stress concentration by using the ML sensor.

![Figure 7-16: Non-coated and coated specimens](image)

During applying the load, all the images are captured by a high speed CCD camera at speed 10 fps. Figure 7-18 shows the sequential original black and white images of the sample coated with ML sensor and Figure 7-19 displays the sequential images of the ML emission after image processing. The ML emission is simply calculated by subtracting the light intensity of the first image from each single image.

A FE model is developed by using available commercial software (ABAQUS 6.13-2) to compare the numerically calculated von-mises stress distribution with the ML emission distribution. According to the Figure 7-20 the distribution of the ML light emission is correlated to the von-Mises stress distribution from FE simulation and also the location of the maximum stress concentration is very well matched with the FE simulation results.

The results indicate the promising application of ML sensor not only as a non-contact visual sensor but also as a new technique in NDT application and prototype design aspect.
Figure 7-17: Experimental test setup of the prototype sample under mechanical load
Figure 7-18: The sequential black and white images of ML sensor applied to the prototype sample during the load.

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Figure 7-19: The sequential images of change of the ML emission distribution to the prototype sample during the load
Figure 7-20: Stress distribution from the FE simulation results (a) and change of the light intensity distribution from the ML sensor (b) for the industrial prototype specimen at load 200 N
8-1 Conclusions

In this study, characteristics of two most promising ML stress sensing materials, called SAOE and SAOED, are evaluated. The performances of SAOE and SAOED composite disc shape specimens are compared in two different test matrices. Because ML sensors are inherently sensitive to the maintenance and test conditions, the SAOE and SAOED specimens have been tested under the same condition in each test matrix. The following are summaries of conclusions based on the composite disc shape experimental observations.

1) According to results from the first test matrix, both SAOED and SAOE showed increases of the light intensity change as the strain rate increases. Certainly, light emission of SAOE and SAOED is strain rate-dependent. In terms of the sensitivity (light intensity change per unit force), SAOED showed higher sensitivity than SAOE.

2) The results of the first test matrix also demonstrate the repeatability of both SAOE and SAOED sensors, which is a key factor for sensors.

3) The results of two test matrices illustrate that maximum light intensity of the SAOED material is more sensitive to changes in strain rates than SAOE.

4) Although SAOE saturates quickly, the SAOED does not saturate at the defined strain rates. This implies that the performance of SAOED is better than SAOE’s as an ML stress sensor.
5) SAOE showed saturation of light emissions; however its initial sensitivity to the strain was higher than SAOED. This implies that SAOE is more suitable to sensors for detecting dynamic active cracks.

Also, a predictive ML transduction model is proposed to predict the ML light intensity from SAOED thin-film coating sensor under mechanical loading. Comprehensive experimental test data with different stress levels and strain rates at different PL decay time intervals is studied to develop the ML transduction model. Mechanical energy applied to the ML sensing film is transduced to incremental change of total light emission ($\Delta I$), which can be divided into a net ML emission ($\Delta I_{ml}$), stress-free PL decay ($\Delta I_{pl}$), and additional stress-induced PL decay ($\Delta I_{\mu}$). MGGP is utilized as a tool to generate the predictive models of three components of the total ML+PL light intensity. The results from the predictive model are in good agreement with experimental results. A sensitivity analysis is performed in order to further validate the models. The interaction of the ML phenomena with PL decay and effects of strain rates and PL decay time intervals were investigated, and the results showed that the relative total ML+PL light intensity emission has a linear relationship with the stress square or mechanical strain energy. By combining PL decay effects on the ML phenomenon, it was proved that the mechanical strain energy is transduced to optical energy. Findings from this investigation could lead to a reassessment of ML behavior, both physically and phenomenologically. The proposed predictive models can be used for the design of ML sensing film and its applications to the calibration process.

A new calibration model is proposed to predict the full-field effective strain measurement from the ML thin sensing film for the first time. The incremental values of step time ($\Delta t$), change of the ML light intensity ($\Delta LI$), and effective strain from previous step are incorporated into the MGGP model to predict a calibration equation of the change of effective strain in each time step. The verification of the calibrated effective strain values showed that the proposed calibration model can predict the real-life strain values accurately. Three different experimental tests were conducted in order to validate the performance of the proposed calibration model. For all three experiments, FE simulation model were developed and the distributed effective strain values from ML sensor were compared with FE simulation results. The comparison of the ML and the finite element effective strain values clearly showed that the ML sensing
film and the calibration model can be effectively used as a new non-contact full-field strain sensor. However, our proposed model is an empirical model that can predict ML light intensity from experiments conducted with the same coating design (i.e. thickness and loading ratio of ML particles to resin). It is important to note that for an ML coating with a different thickness or concentration ratio, the parameters of the proposed model will need to be recalibrated with new test data.

ML sensor was employed to visualize the crack propagation on two different fiber reinforced prismatic concrete specimens. Also, the application of the ML sensor in a prototype design aspect was studied on an industrial prototype product and the ML distributed light emission indicated a good agreement with stress distribution from FE simulation.

In summary, this research proposed and realized a new distributed stress sensor and NDT method by using ML sensing materials. Findings from this study lead to a reassessment of ML behavior and are crucial in development of a commercial non-contact optic sensor to be used as a new full-field strain measurement technology.

8.2 Future Works

Future works in ML sensor can be summarized as follows:

1) The validation results in this research showed that the ML sensor is not sensitive enough to measure the effective strain values for lower strain levels and 0.0005 is the minimum reliable effective strain value that can be measured by the proposed calibration method. In order to improve the sensitivity of the ML sensor three different approaches can be identified. The first approach is to use the total ML light emission instead of ML light emission and include the instantaneous PL decay effect in the calibration model. The second approach is to improve the sensitivity of the ML particles by changing the ML crystals properties. This includes the manufacturing process of the ML particles, changing the size and shape of the ML particles, and etc.. For instance, using different size or shape of ML particles would change the external surface of the ML particles and can affect the transition phase of the mechanical energy from optical epoxy matrix to the ML particles which yields to different performance of the ML sensor. And finally, the third approach is to add fillers (i.e. micro glass fibers) to the sensor to increase the ML sensitivity. These fillers should be optical transparent materials not to block the light emission of the ML particles. Thus, more
researches should be conducted in order to improve the sensitivity of the ML sensor following three different approaches.

2) All the experimental tests in this study have been conducted in a room temperature. Based on our observation, ML light emission is very sensitive to the temperature and any increase or decrease in room temperature can change the ML light emission and ML sensor performance accordingly. Therefore, a comprehensive experimental study should be conducted in order to investigate the effect of temperature on ML performance. Also, other environmental conditions (i.e. humidity and durability) should be considered to evaluate the performance of the ML sensor.

3) In this study, all the experimental tests have been conducted in the laboratory under ideal conditions. In order to evaluate the performance of the ML sensor as a potential new non-contact sensor, in-field tests need to be performed. Conducting the in-field test is a crucial step for developing the new strain measuring technique and can reveal the challenges of using the ML sensing film in real-life.


95. *PHOTOMULTIPLIER TUBES, Basics and Application*.


113. PhotoStress Instruments, Tech Note TN-702-2, VISHAY PRECISION GROUP.


