

Thermal, Mechanical, and Optical Characterization of Luminescence - Doped PDMS Thin Film Sensors

Andrei Fendley, CRESH student, Germantown High School, Kian Ziai, Arlington High School
Mentor: Dr. Firouzeh Sabri, Department of Physics and Materials Science, University of Memphis, Memphis, TN

Background

Luminescence means giving off light; most things in our world produce light because they have energy that originally came from the Sun, which is the biggest, most luminous thing we can see. It is a mechanism by which (light) photons are created without the use of heat [1,2,3].

Triboluminescence is a mechanism by which (light) photons are created as a result of friction or instant force (pressure) experienced by the material. The property that some materials become luminous upon being scratched, crushed, or rubbed. Examples of substances exhibiting triboluminescence include the minerals fluorite (CaF₂), sphalerite (ZnS), and wintergreen LifeSavers! [4,5,6].

Current Challenges: Almost all luminescing materials are currently available in a powder form with particles sizes in the nm-um range. Such small particle sizes create challenges for design of advanced sensors due to the hazardous nature of nanoparticles.

Goal of the Study: To create thin-film flexible sensors that are triboluminescence and the powder is fully encapsulated and can produce sufficient output (light emitted) that can be detected above noise level. Full characterization of all the material properties is necessary in order to understand the limitations of the performance of the sensor

Sample Preparation

Polymer used for encapsulation : Sylgard 184 (part A and B), also referred to as PDMS in this poster

ZnS:Mn was the triboluminescence powder.

Mixed polymer and ZnS:Mn at different ratios

We stirred it to make a homogeneous mixture

Outgassed the mixture in a vacuum oven

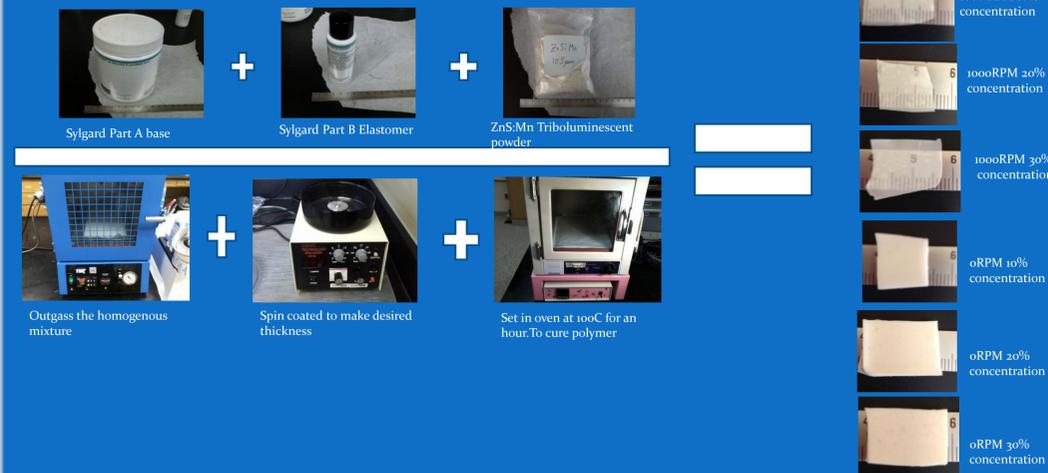
Used the spin-coater to make thin films on a sacrificial base, cured, then peeled from the substrate.

Characterized the material properties of the films made using the techniques mentioned in the section below.

RPM/Percent.	10	20	30
0	✓	✓	✓
1,000	✓	✓	✓
1,500	✓	✓	✓
2,000	✓	✓	✓

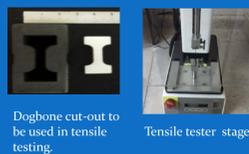
Table 1: List of samples made for this study.

Flow chart showing synthesis process:



Sample Characterization

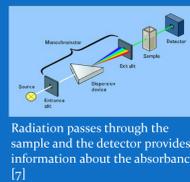
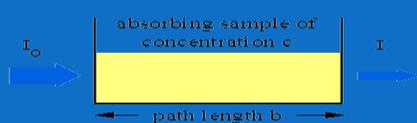
Tensile testing: The tensile behavior of the doped polymers was tested using an ESM301 Motorized test Stand.



UV-Vis Spectroscopy: The EVOLUTION 220 UV-Visible Spectrophotometer was used to measure absorbance / transmission in the UV and visible light range for each sample type.

Beer-Lambert Law is the linear relationship between absorbance and concentration of an absorbing species [7,8].

$A = \epsilon \cdot b \cdot c$
A = measured absorbance
 ϵ = molar absorptivity coefficient
B = pathlength
C = Analyte concentration



Drop tower tests: Intensity of emitted signal (light seen) as a function of impact force was measured using a home built drop tower and Agilent 6.5 Digit Multimeter connected to the sample chamber.

Expansion rate of Sylgard 184+ ZnS:Mn as a function of temperature: The thermal response of the sensor was measured by means of a MK 1000 Temperature Controller positioned on the stage of a Unitron Examet-4 Reflective Microscope.

References

[1] http://depts.washington.edu/crsh/crsh/theses/thesis_luminescence_phenomena.htm [2] Sabri, Firouzeh, Kyle J. Lynch, and Steve Allison. "Polymer-Encapsulated Phosphor Particles for In Vivo Phosphor Luminescence Applications." *International Journal of Polymeric Materials and Polymeric Biomaterials* 64.3 (2015): 690-694. [3] Allison, Stephen W., et al. "In Vivo X-Ray Imaging of Phosphor-Doped PDMS and Phosphor-Doped Aerogel Biomaterials." *International Journal of Polymeric Materials and Polymeric Biomaterials* just-accepted (2015). [4] Lotters, J. C., et al. "The mechanical properties of the rubber elastic polymer polydimethylsiloxane for sensor applications." *Journal of Micromechanics and Microengineering* 7.3 (1997): 145. [5] Blasse, George, B. C. Grahnaier, and B. C. Grahnaier. *Luminescent materials*. Vol. 44. Berlin: Springer-Verlag, 1994. [6] Harvey, E. N. *A History of Luminescence*. Philadelphia: American Philosophical Society, 1957. [7] http://hplc.chem.shu.edu/NEW/Undergrad/Molec_Spectr/Lambert.html [8] http://faculty.sdmiramar.edu/faculty/sdcd/garces/LabMatters/Instruments/UV_Vis/Cary50.html

Results

Optical Micrographs of Thin Film Sensors:



Optical microscope images of ZnS:Mn particles cured in a polymer mix under different spin conditions. Image magnification: X 5.

Particle clusters formed when mixed with the polymer

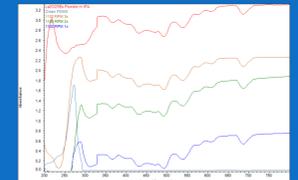
UV-Vis Spectra of Thin Film Sensors:



(a) Triboluminescing particles and PDMS



(a) Triboluminescing particles and PDMS



(c) Thermo-luminescing particles and PDMS

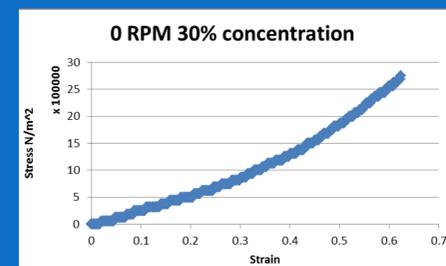
The signature peaks associated with each material (polymer, luminescing nanoparticles) were measured separately and then compared with the combined material behavior. The amount of absorbance depends on the RPM which effects the path length (thickness) of sample as well as the concentration. The graphs follow the Beer Lambert law.

Effect of Spin Speed on Thickness:

Thin Film Sensor thicknesses (um)				
RPM	0	1000	1500	2000
Doping level				
10%	1620	100	86	66
20%	2000	140	93	66
30%	2613	186	140	73

Table 2: Sensor thicknesses measured as a function of RPM.

Tensile Behavior:



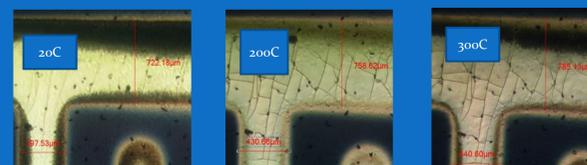
Drop Tower Tests:

The intensity of the emitted light due to the impact force depended on the particles concentration. The force of impact was kept constant for all samples. Some samples were bright enough to be seen by naked eye.

RPM	Volts(v)		
Concentration	10%	20%	30%
0	0.021*	0.0223*	0.008*
1000	0.008*	0.0063	0.0056
1500	0.007*	0.0063	0.008
2000	.0063*	0.007	0.0053

*Light was seen by the human eye

Table 3: Emission intensity values



Temperature Tests:

Base temperature of polymer samples was raised from room temperature up to 300 C (almost) and cycled back to room temperature, while observing the fiducial marks under a microscope at a fixed magnification in order to measure the expansion rate of the material. Only pure PDMS was tested since the thin films created would buckle up and curl and therefore accurate distance measurements was not possible.

Table 4: Expansion rate as a function of temperature for pure PDMS polymer

ΔT	ΔX	ΔY
C	um	um
0	0.00	0.00
20	0.00	0.00
40	0.00	23.18
60	6.63	23.18
80	6.63	23.18
100	36.44	43.06
120	39.75	43.06
140	53.00	59.63
160	53.00	59.63
180	69.57	59.63
200	75.19	59.63
220	86.13	59.63
240	86.13	69.56
260	109.32	82.81
280	122.57	89.44
300	125.89	89.44
320	135.82	106.00
340	135.82	106.00
320	135.82	106.00
300	122.57	86.13
280	109.32	86.13
260	102.70	86.13
240	92.76	86.13
220	92.76	62.94
200	72.88	62.94
180	46.69	46.37
160	36.44	23.18
140	13.25	23.18
80	69.57	9.93
60	3.31	6.63

Summary, Conclusion, & Future Work

- A series of thin film impact sensors were created such that the sensing powder was fully encapsulated inside a polymer, allowing for safe handling
- We proved that it is possible to get detectable output signal from um thick films
- The composite material (thin film sensor) was still elastomeric at a doping concentration of 30%
- Temperature dependence expansion measurements proved to be technically challenging and not completed during this portion of the study.
- We would also like to perform spectral studies before and after the drop tower test to see if we can identify damage to the sensors (dopants) as a result of the drop tests. We did not have time to complete that expt. this year.
- In the future we will repeat this study with aerogels instead of PDMS. Other factors we could consider changing would be thermoluminescent powder, different concentrations of powders, and different RPMs. We will also do consistent damage on the polymers and see using the spectrometer how damage affects the absorbance of the polymers. Luminescence is a very good sensor to be used in various situations.

Acknowledgments

Dr. Firouzeh Sabri, Department of Physics and Materials Science, University of Memphis, Memphis, TN, Kate Mitchell, Dept. of Physics and Materials Science, University of Memphis, TN