

Investigating Properties of Fluorescent and Phosphorescent Materials



Application Note

KEYWORDS

- Phosphorescence
- Photoluminescence
- Time-resolved analysis

TECHNIQUES

- Fluorescence

APPLICATIONS

- Materials analysis
- Phosphor quality control

Remember a scene like this from childhood? Your folks just gave you 25 cents, you slid the quarter into the arcade vending machine, turned the handle, and were rewarded with a flimsy plastic bubble encapsulating a neon-green bouncy ball. Your next priority: Race home, hold the ball under the brightest light bulb you can find for 10 minutes, then go into a room with all the lights off and watch how brightly the ball glows. Instant entertainment.

That was great fun some decades ago, but now you find yourself a bit older and more likely to be reading articles about the latest in science and technology. So, what was going on with those glow-in-the-dark toys from your childhood? And are those same properties at work today in more vital applications and industries?

Introduction: Phosphors in Today's World

Photoluminescence is the property of a substance to absorb photons within some energy threshold and then re-emit the energy at longer wavelengths. Typically, photoluminescence

is split into two classes, fluorescence and phosphorescence.

We're all familiar with fluorescent paint, which can bring any party to life with a proper distribution of UV black lights. But this UV excitation is consistently needed for fluorescence to occur since it exhibits such a fast decay, usually on the nanosecond or faster time scale. What makes glow-in-the-dark materials special is that even when the excitation lights go out, there is still glow. This is due to a much slower transfer process involving forbidden quantum states that can take seconds to minutes to hours for total relaxation.

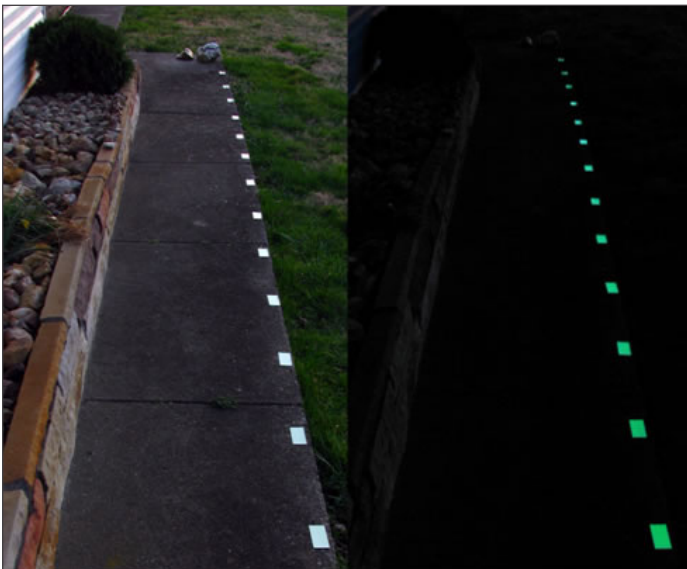


Figure 1. Photoluminescent safety markings line walkways and other paths.

Today, photoluminescent materials have been harnessed for many practical and life-saving uses beyond children's toys (**Figures 1, 2**), and in the post-9/11 world have become written into International Building Code (IBC) and International Fire Code (IFC) standards for egress and stairwell markings (Jessup, 2018). Section 1025 of the 2015-IBC states that self-luminous markings are required on all exit route steps,

landings, handrails, perimeters, obstacles and doors in many classes of buildings of a certain size, including hospitals, schools, businesses, hotels, public assembly venues and residences (American PermaLight, 2018). The ability of these markings to light the way is not arbitrary; there are standard ASTM methods to check the photoluminescence and quantify its decline to a millicandela per square meter level (mcd/m²) specification.



Figure 2. Photoluminescent tape has many safety applications.

Optical Analysis Tools from Ocean Insight

Ocean Insight is a well-known supplier of tools for the investigation and quantification of fluorescent and phosphorescent materials. Recently we've expanded our sensing toolbox to include something very exciting: a highly tunable, variable waveform LED source for both steady-state and time-resolved analyses. The LSM-Series LED Light Source is a multi-component package that allows versatility in an intuitive form factor (**Figure 3**). The touch-screen controller module supports steady-state and continuous-wave-

form emissions at a user-defined power level. Waveforms include square, sine and triangle by default but also support external waveforms driven via a function generator. Narrowband LEDs are available between 265-880 nm, and a warm white broadband source is also available for absorbance and reflectance applications.



Figure 3. LSM Series LEDs operate via a smart controller that allows users to vary power level and waveforms.

To demonstrate the value of these new LEDs for phosphorescence we acquired several phosphor materials and quantified them using Ocean Insight systems.

Manufacturers of photoluminescent safety markings need stronger methods and tighter QC protocols to ensure consistent products. Researchers investigating the next generation of these materials need more advanced tools to quantify the new materials and compare them to the old. Architects, safety inspectors and regulators need new approaches to verify that these markings meet the current standard, and could use additional parameters to define what “good” and “bad” markings will look like in the future.

Experimental Setup

The following phosphorescent compounds were obtained from Millipore-Sigma:

- Strontium aluminate doped with Europium and Dysprosium. You may be more familiar with this compound by its commercial name, LumiBrite, which is often used to make watch hands glow in the dark.
- Barium Magnesium aluminate doped with Europium
- YYG 557 230 isophor®

We then configured these Ocean Insight spectroscopy products for the setup:

- LSM-Series LED light sources
- Ocean FX high speed spectrometer. We used the OCEAN-FX-XR1 extended-range option (200-1025 nm).
- QR600-7-UV 6-around-1 premium fiber optic reflection probe
- OceanView operating software

For each compound a small mound of raw powder, about 0.2 g, was poured into a plastic weigh boat. The fiber optic probe was positioned over the powder facing business-end down, and brought as close as possible without contacting the material, within 3 mm.

The luminosity of these materials is nothing short of amazing, and several folks around the lab made the comment that it appears these materials somehow emit more photons than they absorb (of course, we are simply not seeing the UV excitation nearly as efficiently).



Steady State Observations: Wavelength Optimization

LSM-Series LEDs are versatile, offering a wide range of wavelength options. By working through the easily changeable LED selections, we can quickly find the optimal excitation wavelength for each of the compounds to be measured.

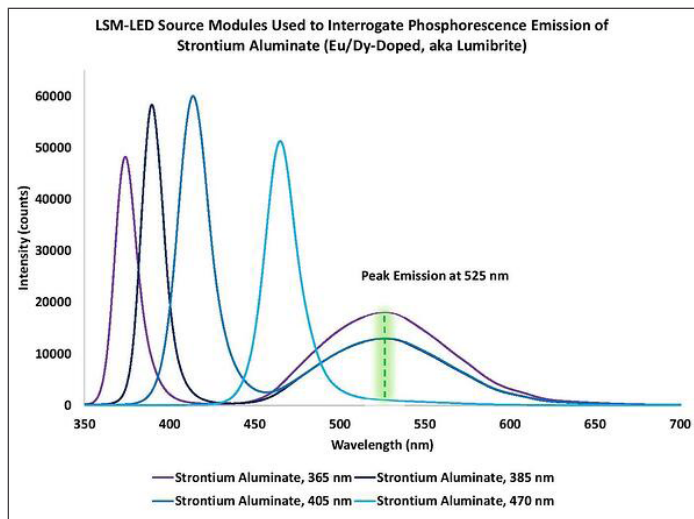


Figure 4. Strontium aluminate phosphorescence emission response

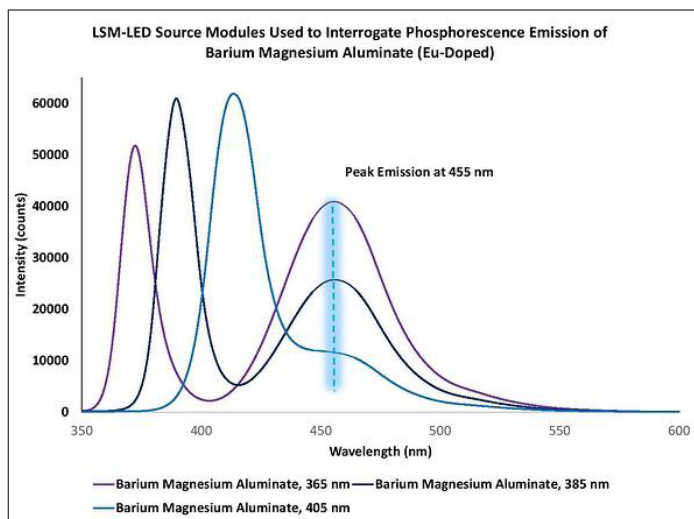


Figure 5. Barium magnesium aluminate phosphorescence emission response

We discovered that 365 nm is an ideal excitation wavelength for the two compounds doped with rare earths (Figures 4, 5), while 405 nm is the best choice for the YYG 557 230 isophor® ma-

terial (Figure 6). For safety-marker manufacturing, for example, knowing the precise peak maxima for these chemicals is useful during QC and post-implementation inspection steps. Also, the broadband distribution of the chemical emissions provides a deeper level of understanding and quality verification.

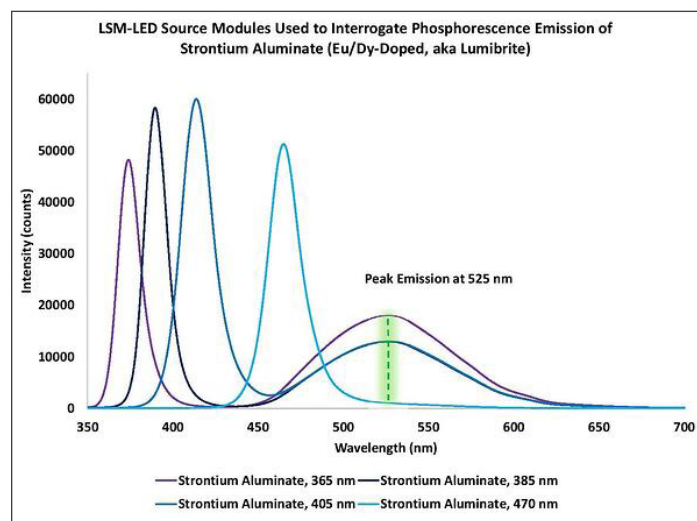


Figure 6. Isophor exhibits strong phosphorescence emission response at 560 nm.

Time Resolved Analysis: Waveform of Your Choice

Now that the optimal LEDs have been determined, we can begin to take advantage of another strength of the LSM-Series LEDs -- their tightly controlled and highly customizable waveform options. Time-resolved spectroscopy offers advantages not found in the steady-state realm, which are enabled by the high-speed Ocean FX broadband spectrometer. With 10-microsecond integration time and a unique Burst Mode option, the Ocean FX can provide much deeper insight into the inherent physical parameters of a compound that would remain hidden with traditional spectrometers.



Square waves, or a simple pulse, have many uses across industry and are a standard approach to quantifying the decay time of a photoluminescent material. The LSM-Series LED module was set to a 20 Hz square wave feeding to the 365 nm excitation, and the resulting 525 nm emission was observed from the Lumibrite compound (**Figure 7**).

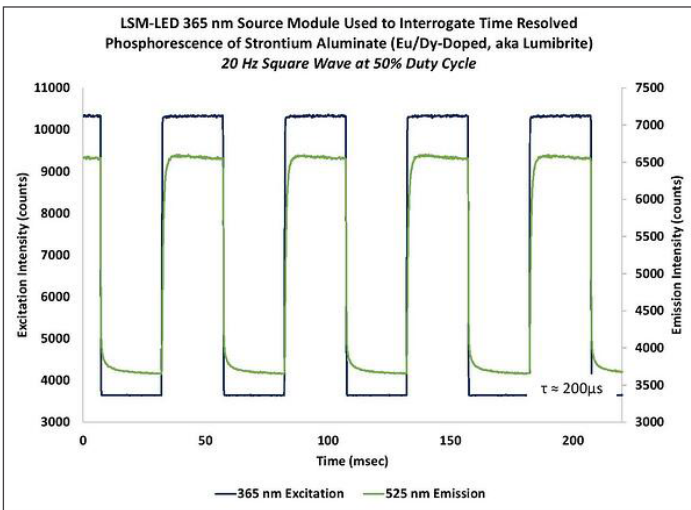


Figure 7. With LSM Series LEDs, users can adjust waveforms to better quantify the decay time of photoluminescent materials.

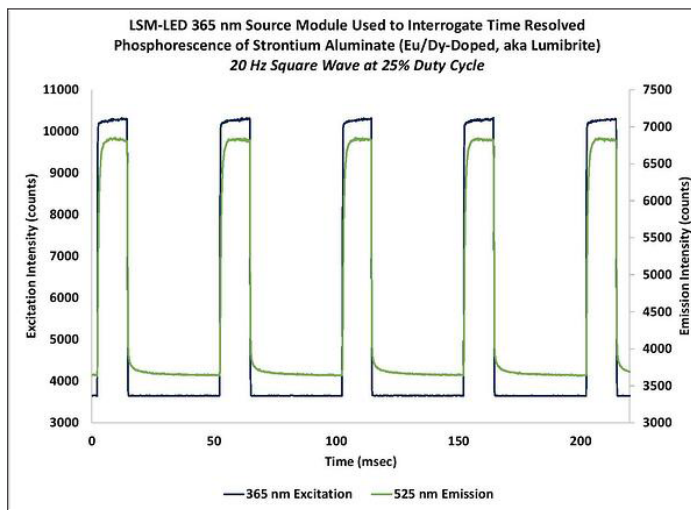


Figure 8. The duty cycle of an LSM LED can be adjusted to control phosphorescence decay times.

Perhaps we've observed this and decided not so much time is needed for the LED to be on, but we could use additional off-time to let the phosphorescence decay a bit further. The LSM-Se-

ries module allows total control of square-wave duty cycles to adjust wave profiles based on your compound. **Figure 8** shows the duty cycle shifted to 25% so that we achieve our desired additional off-time while maintaining the overall system clock at 20 Hz.

Our other aluminate compound exhibits a slightly different response, where the upcharge is essentially instantaneous and then begins to relax despite excitation power still on (**Figure 9**).

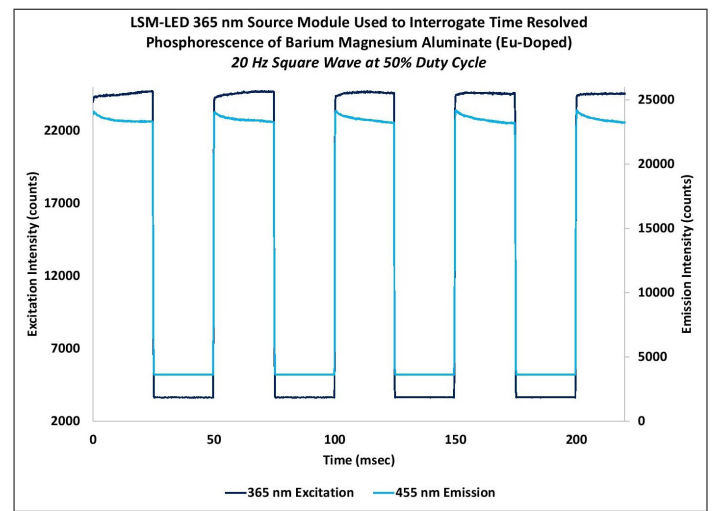


Figure 9. Phosphorescence emission of barium magnesium aluminate.

Another popular waveform in the world of electrical engineering is the classic sine wave, which has its own set of mathematical uses when working with phase shifts. Setting the LSM-Series module to the sine wave setting at 100 Hz, the Lumibrite compound was observed with the 365 nm excitation. This allows us to calculate the phase shift in degrees, which is another inherent optical property of the compound in its current state (**Figure 10**).



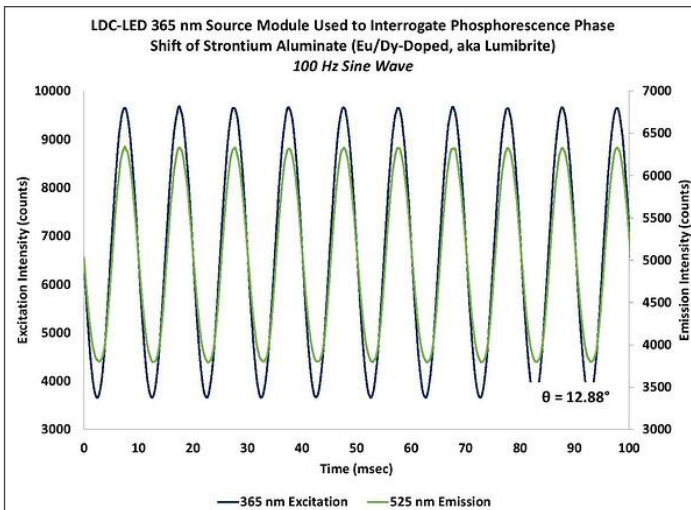


Figure 10. Sine waveforms made possible with LSM LEDs provide a different level of insight into the phosphorescence phase shift of strontium aluminate.

Versatility continues with options for a triangle wave and for custom waveforms generated from a function generator. For the researcher developing a new luminescent material, the luminescent safety-marker manufacturer monitoring their product, and the inspector ensuring these products are providing sufficient guidance in case of emergency, our spectral tools help us to identify, qualify and uncover additional dimensions of each compound.

Conclusion

Whether taking initial steady-state observations of a material or diving into microsecond-scale time-resolved analysis, the LSM-Series LED module and Ocean FX high speed spectrometer work in harmony to offer the precision results you expect. Work with us to expand your optical toolbox with the new LSM-Series LED Light Source Kit, and be prepared for the next photoluminescent compound that bounces your way.

References

American PermaLight. (2018). IBC/IFC Compliant UL1994 Egress Path Markings. Retrieved from American PermaLight: <https://www.american-permalight.com/ibc-ifc-compliant-ul1994-listed-egress-path-markings/>

Jessup. (2018). Product Compliance of Photoluminescent Films. Retrieved from Jessup Adhesive Coated Films: <https://www.jessupmfg.com/faq/>



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