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Daniel Duong

The Synthesis and Preparation of Upconverting and Downconverting Phosphors for Sensing Applications

Faculty Sponsor

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Introduction

Thermometry has always been important for scientific and industrial applications. Its usage ranges from environment sensing and climate control to medical uses. Thermometry plays an important role in determining if a region of space is suitable to the application at hand, from habitable conditions and possible hazards to the usage of electronics. It helps determine how certain materials will behave, thus informing the best way to prepare accordingly.

Just as the applications of thermometry have a wide array, so are the types of thermometry, each with their respective strengths and weaknesses. One type of thermometry uses thermal expansion. A classic example of this would be the mercury thermometer. The mercury expands as it is heated, and the volume is then used to determine the temperature. While useful for a broad range of applications, a downside is that it must be in contact with the material itself. Other types of thermometry require a medium to travel through, are not highly accurate, or cannot detect a wide range of temperatures (Aryal, 2018). For the purposes of space exploration and settlement, these factors play a significant role in determining the type of thermometry used. As a result, phosphor thermometry is a strong contender to be the best way to deal with these issues.

Phosphor thermometry operates by using photoluminescence, thus avoiding all of the issues above. This occurs by shooting a short pulse laser at the phosphor, enabling the electrons to reach an excited state, and measuring the emissions from the electrons as they fall back to ground level (Aryal, 2018). This is why the phosphors are described as upconverting and downconverting. For the purposes of this experiment, the decay rate will be used to determine the temperature, as it is the most widely-used method, and a method which is compatible with a configuration which will be stated later (Allison et al, 2017). Phosphor thermometry does have one weakness, however. The phosphors are in powder form, which makes them difficult to handle.

Due to their small particle size, a variety of issues arise. They will easily cross contaminate with other materials, messing up measurements. In addition, their high surface charge causes them to adhere to most materials as well as cluster together, creating a non-uniform amount of the powder for sensing. Outside forces will cause the powder to disperse. In this form, the phosphor powders are hazardous, not only to people, but also to the environment, therefore necessitating Environmental Protection Agency regulations. Lastly, the phosphors are reusable, but not recoverable in this state, and can only be utilized on a limited number of surfaces (Rietema, 1991). Thus, the proposed

solution is to encapsulate the powders in Sylgard 184.

Sylgard 184 is classified under the family of polydimethylsiloxane (PDMS). PDMS is a common elastomer used for space exploration due to its insulating abilities, flexibility, non-flammability, and easy synthesis. Especially critical in this experiment is PDMS's characteristic of being inert (Fontenot et al, 2016). This allows it to be doped with different materials, enabling the creation of composites. By forming a composite with the phosphor powders, the phosphor itself becomes easier to handle and thus, more applicable. PDMS is also transparent in the visible spectrum, thereby not distorting the photoluminescence of the phosphor powders (Allison et al, 2017).

The purpose of this experiment is to create a method in which the phosphor powders can be reliably encapsulated without the loss of the powder. In addition, the identification of the best suited temperature dependent bandwidths will be identified. Finally, an investigation was made to see if the encapsulation process had any effect on the excitation or emission behavior of each compound.

Materials

Thirteen different phosphor powders were acquired for this research. Eight of the powders were acquired from Intelligent Materials, and the other five were acquired from the University of Louisiana, Lafayette. The powders acquired from Intelligent Materials were Yttrium, Lanthanum, Lanthanum/Gadolinium, 620 Nitrate, 630 Nitrate, Zinc Sulfide UV (Green), Bismuth, and Zinc Sulfide UV (Blue). From the University of Louisiana, Lafayette, three Europium Tetrakis powders and two Magnesium Tetrakis powders were acquired (Fontenot et al, 2012; 2015). Sylgard 184 was used as the encapsulation elastomer.

Procedures

The same general procedure was followed for the majority of the composites. First, Sylgard 184 and the curing agent were mixed at a ratio of ten to one to form PDMS. This ratio was predetermined by previous experiments. Following this, the sample was outgassed for four to five minutes. The PDMS was mixed with the phosphor powder at a ratio of twenty-two to one and mixed by hand thoroughly for three to four minutes. The sample was outgassed a second time for four to five minutes. Within the next three to four minutes, the mixture was poured into the dog bone mold and outgassed completely. Lastly, it was cured in an oven at 100 °C for an hour, cooled, and removed from the mold. Aside from this synthesis method, the Europium Tetrakis was

ground from crystals into a powder. As for Bismuth, an eighty-micrometer sieve was utilized to acquire a more homogeneous powder size.

To measure the photoluminescence of the powders, a 405-nanometer laser was first used to excite the electrons. A PDMS layer of 6.26-millimeter thickness was placed on top of the powders. The function generator was set with a square wave at a 1.5 millisecond pulse. A photomultiplier was placed to detect the luminescence of the powders. A variety of lenses were used in order to determine the temperature dependent bandwidths. The stage was connected to the temperature controller in order to vary the temperature of the powders. The powders were given five minutes to rise to the desired temperature if the temperature increment was five degrees Celsius, and eight minutes if the increment was ten degrees Celsius. The photomultiplier was then output into an oscilloscope in order to see the decay of the photoluminescence. From there, the decay rate of each of the powders at elevated temperatures were determined.

The exact same procedures were utilized to determine the decay rates of the composites. One exception is for the Zinc Sulfide UV (Green) for both the powder and composite form: a pulse length of 2.0 millisecond pulse length was utilized since it took the powders longer to reach a steady excited state. For the phosphors with the decay rates that were analyzable at room temperature, they were tested from room temperature to 200 degrees Celsius in five- or ten-degree increments.

Results

For the majority of the phosphor powders, they were successfully encapsulated into PDMS. Table 1 displays which materials were cured successfully and which were not. Other methods of curing were also tested on the Lanthanum/Gadolinium samples such as curing at 200 degrees Celsius and using UV curing. Unfortunately, neither of these methods worked. The composites remained liquid, indicating the powders were interfering with the curing chemical reaction. For the successfully cured samples, the physical characteristics of each composite varied and should be investigated at a later date.

Phosphor Powder	Successful Curing?
1: Yttrium	Yes
2: Lanthanum	Yes
3: Lanthanum/Gadolinium	No
4: 620 Nitrate	Yes
5: 630 Nitrate	Yes
6: Zinc Sulfide UV (Green)	Yes
7: Bismuth	No
8: Zinc Sulfide UV (Blue)	Yes
B: Europium Tetrakis #1	Yes
C: Europium Tetrakis #2	Yes
D: Europium Tetrakis #3	Yes
F: Magnesium Tetrakis #1	Yes
G: Magnesium Tetrakis #2	Yes

Table 1. Curing Composites

Next, the optimal wavelengths for each phosphor were observed and recorded. Table 2 shows which wavelength lens should be used for each phosphor.

Phosphor Powder	Optimal Wavelength
1: Yttrium	460 nm
2: Lanthanum	460 nm
3: Lanthanum/Gadolinium	460 nm
4: 620 Nitrate	650 nm
5: 630 Nitrate	650 nm
6: Zinc Sulfide UV (Green)	540 nm
7: Bismuth	540 nm
8: Zinc Sulfide UV (Blue)	460 nm
B: Europium Tetrakis #1	650 nm
C: Europium Tetrakis #2	650 nm
D: Europium Tetrakis #3	650 nm
F: Magnesium Tetrakis #1	460 nm
G: Magnesium Tetrakis #2	460 nm

Table 2. Powders and Composites Optimal Wavelength

The optimal wavelength was determined by selecting the most prevalent and analyzable decay rates. An example can be seen in Figure 1.

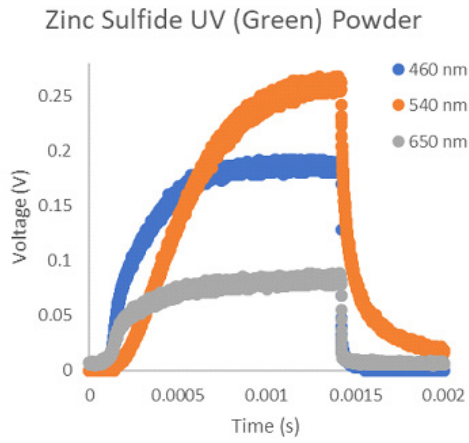

Figure 1. The decay rate for Zinc Sulfide UV (Green) Powder is shown above. It displays the amount of voltage against time.

From the figure above, it can be determined that the optimal wavelength for Zinc Sulfide UV (Green) is 540 nm due to the longer decay time. In addition, both the powders and the phosphors had the same optimal wavelengths. This is evident in Figure 2.

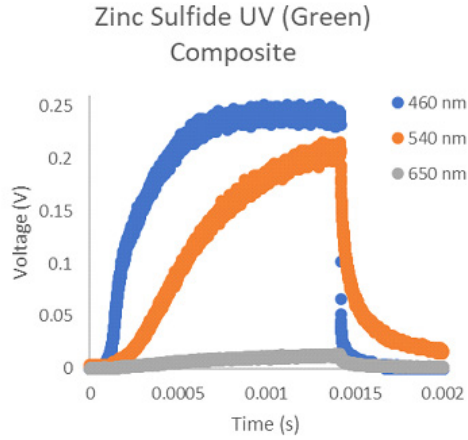

Figure 2. The decay rate for Zinc Sulfide UV (Green) Composite. It displays the amount of voltage against time.

Lastly, when setting up the decay behavior of the photo luminescence against the natural logarithm of the temperature, the same linear lines were evident between the powders and their respective composites. The standard data is seen in Figure 3.

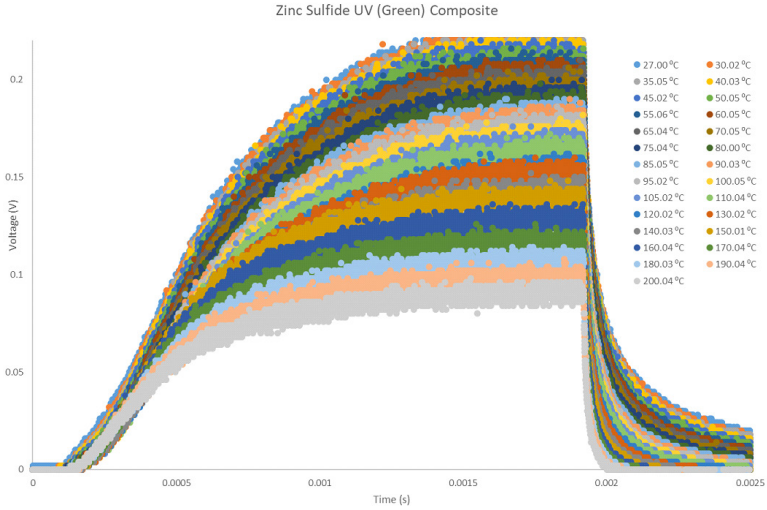

Figure 3. The decay rates for various elevated temperatures for Zinc Sulfide UV (Green) Composite using a 540-nanometer wavelength lens. A look at the data shows that higher temperatures are correlated with shorted decay rates.

When plotted in a log plot, the linearity of the correlation can be seen.

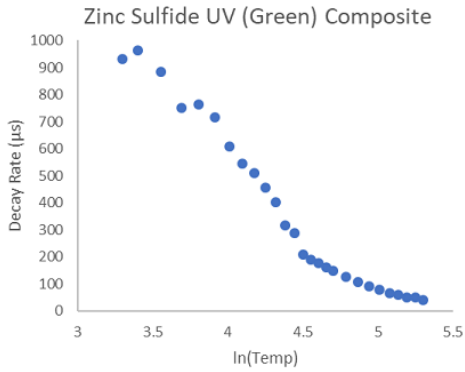

Figure 4. The decay rates plotted against the natural logarithm of the temperature. This is for the Zinc Sulfide UV (Green) Composite.

There also appears to be two separate linear sections in the correlation, which should be investigated at a further date. The Zinc powder and the Europium Tetrakis samples were also measured and a linear correlation is also seen. All of the other composites' decay rates were too short to measure at elevated temperatures.

Conclusion

The majority of the powders were successfully encapsulated by PDMS into composite form. In addition, the encapsulation process does not affect the readings on the decay rates. As a result, the benefits of the phosphor powders are retained while the weaknesses are eliminated. With the optimal wavelengths analyzed, the correct lens can be chosen to observe each phosphor powder.

As the composites have been analyzed, the phosphor samples can be used as a temperature sensing apparatus. The composites can be utilized where traditional methods of thermometry are lacking. Since taking readings is non-contact, accurate, and quick, this form of thermometry has many advantages, and one of the best utilizations is in space applications where conditions make temperature readings difficult.

In the future, it would be desirable to see lower temperatures explored. In addition, other characteristic tests of the composites would be useful, as while the effects of PDMS on the powders have been observed to an extent, the effects on the characteristics of PDMS has not. The potential of temperature flux has not been analyzed. It can be achieved by layering the different composites on top of each other and reading the different decay rates. Finally, encapsulation of the powders in other materials such as aerogels would be useful for future study.

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