

# USE OF PHOSPHOR COATINGS FOR HIGH TEMPERATURE AEROSPACE APPLICATIONS

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## ABSTRACT

Phosphor thermometry has been used for many years for non-contact temperature measurements. Aerospace systems are particularly prone to adverse high temperature environments, including large blackbody background, vibration, rotation, fire/flame, pressure, or noise. These environments often restrict the use of more common thermocouples or infrared thermometric techniques. Temperature measurements inside jet turbines, rocket engines, or similar devices are especially amenable to fluorescence techniques. Often the phosphor powders are suspended in binders and applied like paint or applied as high temperature sprays. Thin coatings that are less than 50  $\mu\text{m}$  thick are used on the surfaces of interest. These coatings will quickly assume the same temperature as the surface to which they are applied. The temperature dependence of phosphors is a function of the base matrix atoms and a small quantity of added activator or “dopant” ions. Often for high temperature applications, the selected materials are refractory and include rare earth ions. Phosphors like  $\text{Y}_3\text{Al}_5\text{O}_{12}$  (YAG) doped with Eu, Dy, or Tm,  $\text{Y}_2\text{O}_3$  doped with Eu, or similar rare earth compounds, will survive high temperatures and can be configured to emit light that changes rapidly in lifetime and intensity. Recently, a YAG:Cr phosphor paint emitted fluorescence during short duration tests in a high Mach number hydrogen flame at 2,200  $^\circ\text{C}$ . One of the biggest challenges is to locate a binder material that can withstand tremendous variations in temperature in an adverse aerospace environment. This presentation will give research results applicable to the use of phosphors for aerospace thermometry. Emphasis will be placed on the selection of phosphor and binder combinations that can withstand high temperatures. Evidence for light pumping for  $\text{Y}_2\text{O}_3\text{:Cr/YAG:Ce}$  mixture and preliminary triboluminescence results for  $\text{ZnS:Mn}$  will also be presented. These results are the first step towards the development of a smart material damage sensor.

## INTRODUCTION

Phosphors are fine powders that are doped with trace elements that emit visible light when suitably excited. Many of them are ceramics that can withstand high temperatures. The fluorescence characteristics change with temperature. A phosphor coating can indicate the temperature of the surface. There are a wide variety of ceramic phosphors that survive hazardous physical and chemical environments, are insoluble in water, durable, and easy to apply. Some phosphors have no trouble surviving and functioning in high temperatures such as those present during combustion.

The basic principle of thermal phosphors is well established, and researchers at Oak Ridge National Laboratory (ORNL) have demonstrated several useful applications<sup>1-7</sup>. This thermometry method relies on measuring the rate of decay of the fluorescent response of an inorganic phosphor as a function of temperature. Having calibrated the phosphor over the desired temperature range, a small surface deposit of phosphor is excited with a pulsed laser and the fluorescent decay is measured (typically in less than 1 ms) to calculate the temperature of the substrate. In many instances, like for a continuous steel galvanneal process, a simple puff of powder onto the surface provides an adequate fluorescent signal.

Often temperature measurements are made using thermocouples or optical pyrometry. However, in situations where rapid motion or reciprocating equipment is present at high temperatures, it is best to use other techniques. For certain phosphor paints, the prompt fluorescence decay time ( $\tau$ ) varies as a function of temperature and is defined by

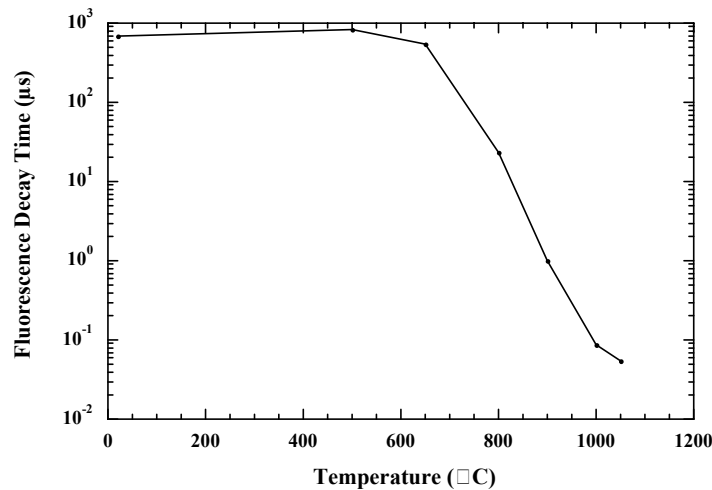
$$I = I_0 \exp\left\{-\frac{t}{\tau}\right\}, \quad (1)$$

where:

$I$  = Fluorescence light intensity (-),

$I_0$  = Initial fluorescence intensity (-),  
 $t$  = Time since cessation of excitation (s), and  
 $\tau$  = Prompt fluorescence decay time (s).

The units for the fluorescence light intensity are arbitrary. The time needed to reduce the light intensity to  $e^{-1}$  (36.8%) of its original value is defined as the prompt fluorescence decay time. An example of this quantity for  $Y_2O_3:Eu$  as a function of temperature is shown in Figure 1. The decay time decreases by almost four orders of magnitude when the temperature is raised from 600 °C to 1,000 °C.



**Figure 1. Prompt fluorescence decay time for  $Y_2O_3:Eu$  as a function of temperature**

Triboluminescence (TL) is light produced while striking or rubbing two pieces of a material together. Sir Francis Bacon first studied it 400 years ago and is basically defined as light from friction, since the term comes from the Greek *tribein*, meaning "to rub," and the Latin prefix *lumin*, meaning "light". A good history and review of research to 1977 is Walton<sup>8</sup>. Since then, much work has been done on characterization of materials, mechanisms, and possible applications. Of particular note are the work of Chandra and Zink<sup>9</sup> and Sweeting<sup>10</sup> on characterization and mechanisms; and of Sage, Bourhill, et al.<sup>11</sup> and Xu, Akiyama, et al.<sup>12</sup> on the use of TL as a stress and damage sensor.

This paper will give an overview into research to find binder and phosphor combinations that can emit light and remain mechanically viable at high temperatures. Emphasis will be placed on developing techniques for the application and the pre-treatment of candidate binder and phosphor combinations. Preliminary triboluminescence results for  $ZnS:Mn$  will also be presented. Both of these efforts were completed at the Oak Ridge National

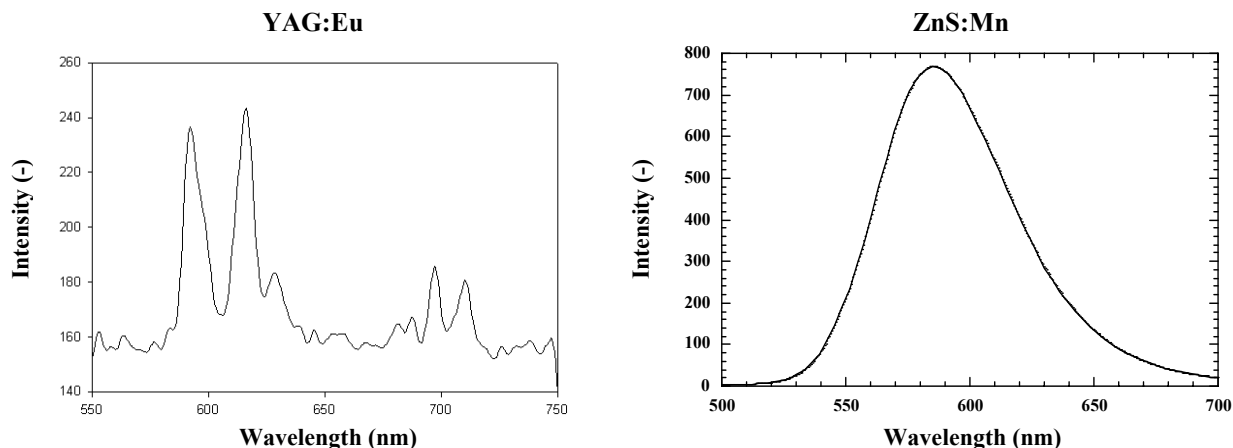
Laboratory (ORNL) National Transportation Research Center (NTRC) in Tennessee.

### **BINDERS**

Each of these three tested materials is designed for use in high temperature applications and can be purchased from Zyp Coatings of Oak Ridge, TN<sup>13</sup>. Several other binders were tried during this research and were found to fail above 1,200 °C.

### **HPC**

When cured, the HPC binder coating is not reactive and is composed of magnesium aluminum silicate. The maximum recommended use temperature for this formulation is 1,500 °C. It is water based with a shelf life of 12 months. The sprayed coating dries at room temperature in 15 to 20 minutes after application. HPC is a softer coating compared to other Zyp coatings. HPC is water-soluble after drying and can be purchased from Zyp for about \$100 per quart.



**Figure 2. Sample YAG:Eu and ZnS:Mn emission spectra**

### **LK**

When cured, the LK binder coating is not reactive and is composed 75% SiO<sub>2</sub>, 20% K<sub>2</sub>O, and 5% Li<sub>2</sub>O. The maximum recommended use temperature for this formulation is 1,100 °C. Engineers at Zyp indicate that LK will survive higher temperatures if additional fillers are added to the mix. It is water based with a shelf life of 12 months. The sprayed coating dries at room temperature in 15 to 20 minutes after application. LK is probably the hardest coating of all the tested high temperature Zyp coatings. LK is water-soluble after drying and can be purchased from Zyp for about \$100 per quart. Often LK and HPC are used in combination. By itself, HPC is thick and difficult to spray. Conversely, LK by itself cannot be used safely above 1,100 °C. The selected combination is easy to spray and provides consistent results to more than 1,100 °C.

### **ZAP**

When cured, the ZAP binder coating is composed of 53.5% P<sub>2</sub>O<sub>5</sub> and 46.5% Al<sub>2</sub>O<sub>3</sub>. The maximum recommended use temperature for this formulation is 1,800 °C. It is water and alcohol based with a shelf life of 12 months. The sprayed coating dries at room temperature in 15 to 20 minutes after application. ZAP is a hard coating that can be used in any atmosphere. Heating to 900 °C for 60 minutes cures the ZAP binder without generating organic ash.

### **PHOSPHOR POWDERS**

A selection of rare earth compounds are used for the phosphor powders in this research:

- Yttrium oxide (Y<sub>2</sub>O<sub>3</sub>) doped with chromium (Cr) and europium (Eu),
- Yttrium aluminum garnet (Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> - YAG) doped with cerium (Ce), dysprosium (Dy), europium (Eu), and terbium (Tb), and
- Zinc sulfide doped with manganese (ZnS:Mn).

The dopant concentrations are typically a few percent by mass and have grain sizes of about 10 μm in extent<sup>14</sup>. These phosphors emit copious fluorescence at high temperatures and can be excited by typical laser wavelengths. The sample emission spectra for YAG:Eu and ZnS:Mn are shown in Figure 2. The YAG:Eu phosphor glows bright orange when irradiated with a standard ultraviolet light. Emission peaks at wavelengths of 595, 611, 631, 695, and 710 nm are clearly visible in the YAG:Eu spectrum.

When excited, ZnS:Mn emits bright yellow luminescence. The broad emission peak is centered at 590 nm and has a full width at half maximum (FWHM) of about 125 nm. Both spectra shown in Figure 2 were collected at ORNL using a LS50-B Perkin Elmer Luminescence Spectrometer.

### **PAINT MIXTURES**

A selected binder and phosphor powder are mixed together to create a strong and durable paint. A maximum of 50% phosphor powder by volume was added to each mixture. Paint mixtures were sprayed on a clean 25 mm x 25 mm (1 inch x 1 inch) ceramic card and cured based on specific binder instructions. Paint uniformity was checked using

black light inspection during application. All samples were inspected after the cure cycle before being exposed to the high temperature environment for 60 minutes. After curing and before thermal cycling, the LS50-B was used to determine the baseline fluorescence spectrum for each paint sample. After each thermal cycle the paint sample was re-analyzed using the LS50-B.

## RESULTS

### Temperature Effects

Results from these measurements can be found in Table 1. Each paint combination was exposed to the high temperature listed in Table 1 for 60 minutes and then slowly cooled. The heating rate was kept small in order to minimize effects due to the difference in expansion coefficient between the paint and the ceramic substrate.

Results shown in Table 1 show that most of the HPC/LK samples survived heating to 1,400 °C. Most of the paints were removed from the surface of the ceramic at 1,500 °C. This data also shows that paint consisting of a 100% ZAP binder and 30% YAG:Dy powder by volume is intact and emits fluorescence after heating to 1,500°C. This paint surface did look

bumpy or mottled after heating. The other ZAP paints were also intact, but with reduced fluorescence. In fact, the 100% ZAP and 30% by volume YAG:Dy paint was also found to emit fluorescence after heating to 1,600 °C as shown in Figure 3. A short wavelength ultraviolet light energized the YAG:Dy phosphor exposed to the three listed temperatures. ZAP binder and YAG:Ce phosphor is the most successful paint designed to withstand temperatures as large as 1,600 °C. Figure 4 shows the ultraviolet-induced fluorescence can be detected from a 100% ZAP and 50% Y<sub>2</sub>O<sub>3</sub>:Eu paint from 1,200 to 1,500 °C. Recently, a LK/HPC binder mixture with a YAG:Cr phosphor emitted fluorescence during three test runs between Mach 2.5-3.0 in a hydrogen flame at 2,200 °C<sup>15</sup>.

In summary, the HPC/LK samples with all phosphor loadings seemed to withstand heating to 1,400 °C. However, most of the coatings for HPC/LK samples were gone at 1,500 °C. ZAP appeared to be the most successful high temperature binder. The ZAP and YAG:Dy paint was able to withstand a temperature of 1,600 °C. The ZAP and Y<sub>2</sub>O<sub>3</sub>:Eu, YAG:Eu, and YAG:Tm phosphors withstood a temperature of 1,500 °C, with reduced visible fluorescence. The ZAP and YAG:Tm paint was difficult to apply.

**Table 1. Binder and phosphor paint results**

Binder (Composition)	Paint		Emission Lines (nm)	Phosphor Emission?				Preparation Comments
	Fraction (Vol. %)	Phosphor		1,200 °C	1,300 °C	1,400 °C	1,500 °C	
100% HPC	20%	Y <sub>2</sub> O <sub>3</sub> :Eu	611			Yes	Yes	Paint mostly gone after heating to 1,500 °C
100% HPC	10%	Y <sub>2</sub> O <sub>3</sub> :Eu	611			Yes	Yes	Paint mostly gone after heating to 1,500 °C
75% HPC 25% LK	20%	Y <sub>2</sub> O <sub>3</sub> :Eu	611				No	All of paint gone after heating to 1,500 °C.
75% HPC 25% LK	10%	Y <sub>2</sub> O <sub>3</sub> :Eu	611				Yes	Most of coating gone after heating to 1,500 °C.
50% HPC 50% LK	20%	Y <sub>2</sub> O <sub>3</sub> :Eu	611			Yes	Yes	Paint mostly gone after heating to 1,500 °C
50% HPC 50% LK	10%	Y <sub>2</sub> O <sub>3</sub> :Eu	611			Yes	No	No 611 nm peak after heating to 1,500 °C.
100% ZAP	50%	Y <sub>2</sub> O <sub>3</sub> :Eu	611	Yes	Yes	Yes	Yes	Paint still intact with diminished fluorescence after heating to 1,500 °C.
100% ZAP	30%	YAG:Dy	585	Yes	Yes	Yes	Yes	Paint still intact after heating to 1,500 °C.
100% ZAP	30%	YAG:Tm	420 480				Yes	Coating intact but poor fluorescence after heating to 1,500 °C.
100% ZAP	30%	YAG:Eu	595 611				Yes	Coating intact but poor fluorescence after heating to 1,500 °C.

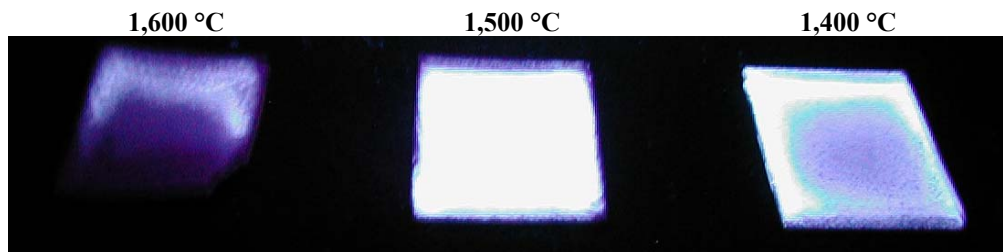


Figure 3. Ultraviolet stimulated fluorescence from a 100% ZAP and 30% YAG:Dy paint (by volume) at 1,600, 1,500, and 1,400 °C

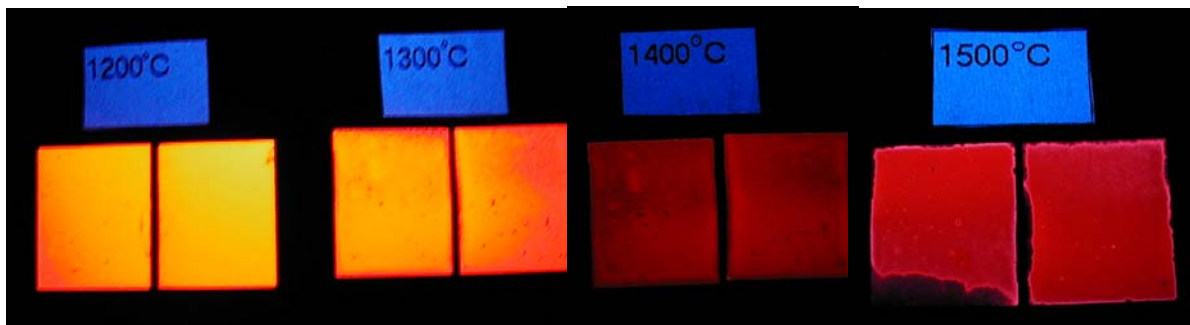


Figure 4. Ultraviolet stimulated fluorescence from a 100% ZAP and 50% Y<sub>2</sub>O<sub>3</sub>:Eu paint (by volume) at 1,200, 1,300, 1,400, and 1,500 °C

### Light Pumping

Fluorescence intensity is an important consideration when deciding on which phosphor powder to use for a selected thermometry application. Measuring the fluorescence decay time and comparing it to a calibration curve like is shown for Y<sub>2</sub>O<sub>3</sub>:Eu in Figure 1, gives the temperature of the painted surface. Often other processes such as infrared emission, smoke, steam, or other high temperature phenomena make it difficult to measure a small quantity of emitted fluorescence. The selected phosphor powder needs to emit sufficient fluorescence when irradiated by an outside source to overcome these difficulties.

An idea that could potentially increase the amount of fluorescence intensity is known as light pumping. Here a mixture of phosphors is used on a high temperature surface. Let's assume that two phosphors A and B each have good high temperature properties. Phosphor A emits weakly in the wavelength band of interest. Phosphor B emits most of its fluorescence in shorter wavelengths (higher energies) than A. Light pumping would result if the light emitted by the A and B mixture would be greater than the separate phosphors. The key to this idea is the fact that B would have higher energy electrons to de-excite. In the mixture, the electrons in some of the higher excited states of B could transition

to states in A that generate the fluorescence of interest and would "light pump" the mixture.

To test this idea, Y<sub>2</sub>O<sub>3</sub>:Cr and YAG:Ce were selected for the A and B phosphors respectively. Y<sub>2</sub>O<sub>3</sub>:Cr is a dim phosphor with a small peak around 690 nm. When excited, YAG:Ce emits bright green-white fluorescence. The broad emission peak of YAG:Ce is centered at 525 nm and has a FWHM of about 100 nm. However, YAG:Ce does emit a small amount of fluorescence around 690 nm, which is the region of interest for Y<sub>2</sub>O<sub>3</sub>:Cr. Clearly, excited electrons in YAG:Ce have higher energies than those for Y<sub>2</sub>O<sub>3</sub>:Cr.

The sample description for the light-pumping test is shown in Table 2. Approximately 10 mL of each powder formulation was well mixed and placed in separate plastic test tubes for use.

Table 2. Light pumping sample compositions

Sample Number	Composition (By Volume)
0	0 % YAG:Ce & 100 % Y <sub>2</sub> O <sub>3</sub> :Cr
1	25 % YAG:Ce & 75 % Y <sub>2</sub> O <sub>3</sub> :Cr
2	50 % YAG:Ce & 50 % Y <sub>2</sub> O <sub>3</sub> :Cr
3	75 % YAG:Ce & 25 % Y <sub>2</sub> O <sub>3</sub> :Cr
4	100 % YAG:Ce & 0 % Y <sub>2</sub> O <sub>3</sub> :Cr

To measure fluorescence intensity, a 2 mL portion of sample was removed from each vial and

placed on a metal plate. The powder was gently pushed down until it was flat with respect to height. An ultraviolet laser was used to excite the mixture. To monitor light intensity, a photomultiplier tube (PMT) was positioned approximately 2 inches (50 mm) above and slightly to the right of the powder. A red filter with a pass centroid of 685 nm was used to remove other wavelengths of light from the PMT. A Tektronix TDS 3052 oscilloscope captures the maximum light intensity in volts in single sequence mode. The room light was turned off to further reduce background noise in the PMT. The gain on the PMT was adjusted using a screw potentiometer until it was about half the total range.

Table 3 shows the results for this preliminary light pumping experiment. Notice that sample 3 (75% YAG:Ce) is slightly brighter than samples 2 and 4 (50% and 100% YAG:Ce respectively). The increased brightness potential for sample 3 is barely statistically significant. It does appear that light pumping is taking place for the  $Y_2O_3:Cr$  and YAG:Ce system. However the results are clouded by the fact that YAG:Ce emits fluorescence around 685 nm, which is significant for  $Y_2O_3:Cr$ . Additional research using a B phosphor that does not emit in the range of A will be completed in the future to answer this question.

**Table 3. Light pumping results**

Sample Number	Volume Percent YAG:Ce	Intensity Amplitude (Volts)	Ratio [YAG:Ce (0%) = 1]
0	0	0.175	1.00
1	25	37.60	214.84
2	50	40.53	231.62
3	75	41.17	235.27
4	100	40.71	232.79

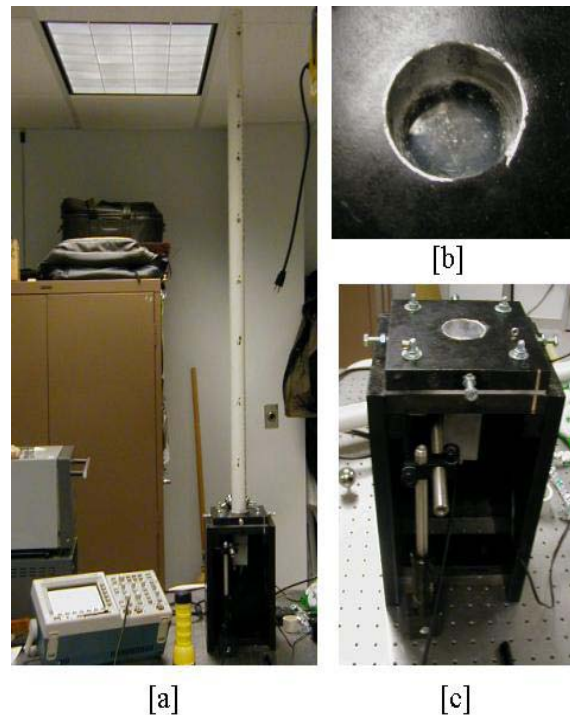
### Triboluminescence

The purpose of this preliminary research was to quantify the relationship between TL intensity and impact velocity for ZnS:Mn. This TL material was purchased commercially and was selected because it emits copious amounts of light when struck with a projectile.

A specially designed drop tower was constructed for less than \$20.00 and is shown in Figure 5. It was made from 0.75 inch (19 mm) medium density fiberboard, 0.25 inch (6 mm) plexiglas plate, and a common 1.25 inch (32 mm) piece of PVC pipe. The pipe has holes drilled in measured increments. These holes are used to place a pin that holds a 1.25 inch (32 mm) diameter steel ball (0.13 kg) bearing in place, and serves as the release mechanism.

The sample powder is placed on the plexiglas plate as shown in Figure 5b. The material is arranged so that it is aligned about the center of the tube. After several drop tests, the plexiglas begins to show pits from the impact of the drop mass. These pits are used to align the sample so that the majority of the material is in the impact zone.

The ZnS:Mn powder can be widely displaced by the impact. After each test, the drop tube is removed, the drop mass is cleaned, and the powder redistributed near the center of the target area. This redistribution is accomplished by placing a small piece of PVC in the tower base and moving the powder into an even layer. After each test, a small amount of the powder is removed when the ball bearing is cleaned. Apparently, this change in sample mass does not appear to affect the intensity or repeatability of the measurements.

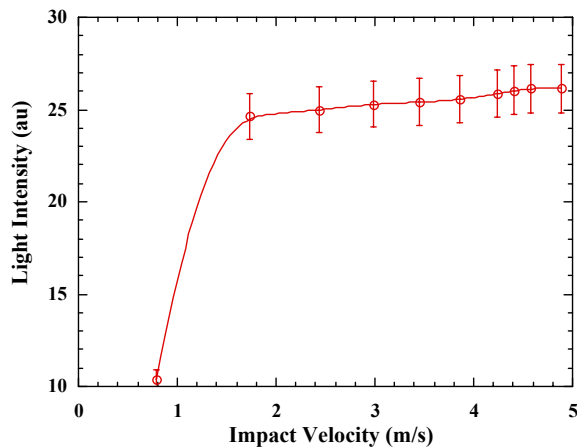


**Figure 5. Experimental arrangement for the drop tower: [a] Drop tower with oscilloscope, [b] sample tray formed by tube support and plexiglas bottom, and [c] drop tower base showing the mounting of the photomultiplier tube (PMT)**

To monitor TL intensity, a photomultiplier tube (PMT) was placed approximately 0.75 inches (9 mm) inch under the plexiglas plate as shown in Figure 5c. In order to reduce effects due to stray light, a 589 nm filter is positioned in front of the PMT. Remember the maximum emission from ZnS:Mn is about 590 nm. The TDS 3052 oscilloscope captures the luminescence data in single sequence mode. The

room light was turned off to further reduce background light noise in the PMT. The gain on the PMT was adjusted using a screw potentiometer until it was about half the total range.

Figure 6 plots the variation in light emission characterized by the output potential from the PMT (volts) versus the drop velocity (m/s). The impact velocity is defined based on Newton's equations for a free falling object. These equations assume that air resistance is negligible and the ball does not make contact with the sides of the drop tube. The approximate measurement uncertainty is about  $\pm 5\%$ , as shown by the error bars plotted in Figure 6.



**Figure 6. Plot of the PMT output potential from a ZnS:Mn powder as a function of impact velocity**

The intensity of the triboluminescent response appears to be a function of impact velocity with two regions of interest as shown in Figure 6. This data is totally consistent with other measurements completed by the authors<sup>16</sup>. The first region is in the threshold velocity range below 2 m/s. The production of TL light appears to have a threshold at about 0.8 m/s. Above the threshold, the projectile has sufficient velocity (or energy) to break ZnS:Mn crystals and to produce TL light. The TL luminescence increases rapidly until about 2 m/s. The second region starts at about 2 m/s and appears to be more like a saturation state, where the slope is much more shallow and would indicate less sensitivity to impact velocity. Understanding these relationships will be very beneficial towards the design of smart materials to detect damage caused by impact of foreign objects. Once an impact is detected the severity can be determined without the need for immediate inspection. This impact data could also be saved in a database and used to determine a maintenance and inspection procedures. It was found that the reduction in TL material encountered in testing does not affect the repeatability of the measurements.

## CONCLUSIONS

Phosphors are powders that are doped with trace elements that give off visible light when suitably excited. Many of them are ceramics and can withstand extremely high temperatures. The fluorescence characteristics change with temperature. A phosphor coating can indicate the temperature of the surface. There are a wide variety of ceramic phosphors that survive hazardous physical and chemical environments, are insoluble in water, durable, and easy to apply. Some phosphors have no trouble surviving and functioning in high temperatures such as those present during combustion.

Results indicate that a 100% ZAP binder and 30% YAG:Dy (by volume) phosphor paint will emit useful fluorescence to 1,600 °C. Other phosphors using the ZAP binder were found to be viable to 1,500 °C with reduced amounts of fluorescence emission. The Y<sub>2</sub>O<sub>3</sub>:Eu phosphor and HPC/LK binder paint combinations were found to stable and to emit fluorescence to 1,400 °C.

It does appear that light pumping is taking place for the Y<sub>2</sub>O<sub>3</sub>:Cr and YAG:Ce system. However the results are clouded by the fact that YAG:Ce emits fluorescence around 690 nm, which is significant for Y<sub>2</sub>O<sub>3</sub>:Cr. Additional research using will be completed in the future to resolve these questions.

The preliminary triboluminescence research shown here demonstrates the ability to systematically reproduce a response in a simple drop tower apparatus. The results from the ZnS:Mn phosphor show an ability to tract variations in the impact energy by the intensity of the response. These tests also show that repeatable results could be obtained without the volume of material being held constant. This fact leads to the conclusion that the light produced was from interaction of the mass with a small number of particles. These results become very important in the development of a smart material damage sensor. First, since the intensity of the response can be related to the impact energy, a sensor can be developed that not only detects an impact but also determines the severity of the strike. This information could be saved and used as part of a maintenance and inspection schedule. The other important fact obtained is that the phosphor can withstand multiple strikes and still produce repeatable data. This is very important since once deployed as part of the smart structure it we would be difficult to replace the expended phosphor. While many questions remain to be answered the basic premise of a triboluminescent based smart material damage sensor can be established.

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