STUDY OF RESBOND® CERAMIC BINDERS USED FOR HIGH TEMPERATURE NON-CONTACT THERMOMETRY

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ABSTRACT

Fluorescence is a good non-contact thermometry technique in hostile environments such as those found at high temperatures. Phosphors are typically rare earth-doped ceramics that emit light when excited. The intensity, rise time, decay time, and wavelength shift of this emitted light can be temperature dependent. When thermographic phosphors are applied to a surface, with an excitation source and a method to characterize the emission provided; it is possible to determine the surface temperature. One of the simpler methods to apply these coating is through the use of temperature sensitive paints (TSPs). These TSPs are created by mixing phosphor with a binder material to form a sprayable coating that can be easily and economically applied to a large area. Ideally, these phosphor paints need to survive at the limit of the existing decay time data, or 1700 °C. The survivability of phosphor paint depends on the physical characteristics of the binder. The goal of this research is to discover binders that will allow phosphor paints to survive at high temperatures. Suitable binders will allow the construction of non-contact measurement devices useful in environments that are not suited for more common thermocouple or infrared devices. For a phosphor paint to be useful at a selected temperature, it must fluoresce when excited and have a measurable decay time. In this study, the Cotronics Resbond® 791, 792, and 793 ceramic binders were evaluated to determine their suitability to serve as binders for a Y_2O_3:Eu phosphor powder. Post-thermal cycling spectral analysis was used to quantify wavelength and intensity changes in emission from ultraviolet excitation. Several of the paints utilizing their binders were able to survive at 1500 °C.

INTRODUCTION

Phosphors are fine powders that are doped with trace elements that give off visible light when suitably excited. Many of these phosphors have a ceramic base and can survive and function at high temperatures such as those present during combustion. When thee phosphor is applied as a thin coating, it quickly equilibrates to the ambient environment and can be used to measure the surface temperature.

The basic physics of thermographic phosphors is well established, and researchers at Oak Ridge National Laboratory (ORNL) have demonstrated several useful applications [ref. 1-12]. The thermometry method relies on measuring the rate of decay of the fluorescence yield as a function of temperature. Having calibrated the phosphor over the desired temperature range, a small surface deposit is excited with a pulsed laser. The resulting fluorescent decay (typically in less than 1 ms) time is measured to calculate the temperature of the substrate. In many instances, (e.g., in a continuous steel galvanneal process) a simple puff of powder onto the surface provides an adequate fluorescent signal [ref. 1-2].

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 many phosphors, the prompt fluorescence decay time (τ) varies as a function of temperature and is defined by:

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

where:

I	=	Fluorescence light intensity (arbitrary units),
I_0	=	Initial fluorescence light intensity (arbitrary units),
t	=	Time since cessation of excitation source (s), and
τ	=	Prompt fluorescence decay time (s) [ref. 1-2].

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 several thermographic phosphors is shown in Figure 1. Notice the fluorescence decay time decreases by four orders of magnitude when the temperature increases from 600 to 1100 °C.

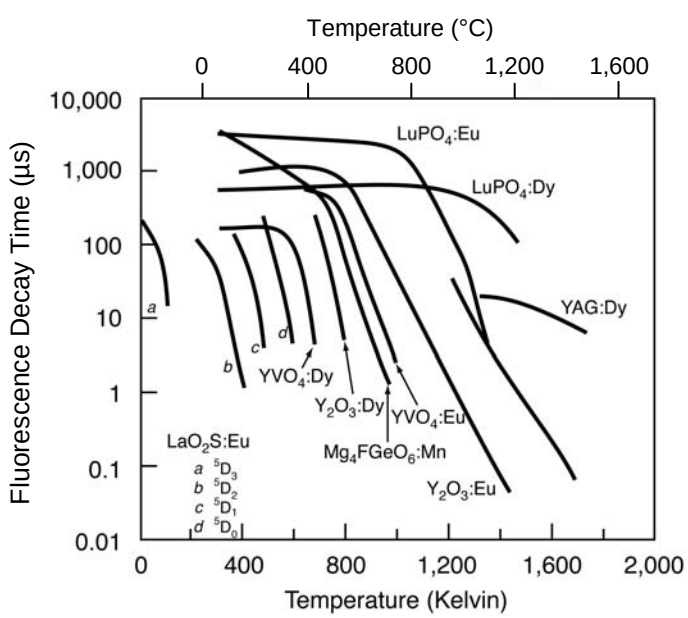

Figure 1. Prompt fluorescence decay time for a selection of thermographic phosphors.

This paper will give an overview into research to find binder and phosphor combinations that can emit fluorescence and remain mechanically viable at temperatures as large as 1600 °C. Emphasis will be placed on developing procedures and techniques for the application and the pre-treatment of candidate binder and phosphor combinations.

HISTORY

In May 2001, a research program was initiated to evaluate TSPs that could be used in high temperature thermometry applications. This three-year program was funded by the NASA John H. Glenn Research Center in Cleveland, Ohio. A research team lead by ORNL was assembled to locate binder and phosphor combinations that can emit fluorescence and maintain structural integrity at high temperatures. The ultimate goal for this research was to locate TSPs that could be used to measure the heat flux and temperature present inside a common turbine or rocket engine. The phosphors in question are typically rare earth compounds that emit copious fluorescence and have grain sizes of less than 10 µm. Binders used for this purpose must: 1) Be easy to apply with an airbrush, 2) Set to a temperature resistant inorganic finish, and 3) Have minimal reaction to the phosphor material.

During the first year of the program, research was completed to determine if heat flux could be measured using a phosphor coating. In the second year, research was completed to find phosphor and binder combinations that can emit light and remain mechanically viable at high temperatures. Results from these measurements can be found in Table I. Each paint combination was exposed to the high temperature listed in Table I for one hour 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.

Table I. Zyp Coatings-based thermally sensitive paint survivability results.

Binder	Paint		Emission (nm)	Phosphor Emission At Given Cycling Temperature?					Comments
	Fraction (Vol. %)	Phosphor		1200 °C	1300 °C	1400 °C	1500 °C	1600 °C	
HPC	20%	Y_2O_3:Eu	611			Yes	Yes		Paint mostly gone after heating to 1500 °C
HPC	10%	Y_2O_3:Eu	611			Yes	Yes		Paint mostly gone after heating to 1500 °C
75% HPC 25% LK	20%	Y_2O_3:Eu	611				No		All of paint gone after heating to 1500 °C.
75% HPC 25% LK	10%	Y_2O_3:Eu	611				Yes		Most of coating gone after heating to 1500 °C.
50% HPC 50% LK	20%	Y_2O_3:Eu	611			Yes	Yes		Paint mostly gone after heating to 1500 °C
50% HPC 50% LK	10%	Y_2O_3:Eu	611			Yes	No		No 611 nm peak after heating to 1500 °C.
ZAP	50%	Y_2O_3:Eu	611	Yes	Yes	Yes	Yes		Paint still intact with diminished fluorescence after heating to 1500 °C.
ZAP	30%	YAG:Dy	585	Yes	Yes	Yes	Yes	Yes	Fluorescence still present after heating to 1600 °C.
ZAP	30%	YAG:Tm	420 480				Yes		Coating intact but poor fluorescence after heating to 1500 °C.
ZAP	30%	YAG:Eu	595 611				Yes		Coating intact but poor fluorescence after heating to 1500 °C.

Three binders, HPC®, LK®, and ZAP®, from Zyp Coatings, Incorporated of Oak Ridge, Tennessee were used in this research [ref. 9-10]. Each of these three binders was designed for use in high temperature applications and are available in the commercial market. A selection of rare earth compounds are used for the phosphor powders in this research: yttrium oxide doped with europium (Y_2O_3:Eu), and YAG doped with dysprosium (YAG:Dy), terbium (YAG:Tb), and europium (YAG:Eu). A selected binder and phosphor powder are mixed together to create a strong and durable thermally sensitive paint (TSP). Detailed information on this research can be found in references 9 and 10.

Results shown in Table I show that most of the HP/LK bound samples survived heating to 1400 °C. Most of the paints were removed from the surface of the ceramic at 1500 °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 1500°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 ZAP binder and 30% by volume YAG:Dy TSP was also found to emit fluorescence after heating to 1600 °C.

BINDERS

During the third and final year of the NASA-Glenn research, several Resbond® ceramic binders manufactured by Coltronics Corporation of Brooklyn, New York were evaluated using a powdered Y_2O_3:Eu phosphor. The main fluorescence emission from Y_2O_3:Eu occurs at a wavelength of 611 nm. Selected material and preparation properties for the tested Resbond binders are given in Table II. The Cotronics series of ceramic binders includes six different formulations. Of these, only the four varieties listed in Table II were tested. During initial research, Cotronics Resbond 795 did not adhere to the surface. Since the other two binders have similar properties, it was considered more productive to focus on the three remaining binders.

Table II. Properties for selected Cotronics Resbond® ceramic binders.

Resbond Composition	**791 Silicate Glass**	**792 Silicate Glass**	**793 Silica Oxide**	**795 Alumina Oxide**
Applications	Adhesives Coatings	Adhesives Coatings Electronics	Bonds Fibrous Materials	High-Purity Binder
pH	Mildly Basic	Mildly Basic	Mildly Basic	Mildly Acidic
Maximum Temperature (°C)	1650	1650	1760	1870
Density (g/cm^3)	1.40	1.20	1.42	1.40
Viscosity (cps)	1500	150	50	500
Cure Time (hours)	24	24	24	2
Cycling Instructions	16 hours 25 °C or 2 hours at 120 °C	16 hours 25 °C or 2 hours at 120 °C	2 hours 25 °C or 2 hours at 175 °C	2 hours 95 °C and 2 hours at 175 °C

SAMPLE PREPARATION

In order to create the TSPs, phosphor powder is mixed with ceramic binder. The compositions for paint samples used in this research are shown in Table III. Typically, the phosphor powder accounts for twenty percent of the paint mixture by volume. In some cases, ten

percent (by volume) of a second powder such as magnesium oxide (MgO_2) is added displacing the binder. This addition was an attempt to increase the thermal conductivity to reduce thermal shock, increase the survivability of the coating, and is discussed in reference 13.

Table III. Tested paint sample compositions (fractions by volume).

Resbond Binder		**Phosphor**		**Water**	**MgO_2**
Product	**Quantity**	**Compound**	**Quantity**	**Quantity**	**Quantity**
791	35%	Y_2O_3:Eu	20%	35%	10%
	40%			40%	0%
	70%			0%	10%
	80%			0%	0%
792	70%	Y_2O_3:Eu	20%	0%	10%
	80%			0%	0%
793	80%	Y_2O_3:Eu	10%	0%	10%
			20%	0%	0%

The paint preparation procedure began by measuring the powdered solids in a graduated capped vial. Individual tubes were vibrated to insure proper volume measurement. Liquids were then added in predetermined amounts. After all the components were added to the cylinder, they were mixed by vigorously shaking the capped vial. The mixture was applied to the surface of a cleaned alumina substrate using a commercial airbrush. The TSPs were allowed to cure according to the manufacturer's specifications as shown in Table II. In the case of Resbond 791, water was added to thin the mixture to make it easier to apply using the airbrush. However the undiluted Resbond 791 paints were applied using a brush, since is difficult to spray and did not give the desired uniform coating.

EXPERIMENTAL METHOD

In order to determine the survivability for a TSP, samples were thermally cycled in a Thermoline 46200 high-temperature furnace. The furnace controls were set to raise the temperature to some predetermined value. The sample was then allowed to remain at this high temperature for one hour or more, followed by cooling to ambient room conditions. To quantify the fluorescence efficiency of phosphor suspended in the TSP, a Perkin Elmer LS-50B spectrophotometer was used to measure the emission spectrum of the sample after each thermal cycle. For each TSP mixture, two samples were made. The first sample was heated through the curing cycle and used as a control. The second was thermally cycled by heating to a set temperature and allowing it to cool back to room temperature. After the sample cooled, the emission spectrum was measured. Figure 2 shows the variation in the emission spectrum for a Resbond 793 and Y_2O_3:Eu-based TSP after thermal cycling at several different temperatures. It is quite obvious that fluorescence intensity for the 611 nm emission line decreases as a function of temperature. In fact, the 611 nm fluorescence emission is reduced to the background level after thermal cycling at 1600 °C. The TSP fluorescence spectrum shown in Figure 2 contained 80% Resbond 793 and 20% Y_2O_3:Eu by volume.

QUALITATIVE RESULTS

After thermal cycling, TSP samples were analyzed under a ultraviolet light to qualitatively estimate the intensity of the remaining 611 nm fluorescence from Y_2O_3:Eu. The TSPs were held at temperature for one hour. Qualitative results for the Resbond binder and

Y_2O_3:Eu TSP samples are shown in Table IV. Notice that all of the tested TSP samples showed some fluorescence after thermal cycling to 1300 °C. Fluorescence was also observed for all but one TSP sample at 1400 °C. Only three TSPs made with Resbond 792 and 793 showed significant fluorescence at 1500 °C. Finally, fluorescence was observed at 1600 °C for one TSP containing 20% Y_2O_3:Eu and 80% Resbond 793 by volume. These results are consistent with earlier TSP research completed during the second year of the NASA-Glenn program [ref. 7-12]. As a group, it appears that TSPs made with Zyp binders were better able to withstand slightly higher temperatures compared to the ones made using any of the three tested Cotronics Resbond formulations. However, selected Zyp and Cotronics based TSPs were both able to survive to 1500 °C.

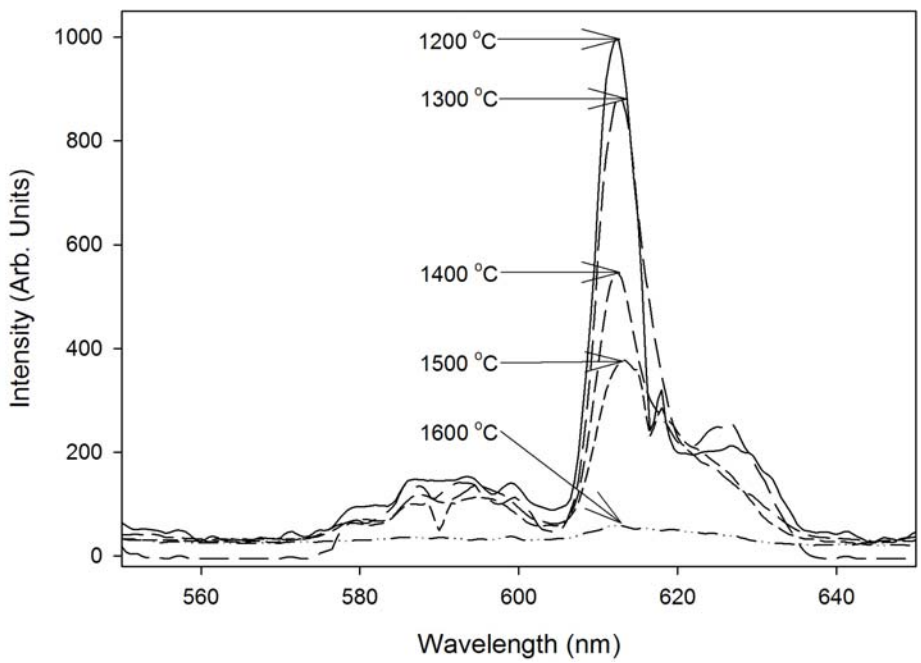

Figure 2. Resbond 793 and Y_2O_3:Eu TSP emission spectrum after thermal cycling.

Table IV. Qualitative results for the Resbond binder and Y_2O_3:Eu TSP samples.

Resbond Binder		**Remaining Paint Components**			**Phosphor Emission At Given Cycling Temperature?**				
Type	**Amount**	**Y_2O_3:Eu**	**Water**	**MgO_2**	**1200 °C**	**1300 °C**	**1400 °C**	**1500 °C**	**1600 °C**
791	35%	20%	35%	10%	Yes	Yes	Yes	No	No
	40%		40%	0%	Yes	Yes	Yes	No	No
	70%		0%	10%	Yes	Yes	Yes	No	No
	80%			0%	Yes	Yes	Yes	No	No
792	70%	20%	0%	10%	Yes	Yes	Yes	Yes	No
	80%			0%	Yes	Yes	Yes	Yes	No
793	70%	20%	0%	10%	Yes	Yes	No	No	No
	80%			0%	Yes	Yes	Yes	Yes	Yes

QUANTITATIVE RESULTS

A quantitative determination of fluorescence intensity as a function of cycling temperature is more complex. It was decided to use a ratio of 0.2 (20%) of the maximum emission intensity as

the criteria to determine the viability of fluorescence for a given TSP sample. If the fluorescence emission is small, it will be difficult to measure the decay time and obtain a corresponding surface temperature. There will come a point in intensity where a phosphor system cannot be used to measure temperature. The decision ratio of 0.2 was completely arbitrary and was based on the observation that the apparent fluorescence measurement uncertainty was about ± 10% (intensity fraction of 0.1), which was two times the measured error for the 611 nm line for Y_2O_3:Eu.

For each tested TSP, the spectral fluorescence intensity was measured using a Perkin Elmer LS-50B spectrophotometer and an ultraviolet excitation source. In each case, the fluorescence was normalized to the Y_2O_3:Eu maximum emission line at 611 nm. Samples were held at temperature for one hour unless otherwise indicated. Quantitative results for each Resbond binder can be found in the sections that follow.

Resbond 791

The evaluation of the Resbond 791 binder was complicated by its large viscosity. This required modifying the application procedure by either applying the paint with a standard bristle brush, increasing the supply pressure to the airbrush, or by thinning the binder. Since the other evaluated Resbond binders had smaller viscosities, it was felt that applying the thick paint with a brush and thinning of binder would provide a more equitable comparison. As such, the Resbond 791 binder was thinned in a one-to-one ratio with water, which aided in the application of the paint. Quantitative results for the Resbond 791 binder TSP are shown in Figure 3.

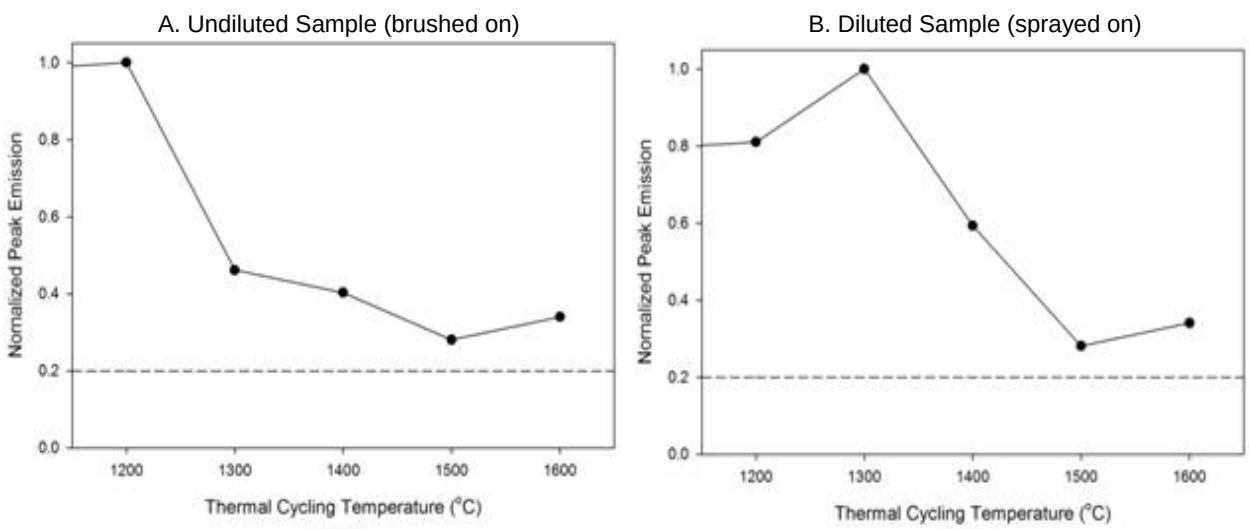

Figure 3. Normalized emission for brushed on and sprayed on Resbond 791 TSPs as a function of cycling temperature.

Tests were performed using brushed on paints. These coatings were thicker than the airbrushed coatings, but appeared to have the same maximum cycling temperature. The plot on the left side of Figure 3 (labeled A) shows the normalized fluorescence emission (611 nm) for the brushed on Resbond 791 paint versus cycling temperature. The brushed on TSP maintained a normalized peak emission greater than 0.2 to cycling temperatures up to 1600 °C. The emission intensity decreased consistently from 1300 to 1500 °C.

The plot on the right side of Figure 3 (labeled B) shows the normalized fluorescence emission (611 nm) for the airbrushed (diluted) Resbond 791 paint versus cycling temperature. The normalized emission spectra for the diluted sample was similar to that measured for the brushed on TSP. It is interesting to note that the intensity of the brushed on samples decreases slightly faster than the diluted TSPs, which could be partially caused by differences in application method. The undiluted samples were brushed on to the substrate surface that typically does not provide as even a coating. The uneven thickness could cause uneven heating and enhance flaking of the TSP.

Resbond 792

For all tested cases, the Resbond 792 TSPs falls below the 0.2 intensity criteria after the first thermal cycle (1100 °C). Visual inspection of the samples in ultraviolet light indicates that there was a significant drop in emission intensity after 1100 °C. These inspections also showed discernable fluorescence emission to 1300 °C and limited fluorescence out to 1500 °C. The dramatic drop in fluorescence intensity would tend to indicate that the Resbond 792 is not suitable for high-temperature TSP applications.

Resbond 793

Quantitative results for the Resbond 793 binder TSP are shown in Figure 4. The Resbond 793 TSP maintained a normalized peak emission greater than 0.2 to cycling temperatures up to 1500 °C. The corresponding 611 nm normalized peak emission decreased uniformly from 1200 to 1600 °C. Visual inspection of the samples indicates that the coating remains relatively bright to 1400 °C. At 1500 °C, the coating is still bright but is starting to flake away from the surface. By 1600 °C, almost all of the coating has flaked off of the surface, but the remaining portion was still discernable. Resbond 793 has a low viscosity, which makes it easy to spray, and has the highest temperature survivability of any of the tested Cotronics binders. It appears to be the best of the tested materials for use in a TSP.

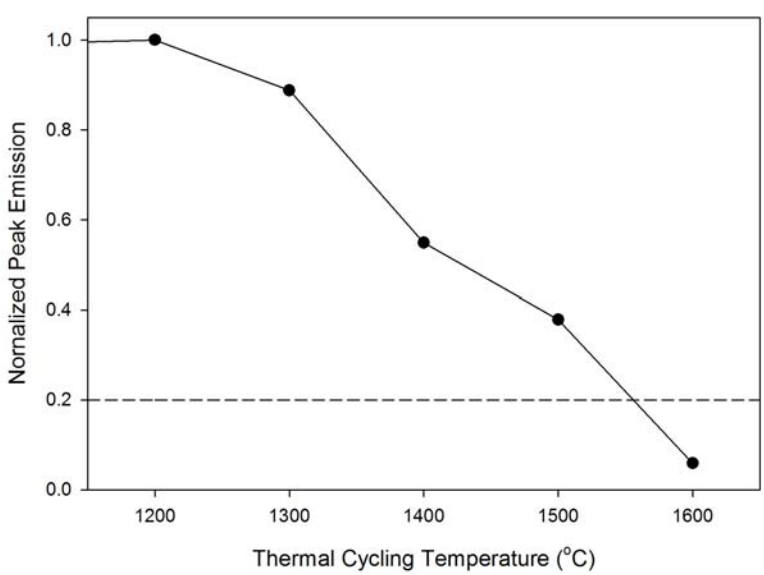

Figure 4. Normalized emission for Resbond 793 TSP as a function of cycling temperature.

Temperature Exposure Time Dependence

The exposure time was originally selected because the anticipated test length was only a few seconds. It was felt that an hour of temperature exposure would well exceed the lifetime of any experimental test series. This exposure time was selected to make this work consistent with earlier research [ref. 7-12]. To date, no research had been completed comparing the effects of exposure time at temperature to the magnitude of fluorescence emission. It was decided to complete a series of tests with a single binder to determine the relationship between temperature exposure time and normalized emission intensity for a Y_2O_3:Eu and Resbond 793 TSP.

To complete these tests, four new samples were prepared and cured as described in earlier sections. The first sample was held in reserve as the post cure control. The remaining samples were heated to 1400 °C for 1, 2, and 4 hours. The 1400 °C temperature was selected because nearly half of the room temperature fluorescence was emitted for this TSP in the previous test series. A separate sample was used in each duration test. The resulting emission spectra (ultraviolet excitation) for Y_2O_3:Eu in a Resbond 793 binder with varied thermal cycling duration are shown in Figure 5. After a one hour exposure, there is a noticeable decrease in fluorescence intensity. In fact, the decrease in intensity in the first hour of the test is surprising since the previous results showed a 40% decrease, while this test had a nearly 70% decrease from the control. After two hours, there is further decrease in intensity to greater than 80% of the control, which indicates that the coating is marginal after two hours of exposure to 1400 °C. After four hours, almost all the fluorescence is gone.

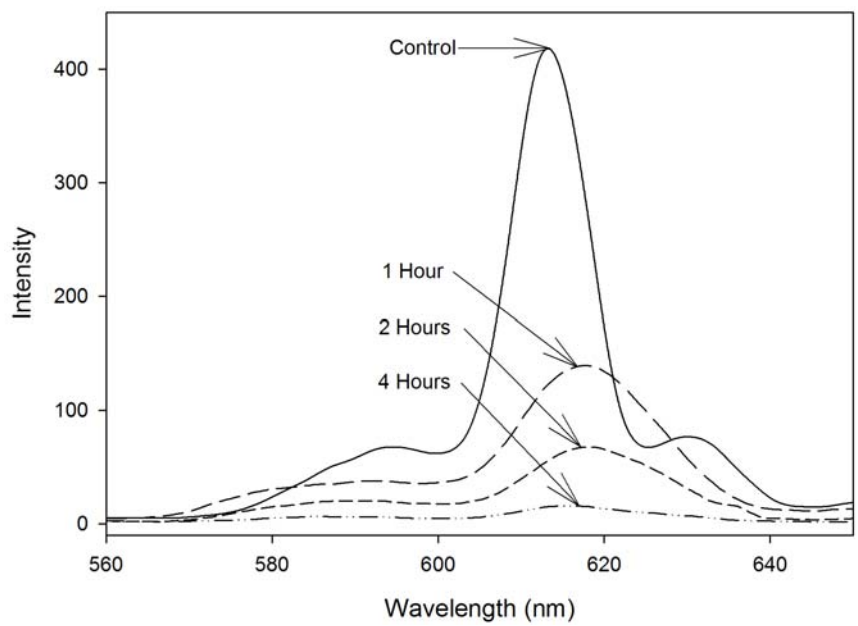

Figure 5. Changes in fluorescence intensity for a Resbond 793 and Y_2O_3:Eu TSP as a function of thermal exposure time.

CONCLUSIONS

Phosphors are fine powders that are doped with trace elements that give off visible light when suitably excited. Some phosphors can survive and function at high temperatures such as those present during combustion. This research provided basic results as to which Resbond-based TSPs can emit fluorescence and remain mechanically viable to high temperatures. All of

the tested Resbond-based Y_2O_3:Eu TSP samples showed some fluorescence after thermal cycling to 1300 °C. Fluorescence was also observed for all but one TSP sample at 1400 °C. Only three TSPs made with Resbond 792 and 793 showed significant fluorescence at 1500 °C. Fluorescence was observed at 1600 °C for one TSP containing Resbond 793. These results are consistent with similar survivability measurements completed with other TSPs. In addition, Resbond 793 appears to have the best TSP characteristics of the Cotronics binders, and it is one of the few binders that survives above 1500 °C. The temperature exposure time was found to be an important consideration for designing a TSP.

After two hours of exposure at 1400 °C, the fluorescence intensity decreases by 80%, which indicates the coating is of marginal use as a TSP. It appears that TSPs made with Zyp binders were better able to withstand slightly higher temperatures compared to the ones made using any of the three tested Cotronics Resbond formulations. However, selected Zyp and Cotronics based TSPs were both able to survive to 1500 °C. Additional research is needed to further quantify these results.

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