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ORIGINAL PAPER

Fabrication of a Bi-luminophore Temperature Sensitive Coating by Embedding Europium Thenoyltrifluoroacetonate (EuTTA) and Perylene in Polystyrene

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Abstract A new bi-luminophore system for optical sensing of temperature is described. The coating was fabricated by embedding europium thenoyltrifluoroacetonate (EuTTA) and perylene in polystyrene (PS) matrix. The luminescence emission of EuTTA was sensitive to temperature whereas perylene emission was temperature-insensitive and was used as a reference. Both luminophores were excited in the UV region of about 330 to 380 nm. The fluorescence emission of perylene and EuTTA occured at 474 nm and 615 nm respectively. The temperature sensitivities of both luminophores were influenced by (i) the type of polymer, and (ii) the concentration of luminophore in the matrix. Combining EuTTA and perylene in polystyrene matrix, a new bi-luminophore temperature sensing coating was developed. The temperature sensitivity of this coating was −1.80%/°C in the temperature range of 5 °C to 50 °C. The emission characteristics of this temperature sensitive coating displayed a fully reversible response to temperature.

Keywords Temperature sensitive coating · Fluorescence · Europium complex · Perylene · Polymer matrix

Introduction

Different optical techniques have been used for measurement of temperature as they have certain advantages like high sensitivity, non-contact and real-time monitoring over a wide range [1–5]. Some of the sensor films undergo a change in absorbance or fluorescence whereas some other sensors exhibit color changes when subjected to temperature changes. The thermochromic response of an organic dye encapsulated in sol-gel or plastic films have been used to sense temperature [1,2]. However, temperature sensing based on luminescence measurements is more suitable for use in remote environments [3–5]. For example, a temperature sensor coating popularly known as temperature sensitive paint (TSP) is used to map the surface temperature distribution of an aerodynamic body [5–10]. TSP is based on thermal quenching of the luminescence and consists of luminescent dye molecules embedded in an oxygen-impermeable binder. It has a much higher spatial resolution compared to the conventional method of thermocouples.

Different luminescent materials such as rare earth phosphors or fluorescent organic dyes are normally used for luminescence-based temperature sensing [3–8]. One of the requirements for the selection of luminophores for a luminescent temperature sensor coating is that they must be oxygen-insensitive. The luminescence intensity and lifetimes of ruthenium diimine complexes are known to be strongly temperature-dependent but they are also oxygen sensitive and hence cannot be used for temperature sensing unless an oxygen impermeable matrix is used for encapsulating them [3]. It has been reported that Rhodamine B in a silicone polymer can be used for temperature mapping in aerodynamic measurements [7]. Campbell et al. have used luminophores such as coumarin, copper octaethylporphyrin (CuOEP) with PMMA and model airplane dope as binders [8]. Europium thenoyltrifluoroacetonate (EuTTA) is another luminescent dye known to possess good temperature sensitivity and no apparent oxygen sensitivity and hence it has been used as luminophore in a commercially available

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TSP formulation with model airplane dope as binder [5,6]. EuTTA in a poly methyl methacrylate (PMMA) film has been used to measure heating of integrated circuits [11]. It has also been reported that tris-ligand chelates of europium show larger temperature dependence than tetrakis chelates [12]. Mitsuishi etal have studied the temperature sensing properties of a Langmuir-Blodgett film containing a tetrakis europium complex, namely EuTTA-phenanthroline complex and found that the decrease in luminescent intensity with temperature was non-linear from 289 to 320 K and temperature coefficient was low in this temperature range [13]. A linear response and a better temperature coefficient of −1.2%/K was obtained for this optode sensor at a higher temperature range of 320 to 370 K. This sensor may not be suitable for surface temperature measurement in wind tunnel studies where the useful temperature range is 10 to 50 °C.

EuTTA has been studied earlier as a temperature sensor in binders like dope and PMMA [5,6,11]. In our previous paper we have reported the temperature dependence of luminescence intensity and lifetimes of EuTTA in different binder polymer matrices such as polystyrene, polyurethane, PMMA, and model airplane dope and compared their photophysical characteristics [14]. The important criterion used for the selection of the polymers is that they must be oxygen impermeable. We found that the temperature sensitivity of lifetime of EuTTA-based coatings was dependent on the polymer matrix and $\Delta\tau/\Delta T$ was maximum for EuTTA in polystyrene in the temperature range of 10 to 60 °C.

Multi-luminophore paints are used to improve the accuracy of pressure mapping in wind tunnel studies [6]. Multi-luminophore coatings have also been reported for oxygen sensing [15–17]. Evans et al have reported a novel dual-luminophore oxygen sensor incorporating two luminescent platinum complexes which differ in their emission colors and oxygen sensitivities [15]. Sensor response is given by a dramatic change in emission color, from red to green at different oxygen concentrations, due to the complete or partial luminescence quenching of one or both of the sensing elements. A dual-emitting luminescent complex immobilized onto an anionic exchanger resin support has also been reported for water-dissolved oxygen sensing [16]. This aluminum-ferron complex displays two different emission bands — a fluorescence emission band which is insensitive to oxygen and an oxygen-sensitive phosphorescent emission. Simultaneous optical sensing of temperature and oxygen has also been reported by combining a temperature-sensitive europium complex and an oxygen-sensitive palladium porphyrin complex in different matrices [17].

Although there are a few publications on temperature sensitive paints in various temperature ranges, all of them use the single luminophore strategy. To our knowledge, there has been only one previous report of a bi-luminophore temperature sensor which is based on perylene and N-allyl-N-methylaniline exciplex inter conversion showing a temperature sensitivity of −1.0%/°C [18]. We report a bi-luminophore temperature sensitive coating that combines a temperature-sensitive luminophore (EuTTA) and a temperature-insensitive reference luminophore (perylene). Both probe and reference luminophores were excited by light of same wavelength. There was no overlap between the emission spectra of the probe and reference luminophores so that emissions from the two components could be completely separated using optical filters. The intensity ratio between probe and reference can eliminate the errors caused by non-uniform illumination of test surface, coating thickness and dye concentration. We found that perylene in polystyrene has negligible temperature sensitivity and possess the other properties required for the reference luminophore. Combining these two luminophores in polystyrene matrix, a new bi-luminophore temperature sensitive coating was developed.

Materials and methods

Materials

The luminophore dyes, europium thenoyltrifluoroacetonate (EuTTA) and perylene were procured from Acros Organics. Polystyrene (average molecular weight 250.000) was purchased from Acros Organics. Other binders such as model airplane dope and PMMA were procured from M/s Southfields Paints Ltd, Bangalore.

Solutions of EuTTA (1.15 mmol l^{-1}) and perylene (3.96 mmol l^{-1}) were prepared by dissolving 0.01 g dye in 10 ml toluene. The coating mixture was formed by mixing known volumes of dye solutions with 10 ml polystyrene solution in toluene (5%). The mixture was stirred well and spray coated on to the aluminum coupons with a white basecoat of epoxy paint. The coupons were allowed to cure at room temperature for about 24 h before measurements. The thickness of the coating was about 20 μm.

Methods

Fluorescence emission spectra of the sensor coatings were recorded using an experimental setup consisting of a Fiber optic spectrometer, model S2000 from M/s Ocean Optics Inc., USA and 300W Xenon arc lamp (model no. 6258 from Oriel Instruments) as excitation source. A bandpass filter (Oriel cat.no.51650) was used to transmit UV radiation from the source in the wavelength range of 300 to 380 nm and to illuminate the coatings. The coatings were

mounted in a sample chamber fabricated in our laboratory so that emission was measured by the front face technique. There was provision to measure emission intensity of the TSP coupons in the temperature range of room temperature to 60 °C. Intensity measurements of coupons in the temperature range of 5 °C to 50 °C were carried out in the PSP calibration system at NAL based on DLR technology. This PSP setup has two peltier-cooled scientific grade 12-bit CCD cameras with resolution of 1280×1024 pixels that are used as detectors to acquire images of emission from the coating. The red and blue emissions from the sample coupons at different temperatures and ambient pressure were measured using the CCD cameras equipped with suitable filters. The data analysis is done by dedicated OMS software of NAL PSP System. The emission intensities at different temperatures were normalized with the intensity at the reference temperature (T_{ref} = 20 °C). Temperature coefficient of the sensor coating is obtained from the slope of the normalized intensity versus temperature plot.

The coupons were illuminated with radiation of 335± 5 nm using a pulsed xenon excitation source. The temperature was increased from 5 °C to 50 °C in steps of 5 °C. The coupons were kept at each temperature for few minutes to establish thermal equilibrium and then the images were acquired at each temperature. Using the image processing software, average summary of the intensity across the coupon was measured.

Results and discussions

Effect of temperature on the fluorescence intensity of EuTTA in polystyrene coating

The molecular structure of EuTTA is well known and is shown elsewhere [6,12]. The luminescent lifetime of this complex is approximately 200 μs at room temperature and it shows practically no quenching by oxygen. In the case of the europium chelates like EuTTA, the excitation light is absorbed by the chelating ligands and transferred to the europium ion, Eu^{3+}. The observed emission is due to the f-f transition within the Eu^{3+} energy manifold. When the complex is excited by UV light in the wavelength range of 300 to 400 nm, energy is absorbed by the S_0 to S_1 transition of the ligand, and this energy is transferred to the Eu^{3+} levels via the triplet state [12]. The strongest emission from the Eu^{3+} ion at around 615 nm is due to the $^5D_0$ to $^7F_2$ transition. The mechanism of the temperature sensitivity of the europium beta diketonate is based on thermal quenching. The major contribution is from the thermal deactivation of $^5D_1$ and $^5D_0$ europium energy levels.

Fluorescence-based temperature sensing coating should be oxygen impermeable in order to avoid oxygen quenching of the fluorescence, which can compete with the thermal quenching. Our previous study had shown that maximum temperature coefficient was obtained for EuTTA/PS compared to EuTTA embedded in other polymers like dope, polyurethane and PMMA [14].

Sensor coupons of EuTTA in polystyrene (PS) were prepared as described in the experimental section using 2 mg dye per 0.5 g PS which is equivalent to 4.6 mmol l^{-1}. Fluorescence emission spectra of EuTTA/PS coating showed an emission peak was observed at around 615 nm. The variation of normalized emission intensity of this coating with temperature was plotted in the temperature range of 5 °C to 50 °C using NAL PSP System and is shown in Fig. 1. The plot was quite linear and temperature coefficient of the coating was obtained from the slope (−2.00%/°C). This is higher than the temperature coefficient values reported earlier for EuTTA/dope coating [5,6].

The temperature sensitivity of EuTTA/PS is due to thermal quenching of luminescence that serves as the basic principle of TSP [5,6]. Generally, the relation between luminescent intensity, I and absolute temperature, T over a certain temperature range can be written in the Arrhenius form

$$\ln\{I(T)/I(T_{ref})\} = E_{nr}/R\{1/T - 1/T_{ref}\}$$

where E_{nr} is the activation energy for non-radiative process, R is the universal gas constant and T_{ref} is the reference temperature in Kelvin. However it has been reported that some TSP systems have shown an inverse relation between normalized intensity and temperature over certain temperature ranges [6].

The effect of EuTTA concentration on the temperature coefficient of sensor coatings was studied. EuTTA/PS coatings with four different dye:polymer concentration ratio

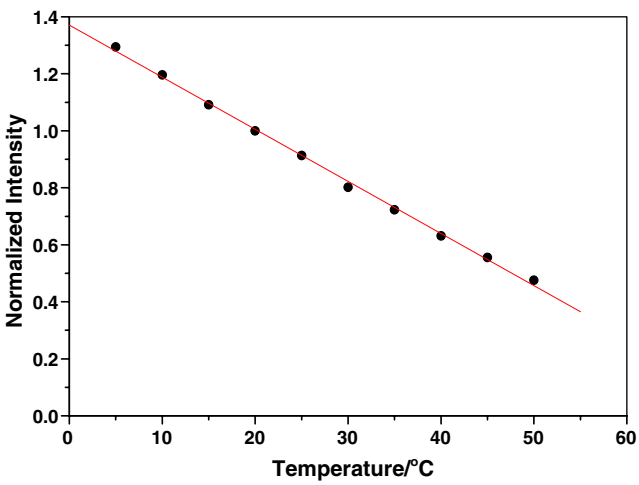

Fig. 1 Variation of normalized intensity (I(T)/I (T=20 °C)) of EuTTA/PS coating with temperature. Linear fit is shown; slope = −2.0%/°C, regression co-efficient, R^2=0.999

were prepared for this study and the results are shown in Table 1. It was found that the temperature coefficient of EuTTA/PS increased with increase in dye concentration in the polymer matrix.

Photophysical properties of perylene coatings with different polymers

Perylene is a polyaromatic hydrocarbon and its molecular structure is well known. Perylene can be excited with UV light in the wavelength range of 300 to 400 nm. Coatings of perylene in different polymers like dope, PS and PMMA were prepared as described in previous section on Materials. The concentration of perylene in the coatings was 1.0 mg in 0.5 g of polymer which is equivalent to 7.92 mmol l^{-1}. Fluorescence emission spectra of these coatings (Perylene/Dope, Perylene/PS and Perylene/PMMA) were recorded at different temperatures from room temperature to 50 °. Figure 2 shows the effect of temperature on the fluorescence emission spectra of Perylene/PS coating. The spectra showed a major monomer emission peak at about 474 nm, shouldered on both sides by minor peaks at 460 and 510 nm. It was seen that the effect of temperature on fluorescence intensity was negligibly small.

Effect of temperature on fluorescence intensity of coatings of perylene in other polymers, namely, dope, PS and PMMA was studied. It was found that the luminescent intensity of perylene in dope was much lower compared to the intensity counts of same concentration of perylene in PS and PMMA coatings. Temperature dependence of the normalized intensities of coatings of perylene in dope, PMMA and PS is shown in Fig. 3. It was found that the temperature sensitivity of perylene/PS and perylene/PMMA were minimal compared to that of perylene/dope.

The effect of perylene concentration in the polymer on the fluorescence emission spectra was studied and is shown in Fig. 4. The concentration of perylene in the coatings was varied as 1.0, 2.0 and 3.0 mg in 1.0 g of polymer (equivalent to 3.96, 7.92 and 11.89 mmol l^{-1} respectively). Figures 4a,b and c show the fluorescence spectra of perylene

Table 1 Effect of dye concentration on the temperature coefficient of the intensity of EuTTA/PS coatings

EuTTA concentration in PS (mmol l-1)	EuTTA: PS weight ratio in the coating	Red intensity (counts)	Temperature coefficient (%/°C)
1.15	1:1000	196	−1.39
2.30	1:500	380	−1.54
4.60	1:250	400	−2.03
11.50	1:100	470	−2.11

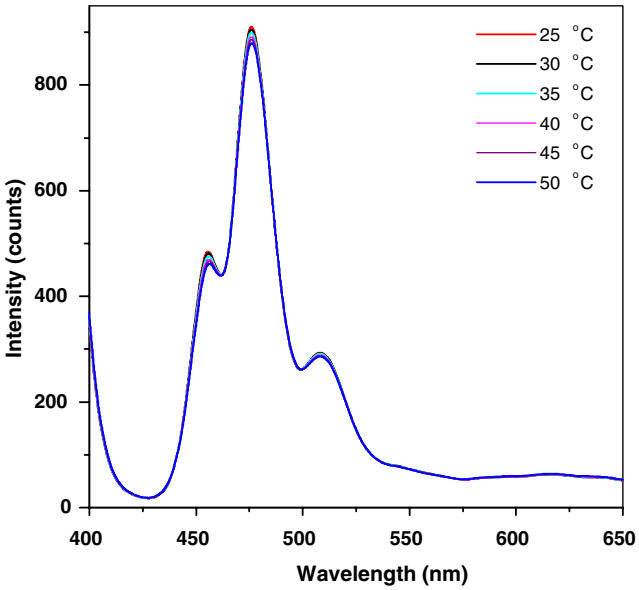

Fig. 2 Effect of temperature on the fluorescence emission spectra of a coating of perylene in polystyrene matrix

in dope, polystyrene and PMMA respectively. It was found that the fluorescence of perylene displayed a marked dependence on the type of polymer matrix as well as on the concentration of the perylene in the polymer. The most striking difference among them was the appearance of perylene excimer emission peak at about 625 nm in the case of perylene/dope coating (Fig. 4a). At lower perylene concentrations (1 mg perylene/1 g dope or 3.96 mmol l^{-1}), only perylene monomer emission peak was observed at about 474 nm. However, as perylene concentration was increased from 1 mg to 3 mg, perylene excimer peak appeared at around 625 nm. The appearance of excimer

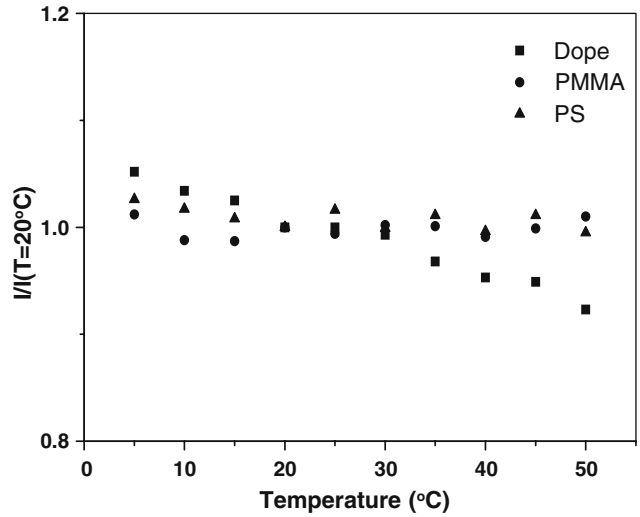

Fig. 3 Temperature dependence of the normalized intensities of coatings of perylene in different polymers, namely dope, PMMA and PS

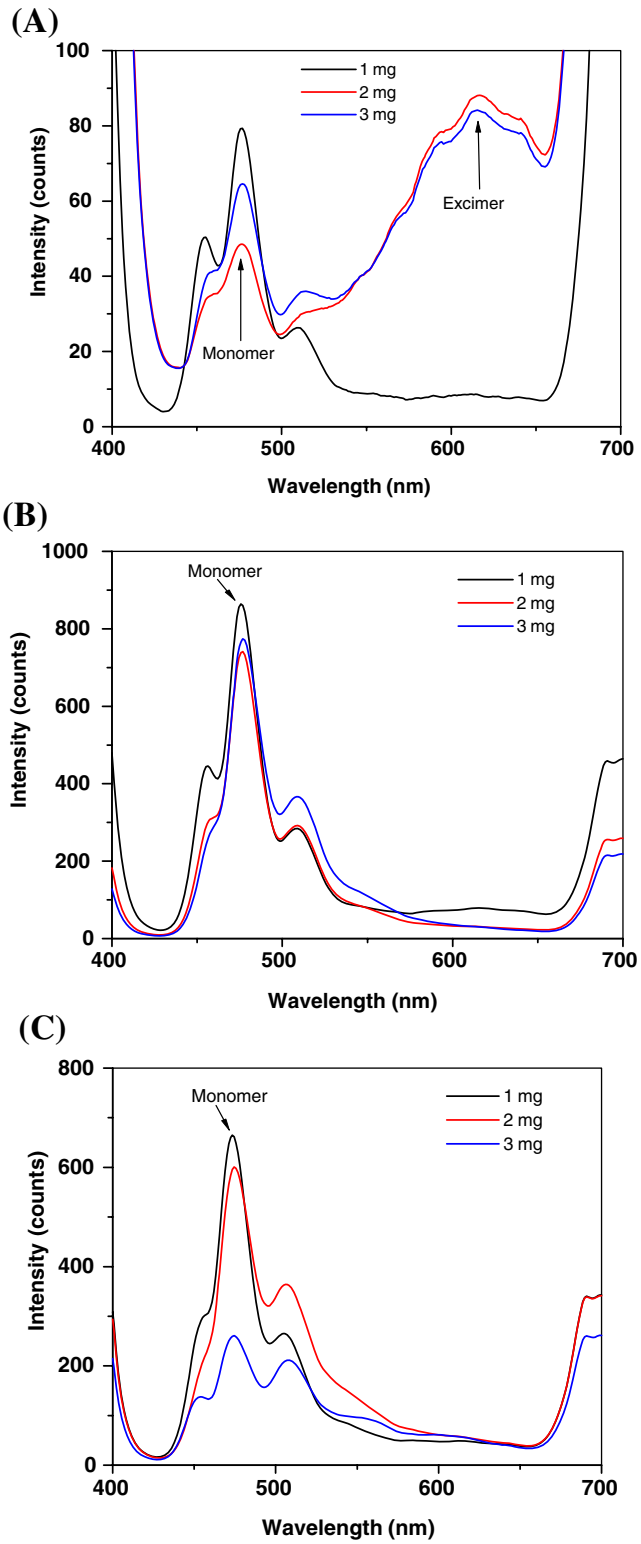

Fig. 4 Effect of perylene concentration on the fluorescence emission spectra of perylene coatings with different polymers **a** dope, **b** PS, **c** PMMA

peak was accompanied by decrease in the emission intensity of the monomer peak. In contrast to the behavior in dope, perylene did not show any excimer emission in PS and PMMA matrices as shown in Figs. 4b,c. Even when perylene concentration was increased from 1 mg to 3 mg, no excimer emission was observed for both perylene/PS and perylene/PMMA coatings. But there was a drastic reduction in intensity with increase in perylene concentration for perylene/PMMA. This could be due to the self-quenching of the fluorescence of perylene monomer. However, in the case of the perylene/PS, the self-quenching of perylene monomer peak at 474 nm was much less compared to that seen for perylene/PMMA. Thus the concentration dependence of intensity was much less for perylene/PS in this concentration range. Further the intensity counts at 474 nm was higher for perylene/PS and perylene/PMMA compared to perylene/dope. Thus the emission characteristics of perylene were dependent on dye concentration and the type of polymer used.

This marked difference in the photophysical behavior of perylene in various polymer matrices is very interesting. Though a lot of work has been reported in literature on the photophysical studies of luminophores in solution, not much work has been done on the luminophore-matrix interactions in solid films.

Bi-luminophore temperature sensitive coating

A bi-luminophore coating containing a probe and a reference luminophore can eliminate the errors caused by non-uniform illumination of test surface and coating thickness. Our study has shown that perylene can be

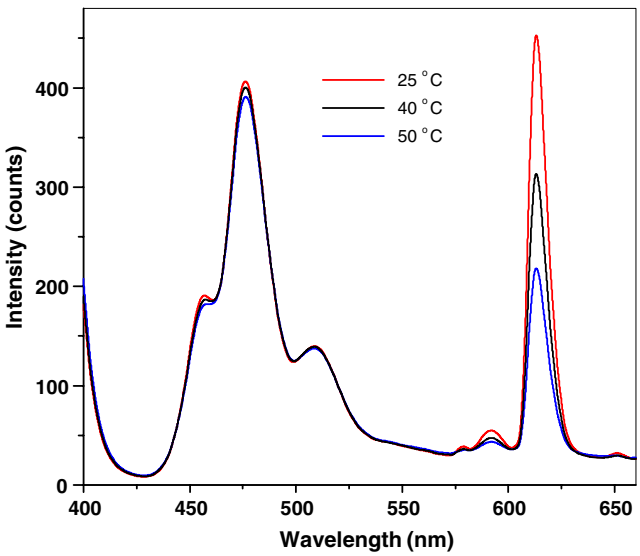

Fig. 5 Effect of temperature on the fluorescence spectra of the bi-luminophore temperature sensitive coating, (EuTTA/ perylene/PS)

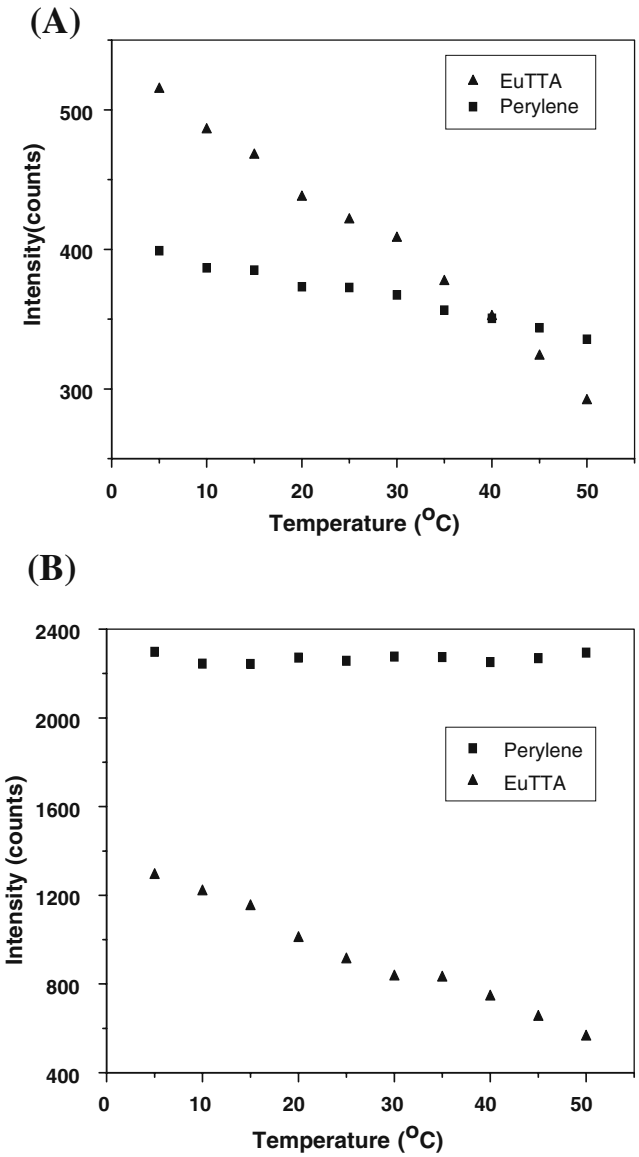

Fig. 6 Variation of luminescent intensity of EuTTA and perylene with temperature for the bi-luminophore coatings, (A) EuTTA/ perylene/ dope and (B) EuTTA/ perylene/PMMA

employed as reference luminophore for the development of bi-luminophore temperature sensitive coating because of its negligibly low temperature sensitivity. The excitation wavelength of both EuTTA and perylene is in the range of 330 to 340 nm and their luminescent emissions are well separated. Perylene has a blue monomer emission at about 474 nm, and the blue emission intensity of perylene/PS is nearly constant in the temperature range of 5 °C to 50 °C. Both perylene and EuTTA are incorporated in polymer matrix to prepare bi-luminophore temperature sensitive coatings.

In order to select the best binder polymer, coatings were prepared by doping EuTTA and perylene in polymer solutions of PS, dope and PMMA. The concentrations of EuTTA and perylene respectively were 2 mg and 1 mg in 0.5 g polymer (4.6 and 7.92 mmol l^{-1}). Fig. 5 shows the effect of temperature on the fluorescence emission spectra of a bi-luminophore coating containing EuTTA and perylene in polystyrene. The blue emission of perylene monomer at 474 nm remained nearly constant whereas the red emission of EuTTA at 615 nm decreased steadily with increase in temperature.

The bi-luminophore temperature sensitive coupons were calibrated in the PSP calibration system. Intensity of EuTTA and perylene versus temperature plots in the temperature range of 5 to 50 °C for EuTTA/perylene/ dope and EuTTA/perylene/ PMMA are shown in Fig. 6. A similar plot for EuTTA/perylene/PS is shown in Fig. 7. Maximum temperature sensitivity was obtained with polystyrene matrix. In order to investigate the reversibility of the sensor performance, intensity measurements at different temperatures were done over a heating run followed by a cooling run. Figure 7 compares the emission intensity at each temperature on both heating and cooling cycles for EuTTA/-perylene/PS coating. As can be seen from Fig. 7, the plots were reversible during both heating and cooling cycles. The coating was stable for several months if stored without exposure to bright light. There was no variation in the calibration results of coatings prepared in different batches and hence the reproducibility was good.

The normalized intensity ratio was obtained by dividing the red counts of EuTTA by the blue counts of perylene at each temperature and normalizing that value with the intensity ratio at reference temperature, 20 °C. From the calibration plots of normalized intensity ratio versus temperature of the three bi-luminophore sensor coatings,

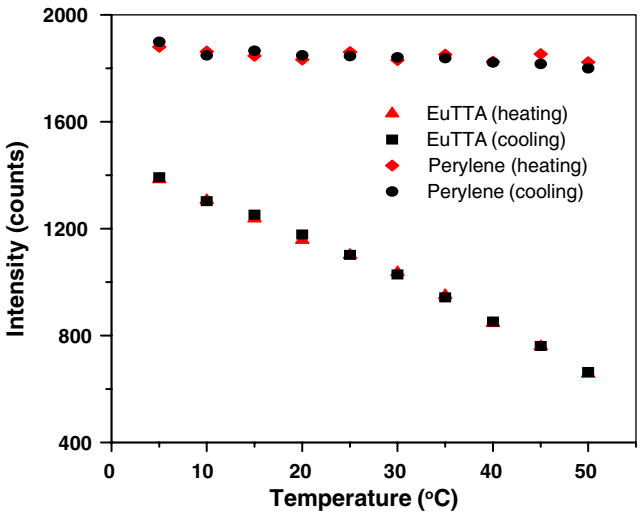

Fig. 7 Variation of the luminescent intensity of EuTTA and perylene with temperature for EuTTA/perylene/PS coating. The graphs compare both heating and cooling data

the temperature coefficients of EuTTA/perylene/dope, EuTTA/perylene/PMMA and EuTTA/perylene/PS were obtained and found to be −0.77, −1.50 and −1.80%/°C respectively. Best linearity and maximum temperature coefficient were obtained for EuTTA/perylene/PS coating. The temperature coefficient of bi-luminophore coating (EuTTA/perylene/PS) is about 10% lower than the EuTTA/PS coating, possibly due to the slight temperature dependence of perylene.

Earlier we have reported the temperature dependence of the luminescence lifetime of EuTTA in PS [14]. We found that the fluorescence decay of EuTTA was double exponential and the mean lifetime decreased with increase in temperature. However, lifetime versus temperature plot of EuTTA-PS was not very linear in the temperature range of 10 to 60 °C. Mean lifetime of EuTTA-PS at T=20 °C was 480.1 μs and $\Delta\tau/\Delta T$ was −1.69 μs/ °C. Therefore, lifetime based temperature coefficient was −0.35%/ °C which is much lower than the temperature coefficient obtained by the ratiometric fluorescence intensity measurements described here.

Conclusions

A new dual-luminophore system for optical sensing of temperature is described. The coating was developed by embedding EuTTA and perylene in polystyrene matrix. Both luminophores were excited in the UV region of about 330 to 380 nm. The fluorescence emission of perylene and EuTTA were well separated. Fluorescence emission spectra of perylene-based coatings displayed a marked dependence on the polymer matrix as well as on the concentration of the perylene in the coating. It was found that coatings containing an optimum concentration of perylene in polystyrene matrix had almost no temperature sensitivity. Thus perylene was used as a reference luminophore in the new bi-luminophore temperature sensitive coating. Combining EuTTA and perylene in polystyrene matrix, a new bi-luminophore temperature sensitive coating was developed. The temperature sensitivity of the bi-luminophore coating was −1.80%/° C in the temperature range of 5 °C to 50 °C. The emission characteristics of this temperature sensitive coating displayed a fully reversible response to temperature. The coating has potential for surface temperature mapping of wind tunnel models.

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