



## A novel pyrene-based binary pressure sensitive paint with low temperature coefficient and improved stability

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### ABSTRACT

Pyrene-based pressure sensitive paints (PSP) have certain advantages due to their high pressure sensitivity and low temperature coefficient but their major drawback is the paint degradation under wind tunnel conditions. This is due to the loss of pyrene as a result of diffusion and sublimation from the coating. We have developed a novel stable binary PSP formulation in which pyrene is covalently bonded to the polymer binder so that paint degradation is prevented. The coating thus obtained is a siloxane-based hybrid organic–inorganic material. The pressure sensitive paint also contains an additional reference luminophore, which is insensitive to pressure but sensitive to intensity variations on the model surface. The second luminophore is incorporated in the paint to correct for the excitation intensity variations during the wind tunnel experiment. The temperature coefficient of second luminophore exactly cancels the temperature coefficient of the pressure sensitive luminophore, thereby resulting in a binary paint with negligible temperature sensitivity.

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### 1. Introduction

The pressure sensitive paint (PSP) technique is an experimental method for the quantitative measurement of surface pressure on wind tunnel models [1–10]. PSP technique has some important advantages over the conventional method of pressure taps that are installed at discrete points on the model in a wind tunnel. The main advantage of PSP is the high spatial resolution so that a complete pressure mapping of the entire surface of the model can be obtained, whereas pressure taps provide data only at pre-selected discrete points. PSP is based on the principle of dynamic quenching of luminescent molecules by oxygen. It contains luminescent sensor molecules embedded in a transparent oxygen-permeable binder. The paint is applied as a thin coating over the wind tunnel models. When the paint coating is illuminated with light of appropriate wavelength, the sensor molecules become excited electronically to a higher energy state. The molecules undergo transition back to the ground state and they emit luminescence. Some of the excited molecules return to the ground state by colliding with an oxygen molecule and this process is known as oxygen quenching. The rate of quenching is proportional to the local oxygen partial pressure, which in turn is proportional to the air pressure. Thus the luminescence of the PSP coating varies inversely with the local air pressure on the surface of the coating.

Paint is an important key element of PSP technique. Conventional pressure sensitive paints are normally prepared by simply mixing and dispersing photo-luminescent materials having oxygen-quenching properties in oxygen-permeable polymer binders [1–7]. The luminescent materials used for PSP may be broadly classified into three categories: ruthenium dyes, metalloporphyrins, pyrene or pyrene derivatives. These luminescent molecules are excited in the ultraviolet or in the visible range and they emit luminescence in the visible region. Pyrene-based PSPs have a special advantage as they have an inherently lower temperature sensitivity compared to other PSPs based on ruthenium dyes and porphyrin dyes.

But pyrene-based paints have a major drawback of significant paint degradation under wind tunnel conditions due to the loss of pyrene by sublimation from the coating [7–9,11].

Pyrene-based pressure sensitive paints are often prepared by mixing pyrene solution with silicone polymer solution as well as other constituents such as cross-linker, catalyst, pigment, solvent, etc. But the fluorescence intensity of these paint coatings degrades steadily with time and rapidly during wind tunnel experiment [7–9]. We have studied the mechanism of degradation of pyrene-based paint coatings and found that paint degradation is due to diffusion and sublimation of pyrene from the coating [11]. It is well established that pyrene excimer formation is a diffusion-controlled process [12]. It is also known that the diffusion coefficient of pyrene in silicone matrix is very high [12–14]. It has been reported that pyrene has mobility in polymers like polydimethyl siloxanes since these polymers have high free volume [13]. Chu and Thomas have

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calculated the diffusion coefficient of pyrene in PDMS resins with high molecular weight and found it to be high. The comparatively long Si–O and Si–C bonds reduce steric conflict, which facilitates freedom of rotation of methyl group about the Si–O and Si–C bonds. This unique structural feature leads to a significant free volume in bulk PDMS. The diffusion of pyrene does not require the movement of the whole macromolecule, but a rotation of the side groups of the resin is sufficient for solute diffusion. The same is true for pyrene in silicone coatings also. The pyrene molecules are in constant motion within the bulk of the silicone coating and when they reach the surface they undergo evaporation.

Therefore the diffusion and sublimation of pyrene from the paint coating has to be prevented to enhance paint stability. Various methods have been employed to improve paint stability. One of the methods is to substitute pyrene by a suitable pyrene derivative [15–17]. But most of the commercially available pyrene derivatives do not exhibit excimer emission when they are embedded in silicone matrix [15]. Earlier we had prepared a pressure sensitive paint using a pyrene derivative (1-decanoyl pyrene butanoate) synthesized by us [16]. This coating had exhibited high quantum efficiency for its excimer emission and a pressure sensitivity of about 70%/bar but the coating on model showed a 10% decrease in fluorescence intensity after 10 wind tunnel blowdowns in wind tunnel. Researchers at ONERA have synthesized new pyrene derivatives and studied their photophysical properties [17]. But these compounds also were not suitable for PSP application as none of these compounds were found to meet all the important PSP requirements like high quantum efficiency for excimer emission, high pressure sensitivity and good stability.

Thus the paint degradation observed during wind tunnel test is a major defect of pyrene-based paints prepared by simply mixing and dispersing pyrene or pyrene derivative in a suitable oxygen-permeable polymer binder. Therefore there is a definite need for preventing paint degradation by immobilization of pyrene on the binder polymer. Engler et al. [8] have described the limitations of conventional PSPs at low speed conditions. The required pressure accuracy is about 0.1% in the typical low speed range between 800 and 1000 mbar. This high accuracy is hard to reach because of the errors related to PSP technique. The main source of error is the temperature changes on the PSP-coated model. Therefore, a temperature-insensitive paint formulation should be used to achieve high accuracy of results in low speed flows. Their studies have shown that their pyrene-based paint formulation was suitable for low speed flows because of its high pressure sensitivity but the drawback of the paint was significant degradation of the pressure sensitive component occurring under flow conditions. Le Sant and Merienne [9] have reported a pyrene-based binary paint containing pyrene and a reference luminophore of gadolinium oxysulfide having very low temperature sensitivity but this paint also suffered degradation in the course of wind tunnel test.

In our approach, pyrene is covalently bonded to the PDMS silicone binder by using a silane-coupling agent, so that it does not undergo diffusion and sublimation. The stability of this novel pressure sensitive paint was assessed by wind tunnel aging tests. It was found that there was no change in the fluorescence intensity (blue emission due to pyrene excimer) of the coating after 50 wind tunnel blowdowns in wind tunnel.

## 2. Experimental

### 2.1. Materials required

Pyrene butanol, tin octoate, dibutyl tin dilaurate (DBT) were procured from Sigma–Aldrich. Isocyanatopropyltriethoxysilane (ICPTES) was from Lancaster. PDMS resins were procured from ABCR GmbH, Germany. Dichloromethane (AR), toluene (AR), ace-

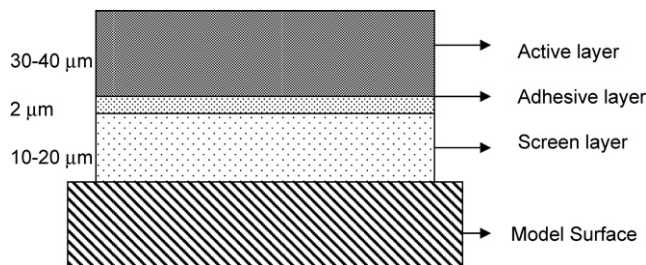


Fig. 1. Different layers of coating the model surface with PSP.

tone, xylene and butyl acetate were from Merck. Red phosphor was procured from M/s Phosphor Technology, UK. All the chemicals were used as received.

### 2.2. Instrumentation

Calibration of the PSP coupons was carried out in the calibration chamber of NAL PSP System. It consists mainly of an excitation source, calibration chamber and detector. Excitation light is provided by a pulsed xenon source filtered in the ultraviolet range  $335 \pm 5$  nm, which is capable of simultaneously exciting both luminophores in the paint coating. The calibration chamber has provision for controlling pressure from 0.10 to 1.60 bar and temperature from 10 to 60 °C. Two peltier-cooled scientific grade 12-bit CCD cameras with resolution of  $1280 \times 1024$  pixels and equipped with suitable filters are used as detectors to acquire images of the blue and red emission from the coating. The data analysis was done by dedicated software of the NAL PSP System. A dry film thickness gauge, QuaNix Keyless was used to measure the thickness of paint coating on the model and coupons.

### 2.3. Preparation of PSP formulation and paint coupons

The novel PSP formulation was prepared by mixing appropriate amounts of pyrene end-labeled polydimethyl siloxane, DBT and a europium-doped rare earth oxysulfide as reference luminophore. The details of the procedure for the preparation of the paint are described elsewhere [18]. Toluene is used as solvent to dilute the paint mixture.

The pressure sensitive paint typically consists of three layers as shown in Fig. 1. They are: (i) screen layer, (ii) adhesive layer, and (iii) active layer. The screen layer is a white basecoat, which creates an optical uniformity on the model surface, enhances the emission intensity signal of the paint and is independent of the model material. The adhesive layer is applied to ensure adhesion between the screen layer and the active layer. The topcoat is the active layer or pressure sensitive layer. A commercially available two-component epoxy paint was used as screen layer as it was found to have good solvent resistance and adhesion to the substrate surface. The thickness of screen layer was in the range of 10–20 μm. A silicone primer GE SS 4044 was applied over the screen layer to improve adhesion of the active layer. The thickness of adhesive layer was about 2–5 μm. PSP coupons and PSP coating on model were prepared by spraying the PSP formulation on the model surface and the aluminum coupons pre-coated with screen layer and adhesive layer.

The model and coupons were cleaned with acetone or a cleaning agent containing acetone, xylene and butyl acetate and coated with screen layer and adhesive layer. It is very important that each layer should be completely cured before application of the next layer. Therefore, the screen layer and primer layer were cured in an oven at 60 °C for 1 h before spraying the PSP formulation. After curing the PSP layer for 24 h under ambient conditions, the coupons were cut into 3 cm × 3 cm size. A small amount of talc was applied

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