

Surface Coatings for Optical Pressure Measurements

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Abstract— A relatively new technique that employs the tools of molecular spectroscopy to measure pressures optically is discussed. Measurements are made via pressure-sensitive molecules attached to the surface of interest. The measurement concept and data-acquisition schemes as well as limitations of the technique are discussed. This paper is focused on identifying the research required to transform this concept into a robust measurement tool. Application goals include enhancement of current steady-state wind-tunnel capability by means of improving measurement accuracy and increasing the operational pressure range of the coatings. These developments will permit the measurement technique to be used in low-speed and high-Reynolds-number wind-tunnel applications. An improved operational range for the coatings will also accommodate studies in turbomachinery. In addition, a principal goal is improving the time response of the coatings to enable transient measurements.

INTRODUCTION

The need in the aerospace community for reliable surface-pressure measurements is tremendous. The ability to map surface pressures non-intrusively with rapid response and virtually limitless spatial resolution would be very useful in a wide variety of applications. Indeed, pressure-sensitive coatings developed for such purposes have already made a significant impact on the area of aircraft model testing [1-6].

There is rapidly growing interest in extending this capability to a variety of wind-tunnel and turbomachinery applications. The accomplishment of these goals requires coating development. Existing pressure-sensitive coatings have been engineered specifically for pressure measurements on stationary aircraft models under atmospheric conditions. Although these coatings could, in principle, be used to address turbomachinery and low-speed wind-tunnel challenges, their utilization would come at the expense of measurement sensitivity and resolution. The relationship between the response of these pressure-sensitive coatings and the measured pressure is nonlinear—sensitivity and resolution are sacrificed as the absolute pressure is increased. Specific coating formulations are characterized

by absolute pressure limits within which they provide pressure measurements with adequate sensitivity/resolution. Furthermore, the coatings exhibit an inherent sensitivity to temperature which has been demonstrated to affect the measurement accuracy. Tailoring the response characteristics of these coatings is a necessary first step to improving the current wind-tunnel capability and unlocking a new world of turbomachinery applications.

The development of current wind-tunnel capability includes improved measurement accuracy and extended operational pressure range. The coating used at Arnold Engineering Development Center (AEDC), for example, is capable of measuring pressures with a nominal accuracy of 0.20 and 0.35 psi at Mach numbers of 0.6 and 0.85, respectively, when applying gross temperature corrections. Significant improvement of the measurement could be achieved if the surface temperature were known at each measurement point. Efforts are underway to develop a dual coating which can be used for the simultaneous measurement of temperature and pressure. Development of such a coating may provide pressure-field data comparable to that available from existing physical pressure-measurement techniques. Improved accuracy would also open the door for use in low-speed wind-tunnel applications, such as airframe and automobile studies, where overall pressure gradients are relatively small (0.03 psi). Extension of the operational pressure range of the coatings will provide the means for measurement in high-Reynolds-number wind-tunnel and turbomachinery facilities.

In the case of turbomachinery, the desirable measurements include pressure-field determinations on 1) the suction- and pressure-surfaces of compressor blades, 2) centrifugal compressor inlets, and 3) compressor cases and hubs. Development of temperature-measurement capability opens up other turbomachinery applications that include identifying combustor hot spots and characterizing the effectiveness of turbine-blade film-cooling schemes.

This surface-coating technology has tremendous potential for development into a robust measurement tool. This paper is intended as a tutorial, providing potential users with a general understanding of the measurement

concept and specific techniques through which the behavior of these sensitive molecules can be exploited to measure pressure and/or temperature. In addition, the paper identifies challenges that must be met through continuing research to ensure the successful application of this technology to a wide variety of interesting and important measurements. While this tutorial emphasizes the underlying photophysical processes that are germane to the pressure-measurement process, an excellent general overview that also addresses hardware issues in detail has been prepared by Crites and Benne [7].

LUMINESCENCE

A number of luminescent molecules, or lumophores, show great promise for optical detection of oxygen (O_2) [8]. When these materials are applied to a surface as a coating, oxygen concentration can be measured over that surface. Since this measured oxygen concentration is directly related to the air pressure at a given temperature, the pressure over the entire surface can be accurately determined. A discussion of the relationship among these luminescent molecules, oxygen concentration, and local pressure is in order.

Two luminescence processes can be effectively utilized to quantitate oxygen. Each of these is described in the context of the Jablonski energy-level diagram for a typical lumophore depicted in Fig. 1 [9]. This diagram is arranged to display electronic and vibrational energy levels with lower-energy states appearing at the bottom of the figure and higher-energy states appearing at the top. Relevant photophysical processes associated with a typical pressure-measurement sequence can be described through the use of this diagram.

Absorption (a) occurs when the lumophore is exposed to electromagnetic radiation of appropriate energy (i.e., frequency). During this process, electrons are promoted from the ground vibrational state of the S_0 electronic manifold to excited vibrational states in the S_1 and S_2 electronic manifolds. Higher-lying excited electronic states not depicted in Fig. 1 may be populated as well. Once it exists in an excited state, the lumophore may undergo a number of processes to return to its stable, lowest-energy configuration. These deactivation processes can be broadly grouped into two general categories: 1) nonradiative deactivation processes in which the excess energy associated with the absorbed photon is transferred to the surrounding medium and 2) radiative deactivation processes in which the excess energy is emitted as light.

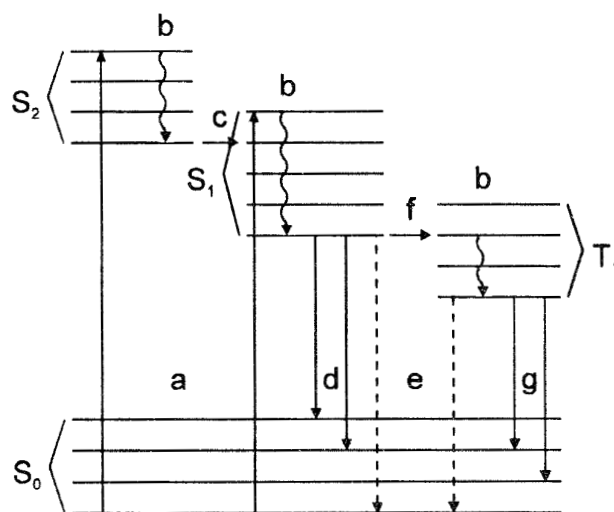


Figure 1. Jablonski diagram depicting the vibrational and electronic states and photophysical processes associated with a typical lumophore.

Immediately following the absorption or excitation event (a), the excited lumophore relaxes to the lowest-energy vibrational state of the excited electronic state (S_1 and/or S_2) through vibrational relaxation (b) in which excess vibrational energy associated with the lumophore is transferred to thermal motion of the surrounding medium. Vibrational relaxation in the S_2 manifold (or in higher-lying excited electronic manifolds) is followed by internal conversion (c) to lower-lying excited electronic states. This nonradiative process couples states of the same multiplicity (multiplicity describes the number of unpaired electrons associated with a particular energy state). In this example S_1 and S_2 are singlet states, i.e., states in which the valence electrons are fully paired. Through nonradiative sequences of internal conversion and vibrational relaxation, the excited lumophore rapidly relaxes to the lowest vibrational state of the S_1 excited electronic state.

From this energy state, the molecule may undergo a number of different processes. Under some circumstances, internal conversion to the ground electronic state, S_0 , with subsequent vibrational relaxation is an important deactivation mechanism. However, fluorescence (d) and external conversion (e) through dynamic quenching are the key processes associated with measurements based on pressure-sensitive coatings. Fluorescence is a luminescence process that couples states of the same multiplicity. During this process, excess energy is emitted as photons to yield

lumophores in the ground electronic manifold. External conversion is a nonradiative process that competes with fluorescence for deactivation of the S_1 excited electronic state. External conversion through dynamic quenching occurs when oxygen molecules collide with the excited lumophore, and energy is transferred from the lumophore to the quenching oxygen molecules. The competition between fluorescence and external conversion (dynamic quenching) permits the quantitation of oxygen.

In addition to internal conversion, external conversion, and fluorescence, the S_1 excited lumophore can undergo intersystem crossing (f), a process in which the excited singlet state couples to a triplet state, T_1 . This event involves a change in multiplicity from the singlet state in which the valence electrons are fully paired to a triplet state in which two electrons are unpaired. The vibrationally relaxed T_1 state can couple to S_0 through intersystem crossing, external conversion (dynamic quenching), or phosphorescence (g). The first two processes are non-radiative, while phosphorescence involves photon emission. Once again, the competition between dynamic oxygen quenching and phosphorescence can be used to quantitate oxygen concentration.

It is important to note that the Jablonski diagram and photophysical processes identified in this discussion best describe the characteristics of “well-behaved” organic lumophores in liquid solution. While an excellent general guide to the spectroscopic behavior of luminescent molecules, this model should be applied with caution to predicting the behavior of specific pressure-sensitive coating formulations. Organometallic lumophores immobilized in solid matrices can display somewhat different characteristics.

FLUORESCENCE vs. PHOSPHORESCENCE

Clearly, a number of important differences exist between the underlying photophysics that produce fluorescence and phosphorescence. Each of the two luminescence processes has advantages and disadvantages when utilized for optical pressure measurements. Specific differences in these processes involve timescales, sensitivities to dynamic quenching, temperature sensitivities, and spectral emission characteristics.

The events that result in fluorescence emission involve fully allowed spectroscopic transitions that occur between states of the same multiplicity. As a result, the timescale for spontaneous fluorescence typically ranges from 10^{-10} to 10^{-6} s. Phosphorescence, on the other hand, involves transitions

that require a change in multiplicity and are, therefore, quantum-mechanically forbidden. Phosphorescence occurs on a timescale ranging from 10^{-4} to 10^4 s as a consequence of this spectroscopic restriction. This enormous difference in emission timescales has significant implications for applying optical pressure-measurement techniques to the study of transient or unsteady phenomena.

In addition to this multiplicity difference between fluorescence and phosphorescence and its effects on the timescales of the processes, it is important to note the differences in the dynamic quenching effects on the processes. Quenching deactivates S_1 to compete with fluorescence and deactivates T_1 to compete with phosphorescence. Because of the long timescales associated with phosphorescence, dynamic quenching that occurs at a given bimolecular quenching rate has a more pronounced effect on a phosphorescence signal than on a fluorescence signal. The long lifetime of the T_1 excited electronic state increases the likelihood that quenching collisions will take place. In other words, quenching competes more effectively with the slower phosphorescence process than with the faster fluorescence process. This lifetime dependence is treated formally in the Stern-Volmer kinetic model described below.

These differences in sensitivity to dynamic quenching have two important implications. First, phosphorescence measurements of oxygen—and, therefore, pressure—are more sensitive than fluorescence measurements. For a given change in oxygen concentration, the change in phosphorescence intensity is greater than that in fluorescence intensity. As a result, phosphorescence measurements permit the experimental resolution of smaller changes in absolute pressure. The second implication involves the effects of temperature on the two processes. Dynamic quenching rates increase with temperature. As a result phosphorescence, which is more sensitive to dynamic quenching, is also more sensitive to fluctuations in temperature. In a coating formulation designed to measure temperature fields, this is a desirable quality; however, this increased sensitivity to temperature can be problematic when making pressure measurements with a phosphorescence-based coating, as noted above.

These differences in timescale and dynamic quenching sensitivity must be considered when designing a pressure-sensitive measurement scheme. In fact, the selection of a luminescence process for a particular pressure measurement will likely involve a tradeoff between optimizing pressure sensitivity (phosphorescence) and time response (fluorescence).

Finally, a practical difference between the two forms of emission involves the wavelength at which emission occurs. As depicted in the Jablonski diagram, the T_1 state from which phosphorescence occurs is typically of lower energy than the S_1 state from which fluorescence occurs. The relationship between the emission wavelength, λ , and the transition energy, ΔE , associated with a luminescence process is given by:

$$\Delta E = \frac{hc}{\lambda} \quad (1)$$

where h is Planck's constant (6.626×10^{-34} Js) and c is the speed of light (2.998×10^8 ms $^{-1}$). Because it occurs from a lower-energy state, the phosphorescence emission is red-shifted with respect to the fluorescence emission, making it easier to isolate spectrally from the excitation source, particularly when a broadband source such as an arc lamp is used to excite luminescence.

MEASUREMENT TECHNIQUE

The effects of dynamic oxygen quenching on luminescence emission are evident in both the intensity and the duration of the observed fluorescence or phosphorescence signal. Figure 2 demonstrates the time-resolved effects of oxygen quenching at various oxygen concentrations in parts per million (ppm) on the fluorescence from pyrene dissolved at the 10-ppm level in isooctane. The change in fluorescence lifetime with increasing oxygen concentration is dramatic. While these particular data have been normalized to a peak intensity of one at each oxygen concentration, a similarly dramatic reduction in both the peak and integrated fluorescence intensities is observed in the raw signals with increasing oxygen concentration. In practice, either the intensity or the lifetime of luminescence emission can be employed to quantitate oxygen concentration.

Luminescence Intensity

In an intensity-based Stern-Volmer model, the effects of dynamic oxygen quenching on the luminescence intensity of a photoexcited probe molecule are described by:

$$\frac{I_0}{I} = 1 + K_q P_{O_2} \quad (2)$$

where I is the measured luminescence intensity, I_0 is the luminescence intensity in the absence of oxygen quenching, P_{O_2} is the partial pressure of oxygen, and K_q is the Stern-

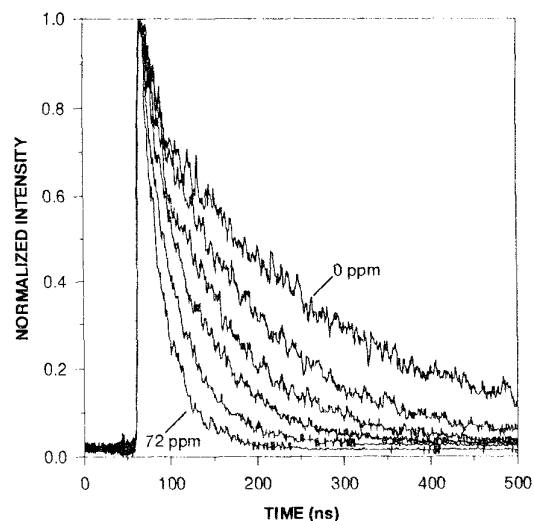


Figure 2. Time-resolved fluorescence signals from pyrene-doped isooctane solutions recorded at various dissolved-oxygen concentrations [10].

Volmer constant. The first step in applying this model to practical measurements involves recording the luminescence intensity as a function of pressure under controlled conditions to calibrate the model and extract a value for K_q , which represents the sensitivity of the measurement.

Analysis of the Stern-Volmer kinetic model reveals that the Stern-Volmer constant, K_q , is given by the product of the unquenched luminescence lifetime, τ_0 , of the selected lumophore and the bimolecular collisional quenching rate, k_q :

$$K_q = \tau_0 k_q \quad (3)$$

This observation quantitatively describes the dependence of the dynamic quenching sensitivity on the luminescence lifetime and explains the difference in sensitivity between fluorescence and phosphorescence. Because fluorescence lifetimes are shorter, K_q is smaller and fluorescence is less sensitive to dynamic quenching.

In actual application of a Stern-Volmer model to the performance of pressure-field measurements, the luminescence intensity at a reference condition, I_{REF} , must be determined. This reference intensity is typically acquired over the surface of interest under quiescent conditions at a

controlled pressure, P_{REF} . When the luminescence intensity at a given test condition, I , is measured over the area of interest, a ratio of the measured intensities yields the desired pressure information:

$$\frac{I_{REF}}{I} = 1 + K_q \frac{P}{P_{REF}} \quad (4)$$

Ratioing the luminescence intensities I and I_{REF} not only provides the desired pressure measurements but also effectively eliminates signal dependence associated with nonuniformities in coating thickness and illumination. Intensities are generally sampled over the area of interest using some type of detector array, such as a CCD (charge-coupled device) camera. The output of the array can be visually represented as an image, with the luminescence intensity displayed in pseudo-color or gray scale.

Because model displacement often occurs between acquisition of the reference (“wind off”) and test (“wind on”) images, one of the most important requirements associated with intensity-based pressure measurements is careful alignment of these two images before the luminescence intensity values are ratioed. Correct alignment is achieved through the use of small circular marks placed directly on the model which appear in both the reference and test images. After alignment is complete, the image-intensity ratio is determined, Equation (4) is applied, and the pressure is evaluated over the entire image plane on a pixel-by-pixel basis.

The principal disadvantage of the intensity method is the requirement of a reference image which significantly increases the amount of data that must be acquired. Furthermore, alignment of the two images to determine the ratio is often limited by the magnitude of the deflections and deformations that occur in the transition from the reference to the test image. Large or complex deflections and deformations inhibit the alignment mark registration, making image alignment difficult and producing blurred images and inaccurate results. If the differences are sufficiently large, alignment could be impossible. In spite of this limitation, the luminescence intensity technique has been successfully used in numerous wind-tunnel experiments.

References 1-6 include examples of successful wind-tunnel pressure-sensitive coating experiments. Figures 3 shows some typical results achieved with the technique described. In the figure, pressure-sensitive coating data is compared to a Navier-Stokes CFD (Computational Fluid

Dynamics) solution. Although this comparison is purely visual, it is clear that CFD does not predict the vortex shedding observed at the notch located on the leading edge of the airfoil. Similar results published in Ref. 9 compare pressure-sensitive coating data to information acquired with a row of pressure taps on an F-18 aircraft model. The coefficient of pressure measured with the coating agrees with the tap data to within ± 0.02 . Because the coating provides a field measurement, a more accurate assessment of the integrated loads on the aircraft can be obtained. Preliminary tests have demonstrated that this technique can be used to make steady-state pressure measurements in rotating machinery as well [11].

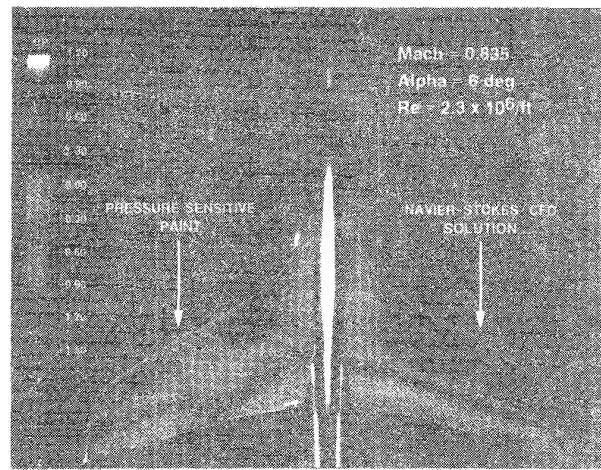


Figure 3. Pressure-sensitive coating/CFD comparison [5].

Luminescence Lifetime

An alternative to ratioing intensity values from reference and test images involves basing the measurement on luminescence lifetimes. The luminescence lifetime can be determined by using a pulsed light source and recording the observed luminescence as a function of time. Alternatively, an amplitude-modulated light source can be employed and the lifetime extracted from frequency-domain measurements of the luminescence intensity [12]. In either case, the relevant Stern-Volmer kinetic model is represented by Equations (5) and (6):

$$\frac{\tau_o}{\tau} = 1 + K_q P_{O_2} \quad (5)$$

$$\frac{\tau_{REF}}{\tau} = 1 + K_q \frac{P}{P_{REF}} \quad (6)$$

where τ , τ_0 , and τ_{REF} are luminescence lifetimes at the test condition, in the absence of quenching, and at P_{REF} , respectively. The advantage of this technique is that the measured decay time is independent of variations in coating thickness and illumination. As a result, the reference image is not required. A potential disadvantage of this technique is the complexity associated with acquiring time- or frequency-domain data; however, a number of schemes have been proposed that utilize imaging detectors to achieve two-dimensional lifetime-based pressure measurements.

Examination of both the intensity and lifetime versions of the Stern-Volmer kinetic model reveals an important aspect of the quenching technique for measuring oxygen concentration and pressure: the measured signal decreases with increasing absolute pressure. This inverse relationship is manifested in a pressure-dependent measurement sensitivity. This effect is clearly demonstrated by the data displayed in Fig. 4. The error bars on these measurements of dissolved oxygen in cyclohexane solutions of pyrene increase with pressure. This effect must be considered when designing a pressure-sensitive measurement system for a particular application. Ideally, coatings will be developed that exhibit high pressure sensitivity and measurement accuracy at specific nominal pressures. This development will provide a suite of coatings from which the experimentalist can select when performing experiments at a given absolute pressure.

REQUIRED DEVELOPMENT

Achieving the goal of extending the usefulness of this new measurement technique to the steady-state and transient applications described above will require that several technical challenges be addressed. These include minimization of the temperature sensitivity of the coatings, extension of the operational range of the measurement technique, and improvement of the schemes for attaching pressure-sensitive molecules to the surface of interest. During coating development, it is important to bear in mind the detection scheme which will be used to quantitate the coating response.

Coating formulations currently in existence for optical pressure measurements have some degree of temperature dependence due to the nature of dynamic quenching. Since changes in molecular activity caused by temperature variations are unavoidable, it seems unlikely that a coating can

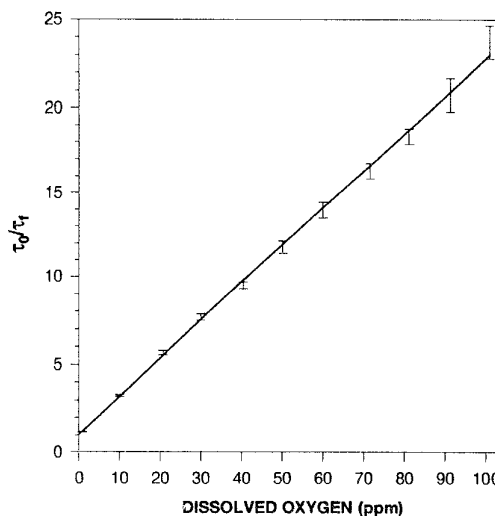


Figure 4. Lifetime Stern-Volmer plot for the quantitation of dissolved oxygen in cyclohexane based on pyrene fluorescence [10].

be designed that will be completely insensitive to temperature. The current solution to this problem involves the use of a temperature-sensitive coating for acquiring temperature data under the same flow conditions used in obtaining pressure data. Efforts are underway to develop a single coating layer that includes temperature- and pressure-sensitive molecules.

Existing coatings have a limited operational pressure range of ~2 atm, with the exception of a coating described in Ref. 13 which is capable of operating over a range of 5 atm. This coating, however, exhibits operation over this increased pressure range at the expense of time response, which is quoted at 0.5 s [13]. Thermal degradation of the pressure-sensitive lumophores in existing coatings limits the maximum operating temperature to ~120°C. Therefore, extension of this technique to higher-temperature applications will require the synthesis of new luminescent molecules.

For unsteady measurements in the first stages of a compressor, for example, a coating must be able to respond to pressure changes on the order of 40 KHz. Current formulas are far from achieving this goal. Bare fluorophores have the potential to measure transient phenomena—typical fluorescence lifetimes cited above

correspond to response bandwidths ≥ 1 MHz. Current methods involve mixing the molecules into a binder which is sprayed onto the prepared surface. Unfortunately, the transport properties and thickness of the binder layer can drastically reduce the response time of the coating. The fastest response time currently available is ~ 200 Hz [12]. Alternative techniques for attaching the molecules to the surface of interest must be sought.

Other concerns involving attachment of the active molecules to the surface of interest include surface roughness and durability. General observation of the various coatings currently available lead to several conclusions. The faster-responding coatings are soft and somewhat tacky. These coatings affect the aerodynamic drag characteristics of the model under study, and their lack of endurance limits their application. Unfortunately, the harder coatings which may be able to survive rugged test environments such as rotating machinery tend to have slow response times. Thus, improved coating hardness and durability are often achieved at the expense of response time. Development of a technique for attaching lumophores to surfaces of interest without compromising their response time or the surface integrity is essential. In addition, such an adhesion process should be both fast and simple; current coatings require one to three days to apply.

It is extremely important to address the measurement system used in this technique. Because of the potential complexity of this technique, a complete understanding of the system is essential. Two principal problem areas are associated with current data-acquisition systems—speed and size. Modern CCD cameras can acquire a single image on a nanosecond timescale; however, acquisition of a series of images at high framing rates requires streak-camera technology at a cost of $> \$200K$ to produce only a modest number of images (i.e., eight). This results from the fact that CCD arrays are read linearly, one pixel row at a time. Most modern cameras feature a million pixels or more, and typical readout times are on the order of milliseconds. Continuing advances in the design and construction of CCD cameras should address this need. With reference to size constraints, methods must be developed to illuminate and image surfaces of interest in situations where both physical and optical access are limited. Fiber-optic technology represents a potential solution to this problem.

SUMMARY

A number of key issues have been identified in this report. Pressure-sensitive coatings based on fluorescence or phosphorescence exhibit particular advantages and dis-

advantages. Fluorescence lifetimes are considerably shorter than those of phosphorescence, and the time response associated with fluorescent coatings can be considerably faster as a result. This is particularly important with respect to transient measurements. Because of this lifetime difference, phosphorescence is more sensitive to dynamic quenching, changes in oxygen concentration (local pressure), and fluctuations in temperature. These increased sensitivities can be advantageous or problematic depending upon the measurement of interest. Whether fluorescence or phosphorescence is employed, intensity- or lifetime-based data acquisition schemes can be designed to measure the oxygen concentration.

Typical two-dimensional data obtained with the intensity-based method are reported. The problems associated with intensity-based measurements include the reference-image requirements, cumbersome image processing and alignment procedures, limitations due to model deflections and deformations, and sensitivity to coating thickness and illumination variations. The lifetime-based measurement, on the other hand, is essentially independent of coating thickness and illumination variations and does not require a reference image. However, no technique currently exists to acquire two-dimensional lifetime measurements. The development of such an imaging scheme will simplify data acquisition and image processing.

Sensitive coatings for optical measurement of pressure and temperature are currently being used successfully in a number of applications. This technique has the potential to accommodate a wide range of applications, including rotational environments. Near-term efforts include characterization and development of sensitive coatings with improved accuracy and extended operating ranges as well as the construction and validation of a high-speed two-dimensional measurement system. Long-term goals include efforts devoted to the development of coatings with fast response times to permit transient measurements.

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