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Non-contact phosphor thermometry for process control

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ABSTRACT

Manufacturing and other industrial processes often require monitoring and control of temperature. Thermometry based on fluorescence properties of surface-bonded phosphors offers a number of advantages over traditional methods. The method is non-contact, remote, and independent of surface optical properties such as emissivity. Only a thin layer, less than 50 microns thick, is required of fluorescent materials that are temperature-active and chemically stable up to temperatures in excess of 1600 C. Phosphor thermometry has been developed from these high temperature extremes all the way down to cryogenic temperatures within liquid helium dewars. The fluorescence effects are stable in time, not subject to drift and need for repeated recalibration. Measurement techniques often involve use of optical fibers and other components that allow access into confined geometries and environments with high vibration, electromagnetic fields, or other extreme conditions. Uses include thermal management of cutting or shaping tools, monitoring of furnace and combustor walls or internal components, assembly components in automated lines, sheet metal surface thermometry, measurement of rotating components in motors, generators, turbine engines, and similar systems, fiber temperature measurement in textile fiber spinning, etc. Fluorescence measurement yields absolute temperatures, not dependent on references, and can have accuracies of less than 1 K, with precisions well below 0.1 K, providing opportunity for ultra high precision process control, life testing, and quality control. Key words: fluorescence, phosphor, temperature, thermometry, fiber optics, control, process, monitor.

1. INTRODUCTION

Many industrial processes, including manufacturing and chemical production, require monitoring and control of temperature. Temperature measurement in the process will often be difficult or impossible by standard contact methods or by use of infrared or pyrometric detectors. These difficulties arise from high-temperature environments, vibration, strong electromagnetic interference, confined space, and from moving components, either rotationally or laterally, that cannot be directly contacted. Thermometry based on fluorescence properties of surface-bonded phosphors offers a number of advantages over traditional methods. The phosphor method is non-contact, remote, and independent of surface optical properties such as emissivity, transmissivity, or the like. Only a thin layer, less than 50 microns thick, is needed of the fluorescent materials that are temperature active and chemically stable, often up to temperatures of 1800 K or higher.

2. PHOSPHOR THERMOMETRIC METHOD

The use of fluorescent materials for temperature determination is a well-established technical area, with considerable activity, especially for ambient and elevated temperatures.¹⁻⁴ The technology, however, also is applicable to measurement of extremely cold objects, extending down to around the temperature of liquid helium, possibly to limiting values near absolute zero.⁵ One major class of fluorescent materials is the group of inorganic oxides, oxysulfides, orthophosphates, vanadates, etc. of the rare earth metals. Other metals, notably zinc, manganese, and a few others, are also constituents of numerous phosphors. Fluorescence in all these materials arises from excited ions, maintained in low

concentrations in the molecular lattice, which have a high probability of de-excitation by a photon emission instead of through other processes in the lattice itself. In the case of rare earth materials, usually another similar rare earth metal is used as a dopant or activator, and in concentrations of less than a percent up to six or eight percent as a practical maximum. These low dopant concentrations have the effect of isolating the dopant ion in the lattice, separating it, in effect, from other ions like itself, so that its behavior under some circumstances is relatively independent of the lattice in which it is bound. As temperature increases the lattice around the dopant increases its vibrations, eventually "intruding" upon the separateness of the isolated ion. When this behavior occurs, the temperature dependence of the fluorescence becomes strong in the region near some temperature, called the *quenching temperature*, and continues with increasing temperature until the emission is extinguished. Since the decay rate, dN/dt , of a given state is proportional to the population in that state, N , dN/dt can be expressed as a proportionality constant multiplied by N , or $-\beta N$. When this expression is integrated, the result is the expression,

$$I = I_0 e^{-t/\tau}$$

where I (proportional to N) is the intensity at time, t , I_0 is the intensity at $t = 0$, and τ (equal to $1/\beta$) is the parameter called *decay time*. The decay time is, by this definition, the time required for the intensity to diminish from some value to $1/e$ of that value, and is a function of the temperature T . To illustrate this temperature behavior for two well-characterized phosphors, Figure 1 shows both the excitation spectrum (for 537 nm emission) and the emission spectrum (from excitation by 337 nm pulsed light) for $\text{La}_2\text{O}_2\text{S:Eu}$ at room temperature. These data were obtained using a Perkin-Elmer spectrophotometer employing a steady-state xenon excitation source. Also obtained from the spectrophotometer, Figure 2 shows excitation spectrum (for 655 nm emission) and the emission spectrum (from excitation by 337 nm light) for $\text{Mg}_4\text{FGeO}_6\text{:Mn}$. The spectra show qualitative relationships among the excitation and emission properties. From examination of the two excitation spectra, it can be seen that 337 nm is well within the band of uv that produces efficient excitation of both phosphors. $\text{Mg}_4\text{FGeO}_6\text{:Mn}$ is particularly interesting in that it can be strongly excited by light in the blue region of the visible spectrum. $\text{La}_2\text{O}_2\text{S:Eu}$, while not excited by a broad band of visible light, is excited by resonant absorption of light of the same characteristic energy as narrow emission lines observed in the blue. The peak at 467 nm in the excitation spectrum for $\text{La}_2\text{O}_2\text{S:Eu}$, for example, is seen in the emission spectrum from 337 nm excitation, shown in Figure 1. Figure 3 is a compilation of fluorescence lifetime (decay time) versus temperature for a variety of thermographic phosphors we have studied. A number of other phosphors with responses overlapping the ones shown are not included, for clarity.

In practice, phosphor thermometry for high-temperature surfaces is often done with a setup similar in concept to that shown in Figure 4. The three major components of the system shown in the illustration are the stimulating sources and optics, photodetection, and signal analysis. Often the stimulating source will be a pulsed ultraviolet laser, such as a simple 337 nm nitrogen gas laser, available in sizes from about 20 cm long on up. Other lasers can be used, providing they can produce a beam that is in the ultraviolet (or sometimes the blue) part of the spectrum. Steady state or modulated light sources can also be used, such as light emitting diodes, xenon or mercury lamps, and the like. In the case of the steady state or modulated sources, a somewhat different set of measurement parameters are required; so, for conciseness, we will discuss only pulsed excitation, realizing that it is only one interrogation technique among many. For high-temperature measurement, however, pulsed excitation has distinct advantages. When surfaces rise to about 700 C and higher, their surface emission moves into the predominantly red region, then on up into yellow, green, and blue. This blackbody emission can

interfere with visible fluorescence that has been stimulated for measurement purposes. When the fluorescence, however, is stimulated by a few nanosecond pulse of laser light, the visible fluorescence in most cases will overwhelm the near-constant blackbody background in the particular wavelength band of interest. Optical fibers are commonly used both to transport the stimulating light pulse to the point of use and return the resultant fluorescence to an appropriate photodetector. Photomultipliers are usually used for the photodetector, although we have recently tested several different types of avalanche photodiode systems. These lower noise, wider dynamic range devices are becoming lower in cost, quite reliable, and somewhat more available. Their major limitation is their optical gain, limited to about one to ten percent of the gain of a photomultiplier.

Electrical signals from the photodetector are recorded as a function of time in an oscilloscope or oscilloscope card associated with a personal computer. Other methods of handling the output electrical signals include integrating and producing a ratio with another reference signal (for normalization purposes), or simply sampling a few points in time with a linear digital detector in order to determine a trend that relates to temperature. In general, however, a full waveform has been recorded and processed digitally in a computer. Often, repeated waveforms, produced by repeated laser pulses, will be averaged to generate a lower noise signal. A typical fluorescence signal, after a possible disturbance from the reflected laser flash, will have a long period, often several decay times, where its natural logarithm is quite linear. The linear slope determines the decay time (see the expression for intensity above), and is extracted between cursors that bound the region selected for stable signal behavior. It is possible to automate the data-taking process by using calibration information in analytical or table-lookup form in the computer code which processes the waveform data. In arrangements of this type, especially when the usual oscilloscope is replaced with a computer card, the thermometry system can be quite compact and easy to transport and set up at measurement locations.

3. PHOSPHOR DEPOSITION AND BONDING

A critical aspect of the phosphor method of surface temperature determination is the need for a strong, thin layer of phosphor that will endure during the measurement time and yield temperatures that are correspondent to those of the surface to be measured. Our experience with high temperature materials is that in many cases surfaces are eroded by some process associated with the particular device. In jet turbine engines, for example, heated high velocity air scours the rotor and stator surfaces, and will often wear off loosely bound layers. The thickness needed for phosphor thermography is in the range of 1 - 50 microns. If cleanliness and care are used, several different methods have been successful in

Figure 1. Excitation and emission spectra for La₂O₂S:Eu.

Figure 2. Excitation and emission spectra for $\text{Mg}_4\text{FGeO}_6:\text{Mn}$.

applying durable coatings in this thickness range. The first and simplest is to use a high temperature *inorganic binder*, stir in the phosphor powder as a suspension, and apply the material by a spraying technique, perhaps with an airbrush. This resultant coating is then heat cured to drive off the volatiles and form molecular bonding configurations. A second common method is to use *electron beam deposition*, produced in a vacuum chamber by electron bombardment and subsequent material vaporization. These coatings tend to form secure surface bonds, especially for thicknesses of about 20 microns or less. Other methods we have used include *ion sputtering*, *epoxy-phosphor mixtures*, *electroplating*, and *laser ablation*. Any of the methods which vaporize the phosphor have the potential of disrupting the molecular configuration. Some phosphors, such as metal oxides, can be reheated in air to reoxidize the material. Other phosphors, particularly those containing sulphur or other ions that are prone to being replaced by more active members of the same chemical family, cannot be deposited by these methods. In nearly all cases it is important to cross-check the temperature calibration of the applied coating, if that is possible, since changes in molecular structure will be reflected in changes in temperature dependence of any fluorescence. Calibration checks can be done in controlled temperature furnaces, which are often available in facilities where high-temperature materials are used, or by locally heating the surface while monitoring it with a contact temperature gauge. In conditions where calibration checks are not feasible, we have applied phosphor with one of the lower temperature methods, knowing that the material calibration will not change due to the application process.

4. PROCESS CONTROL APPLICATIONS

In industrial applications associated with process control optical measurement techniques like phosphor thermometry can be especially effective without adversely disturbing the process itself. Often, optical fibers and other optical components are used for access into confined or hazardous areas. Measurements can be made at some convenient distance, up to many meters away, and signal data can be transported into electrically isolated or safe locations for processing. Capital costs for systems of this type are expected to be low because individual components are inexpensive and available from a number of suppliers. The major development expense is the systems engineering and would not be expected to be higher than similar efforts with other types of standard components. The following examples are representative of industrial and manufacturing process uses for which phosphor thermometry may be especially applicable.

Machine tools employing cutting surfaces that rise to high temperatures during operation, giving rise to corrosion, metal fatigue, or dulling of sharpened edges, can be monitored remotely using the phosphor technique. Thin phosphor coatings on tool surfaces are optically interrogated, the resultant

Figure 3. Decay time versus temperature for various phosphors

fluorescence recorded, and a continuous temperature profile displayed for the operator and any desired performance documentation. In cases like these, contact probes are either likely to have short lifetimes because of vibration, heat, and debris, or if buried within the cutting tool, to give a measure of temperature that does not correspond to the actual cutting surface. The surface phosphor coat, basically a refractory layer, can be very close to the point of cutting contact and on the same surface.

For processes employing furnaces or regions of combustion, phosphor thermometry may be usable to monitor wall temperatures, air temperatures, or temperatures of components being heated. For wall temperatures a phosphor layer can be deposited on metals or

ceramics and accessed either by optical fibers brought through ports or by optical windows. For air (or other gas) temperatures, thin sampling materials coated with phosphor can be used and accessed similarly. Individual components can be separately coated or test pieces, placed nearby, can be coated. In all these cases the phosphor method can usually overcome high-temperature blackbody backgrounds, scattered light from self luminous surfaces, and relatively dirty conditions.

Individual components in assembly lines, i.e., bearings, treated or polished surfaces, regions near welds, bolts, ceramic parts, etc., can be coated (permanently or temporarily) with an appropriate phosphor and remotely monitored for temperature. This approach can be a distinct advantage in large heated process volumes where temperature gradients could be sizable, backgrounds are severe, or absolute component temperature is not known with precision. In other situations, fabrication flaws might express themselves as localized temperature excursions during production, a condition that would be picked up by phosphor thermometry.

Rotating components in motors, generators, turbines, and similar systems are often difficult to monitor because of their rotation, the confined access required, or background light or vibration. In developmental testing, actual fabrication, or as a part of performance and health monitoring systems, phosphor thermometry can provide important temperature information. For shafts or blades, optical methods combined with the thermometry can be used to give instantaneous angular velocities as well as their associated temperatures. This type of component is most likely best accessed by optical fiber configurations, although there are possibilities for use of telescopic instrumentation that would allow measurement at considerable distances.

For *textile and paper production*, small admixtures of phosphor could be used in various situations to allow for temperature monitoring during the process. In slurries, melts, etc., surface dustings of phosphor might also be useful to allow for surface characterization. Some phosphor doped ink or tracer fibers might also be employed in certain situations, perhaps where identification or counterfeit prevention were important. With the several hundred degree useful range of most phosphors, cooling or heating cycles can be followed quantitatively in time. Using two-dimension referencing for the

optical system used, it is also possible to observe shrinkage, displacement, tearing, etc. by observing and quantifying fluorescence.

5. CONCLUSION

Some manufacturing and industrial processes use technology developed decades ago, while other processes depend on advanced state-of-the-air techniques and devices. For both types of process, phosphor thermometry has the potential to add reliable information that will be minimally interfering with the process. Our experience has also shown that the fluorescence technique can give rise to measurement precisions that are often not realizable with any other approach. For surfaces in rotation, for example,⁶ we have performed temperature mapping around rotating perimeters with precisions of about 0.5 C or better. Compatible with use of optical fibers, and able to be performed remotely and in difficult environments, phosphor thermometry represents a potential major improvement in process control and the related issues of quality control, life monitoring, and predictive maintenance.

Figure 4. Typical high-temperature phosphor thermometry design concept.

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