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SURFACE DIAGNOSTICS

S. SVANBERG

Lund Institute of Technology
S-221 00 Lund
Sweden

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A System for Industrial Surface Monitoring Utilizing Laser-Induced Fluorescence

S. Montán and S. Svanberg

Department of Physics, Lund Institute of Technology,
P.O. Box 118, S-22100 Lund, Sweden

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Abstract. A system utilizing laser-induced fluorescence for surface monitoring is described. Results of laboratory and industrial measurements of surface films are reported and further applications of the technique are described.

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In this paper a system for point measurements of laser-induced fluorescence at three wavelengths is described. Simple functions of these three fluorescence intensities can be formed. The system is intended as a general analyzing instrument, especially for on-line process steering and monitoring.

Fluorescence is a well-known physical phenomenon. Through electromagnetic interaction with light, molecules can be excited to higher-lying energy states and subsequently give off their excess energy by emitting photons. This light is termed fluorescence light and is characteristic for the particular molecule. The fluorescence light is always shifted towards longer wavelengths than the exciting light (Fig. 1). This shift is called the Stokes shift and is due to fast radiationless transfers between the rotational and vibrational levels in the upper electronic state [1]. The energy necessary for excitation generally corresponds to a photon in the visible or uv region, typically a few eV. Not all of the deposited energy is given off as fluorescence light, however. Most of it is converted into heat (phonon energy) without any light release. This process is called quenching. Some molecules may be transferred to a triplet state from which radiationless transfers are impossible. The energy is then emitted as phosphorescence light. Phosphorescence is of longer wavelength and has lower intensity than fluorescence light from the same molecule. The triplet states often have a considerably longer lifetime, sometimes of the order of seconds. The fraction of the added energy that is emitted as light is called the quantum efficiency. Molecules with conjugated double bonds are known to have a high quantum efficiency. This property is used in laser dyes.

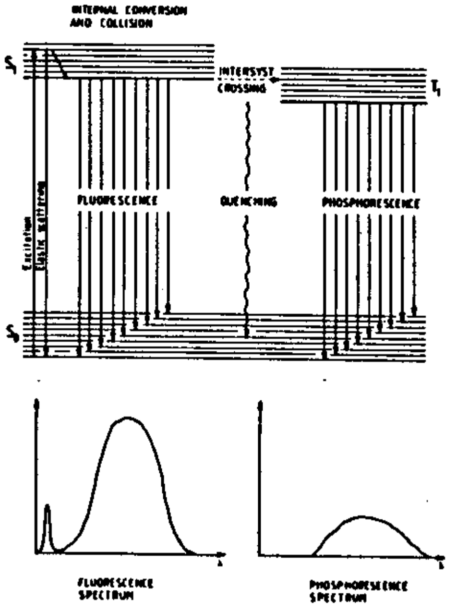

Fig. 1. Schematic molecular energy-level diagram with fluorescence and phosphorescence processes indicated

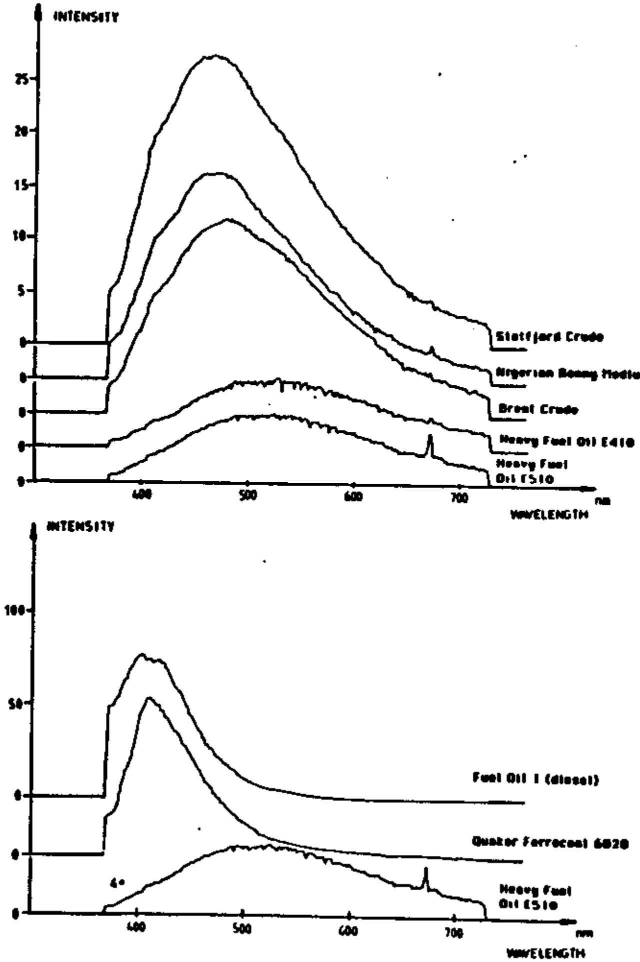

Fig. 2. (a) Spectra of different crude oils and refined products. (b) Spectra of some other oil products. The spectrum of the Heavy Fuel Oil E510 is drawn on a four times enlarged scale. The intensity unit is the fluorescence of Rhodamine 6G in water (70 μg/l)

It is clear that fluorescence can be used for analytical purposes. A chemical analysis in solids and liquids based on fluorescence spectroscopy must be quite rudimentary though, since neither the rotational nor the vibrational structure can be resolved. These structures are generally smeared out over a broad structure because of the interaction with surrounding molecules.

An example of such simple analysis is the characterization of oil products, as shown in Fig. 2. It can be seen that the fluorescence light of refined products is shifted towards shorter wavelengths than crude oils. This can be used for coarse classification of oil slicks at sea. Another example is the possibility of measuring the thickness of thin oil layers on metal surfaces [3]. The metal surface does not fluoresce. The fluorescence intensity therefore increases with the oil layer thickness until the layer is of the same thickness as the optical penetration depth.

The fluorescence spectrum from solids or liquids can have sharp features in exceptional cases. Crystals containing rare-earth atoms or ions, such as $Dy^{+3}:LaF_3$ and $Er^{+3}:CaF_2$, exhibit sharp fluorescence lines that are strongly temperature-dependent. The intensities and lifetimes of the different lines change with temperature, and thus these crystals can be used for non-intrusive measurements of surface temperature on, e.g., moving machine parts using a system with a pulsed uv laser [4–6].

Fluorescence techniques also have several medical applications of interest. The fluorescence microscope and the flow cytometer are well known. In recent years the tumour-marking drug hematoporphyrin derivative (HPD) has experienced much attention [7]. The characteristic fluorescence of HPD may become a powerful tool for early tumour diagnosis. As we have shown [8, 9], an enhanced contrast between tumour and normal tissue can be achieved by considering the function $(A-B)/C$ where A is the intensity at 630 nm, B at 600 nm, and C at 480 nm.

Below, we describe the construction of our fluorescence monitoring system in Sect. 1. Laboratory and industrial measurements are discussed in Sect. 2 and in a final section conclusions from the work is drawn.

1. System Description

A diagram of the system is shown in Fig. 3, and a photograph of the unit is shown in Fig. 4. As a light source a nitrogen laser with the facility for gas exchange is used. The repetition rate is usually 10 Hz. The pulse duration is about 10 ns and the pulse energy is around 1 mJ. The laser beam is shaped by cylindrical and spherical quartz lenses and is directed onto the measurement target by first-surface aluminum mirrors. The beam is filtered with a Schott UG 11 filter, thus suppressing unwanted plasma lines. The light reflected from the filter is directed onto two photodiodes, one of which measures the laser light intensity and the other generates an interrupt to the computer performing the calculations.

The fluorescence light is collected by means of a 15 cm Keplerian telescope. A lens telescope was preferred to a mirror telescope in order to make the apparatus also applicable for short distances. It is also possible to instead transmit the exciting laser light, and to collect fluorescence light, with optical fibres. After collection, the fluorescence light is divided up with dichroic mirrors before it is passed onto photomultiplier tubes (PMTs) that are used as detectors (Hamamatsu R 105, S-4 response). Normally two cut-off filters (Schott WG 360 and GG 375) are placed in the common beam path to suppress elastically scattered laser light. These filters can be removed if the elastically scattered signal (the LIDAR signal) is wanted. It is also possible to insert other filters after the dichroics to select narrower wavelength bands. The signals from the PMTs are detected by gated integrators (Evans Associates, Model 4130). The gate pulse is generated from the laser trigger unit. This is the only electrical connection between the laser and the detection electronics. This construction was chosen to suppress radio-frequency interferences from the laser discharge. The integrators are read out by analog to digital converters (ADCs) and reset after each laser pulse. Averaging and all other handling of output signals is performed by a small microcomputer system built up around a Zilog Z80 CPU. Communication with the computer is via a terminal (serial, V-24). This allows the system to be run by remote control (i.e., the terminal may be situated some 100 m away). Alarm signals are produced when the measure value exceeds either an upper or lower alarm limit. The alarm limits, as well as the number of pulses to be averaged and the strip-chart recorder range are set from the terminal. The measuring function, a simple algebraic function of the three intensities, is also set from the terminal. The recorder signal is also available for process steering purposes. The microprocessor system contains about 6 kbytes assembly software, all in EPROM (2764). Despite the large volume, the program structure is very simple, as can be seen in Fig. 5.

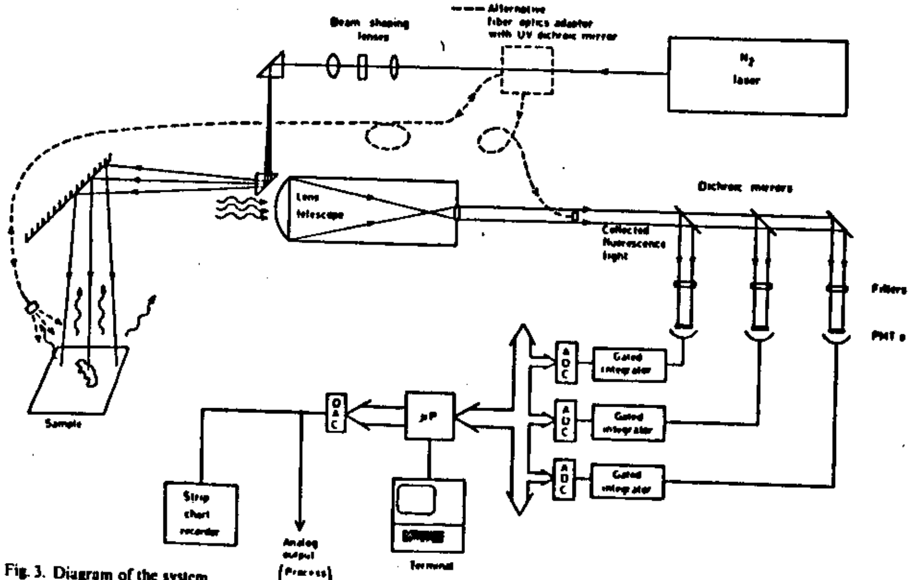

Fig. 3. Diagram of the system

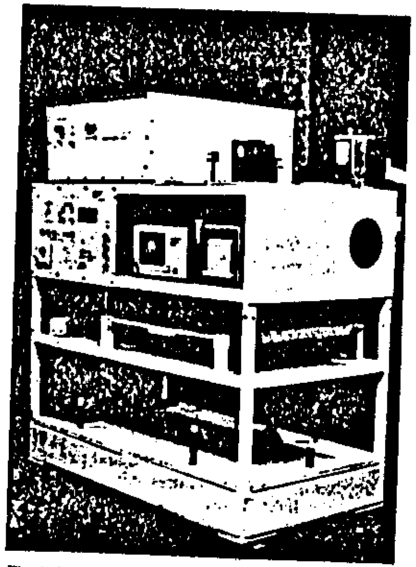

Fig. 4. Photograph of the system

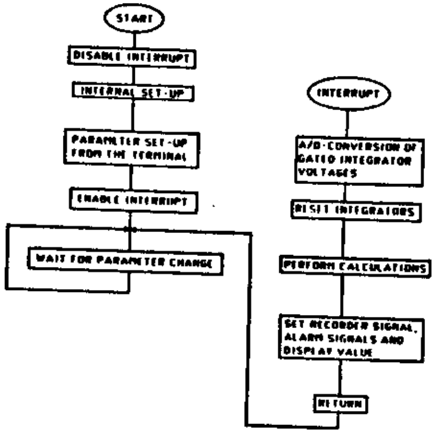

Fig. 5. Flow chart of the microprocessor program

In addition to the home-built laser two commercial sealed N_2 lasers have been tested with the system, a Laser Science Model VSL-337 and a PRA Model LN 250. The lower power of the VSL-337 (120 μJ vs. 250 μJ for the LN 250) has not been a problem in the measurements performed so far, but the greater command jitter, 100 ns vs. 10 ns for the LN 250 has, since this affects the gate pulse timing.

2. Measurements

2.1. Laboratory Measurements

In preparation for industrial measurements, spectra of various materials were recorded in the laboratory using the set-up shown in Fig. 6. Here, a Tracor

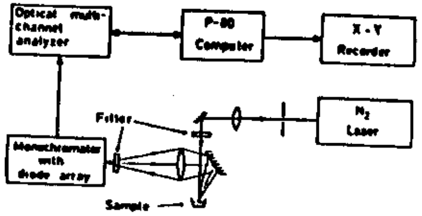

Fig. 6. Laboratory set-up for studies of laser-induced fluorescence

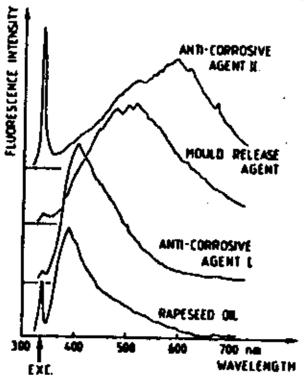

Fig. 7. Fluorescence spectra of different surface films on steel plates

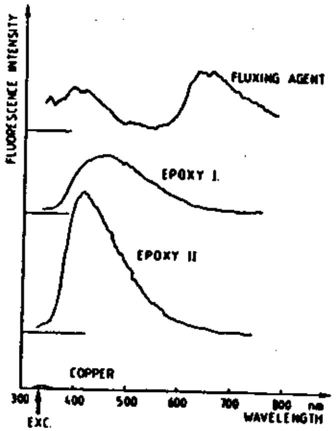

Fig. 8. Fluorescence spectra of interest in connection with inspection of printed circuit boards

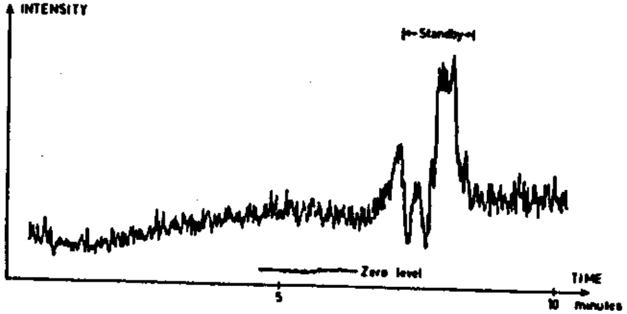

Fig. 9. Recording from the recoiling line at SSAB, Domnarvet, Borlänge, Sweden. Only residues from rolling oil emulsion were left on the steel sheets. In the standby region the coil was removed and the coiler made ready for the next coil

Northern IDARSS (Intensified Diode Array Rapid Scan Spectrometer) with a Model TN-1223-4IG detector in the focal plane of a Jobin Yvon UFS-200 spectrograph was used. The whole spectrum is recorded for each laser pulse and optimum wavelengths and contrast functions for a particular application can be determined. Examples of different spectra are shown in Figs. 2, 7, and 8. As an intensity standard a low-molar solution of Rhodamine 6G in water (70 μg/l) with a particular layer thickness was used. The fluorescence intensity of this standard at its peak wavelength was the intensity unit. It is clear that despite little structure in the spectra, it is easy to find a function of intensities in different wavelength bands that will distinguish between different substances. Extensive work on the study of spectra of various substances has been reported in [3].

2.2. *Industrial Measurements*

An industrial test run was performed at the steel plant SSAB Domnarvet, Borlänge, Sweden, which produces low-alloyed cold-rolled strip steel. The thickness of oil layers was studied at different process lines. The results in Fig. 9 are from a recoiling line where oil residues from rolling oil emulsion are left on the strip steel. The fluorescence light level here is very low and, as can be seen, the noise level is high. Despite this clear differences can be distinguished in the levels for different parts of the coils. Another measurement was done at the end of the continuous annealing line where the steel sheet is oiled for rust protection. Recorder strips from that run are shown in Figs. 10 and 11. At the end of this line there are two coilers. The measurements were performed at one of these and therefore only the oil layer on every other coil was monitored. This is marked in Fig. 10. The sharp peaks in the oil fluorescence (*d*) originate from a stop that was made in order to control the width and thickness of the strip. When the strip was stopped the oil layer grew thicker and when the strip accelerated after the stop the thickness was reduced. On this occasion the oil slushing machine was not computer-regulated as it usually is. The level in the standby region is due to a step in the process when the coil was removed and the coiler was made ready for the next coil. Figure 11 contains a simultaneous recording of the fluorescence intensity and the strip speed at the continuous annealing line. In the five cycles in the figure, the oil slushing machine was computer-regulated in order to keep the oil layer thickness constant within the same cycle. It can be seen that despite this, the oil layer becomes thinner during acceleration and thicker when the strip speed is reduced.

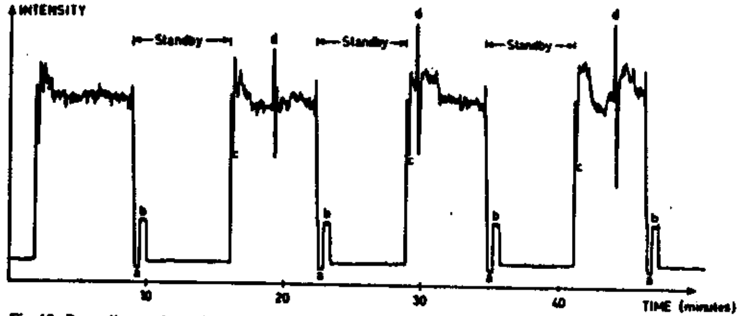

Fig. 10. Recording at the end of the continuous annealing line at SSAB, Domnarvet, Borlänge, Sweden. The coiling of four steel rolls with varying oil film thickness is monitored. At *a* the coil was removed, at *b* the strip guide was placed around the coiler and at *c* the belt wrapper passed the measuring point. At *d* the band was stopped to measure the width and thickness of the strip

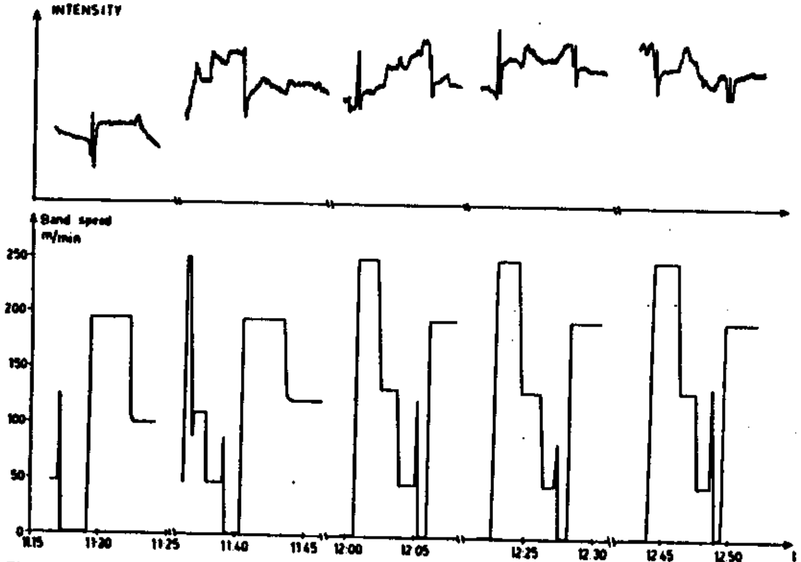

Fig. 11. Simultaneous recording of strip speed and fluorescence intensity at the same measuring place as in Fig. 10. The oil slushing machine was computer-regulated in order to keep the same oil thickness during each of the five cycles shown in the figure

In both cases the distance to the target was about 5 m, the spot size $3 \times 3\,\mathrm{cm}^2$ and the gate width 500 ns. The measurements were performed in full ambient light

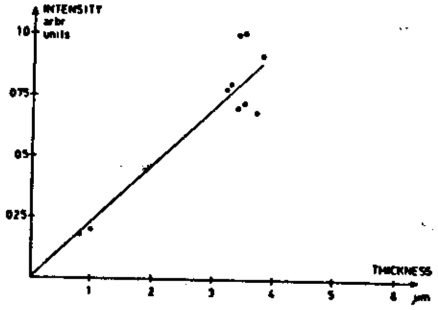

Fig. 12. Intensity vs. film thickness for Quaker Ferrocoat 6828 rust protective oil

(indoors) and in the coiler measurements the target was illuminated with a spotlight in order to monitor the coiler with a video camera from a control station. No influence from this light was seen in the measurements. For calibration in these kinds of measurements, small steel plates, $9 \times 9\,\mathrm{cm}^2$, were weighed with a resolution of 0.1 mg and dipped in different solutions of the oil under study and trichlorethylene. The plates were then allowed to hang overnight so that the solvent evaporated. The fluorescence level was measured with the system on both sides of the plate, the spot size still being $3 \times 3\,\mathrm{cm}^2$. Thus an average value over that spot was obtained. Also a reference signal, as described above, was recorded. The plates were then weighed again. The oil layer causing the difference was supposed to be equally distributed over the plate. The result can be seen in Fig. 12.

Other early tests have also been made to investigate the possibility of characterizing marine oil slicks from an airborne system of this type. In this investigation the target was placed outdoors at a distance of about 70 m. The oil used was a rust-protective oil, which is not relevant since it is not transported at sea. This oil is compared with some relevant oils in Fig. 2. It was found that a pulse energy of about 0.3 mJ was required to get a good signal-to-noise ratio in clear sunny weather with a gate width of 500 ns. In order to get such high pulse energies a frequency-tripled Nd : YAG laser was used. A more detailed description of these measurements has been given in [11].

3. Discussion

Our experiments show that laser-induced fluorescence can be quite useful for industrial monitoring of surface films. A surface monitoring system of the general type described has many other applications than the measurements described above. Apart from the possibility of characterizing oil slicks, it is clear that surface temperature measurements and early tumour detection using HPD are well suited to such a system. A common important feature in such measurements is the non-intrusiveness. The apparatus can be placed at a convenient distance from the target and because short laser pulses are used combined with gated detection, full ambient illumination can be used.

During the work the advantages of monitoring dimensionless functions of fluorescence intensities have been very clearly shown. Apart from the fact that such a function very well may be the best way to enhance the studied phenomenon, a dimensionless function is completely insensitive to fluctuations in laser power, changes in the target surface topography and distance variations.

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