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Ultraviolet Sensors for the On-Line Monitoring of Sterile Packaging Materials*

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Abstract

The importance of human life and the continuing endeavors to enhance human well-being have sparked great interest in the design of sensors and computer-aided on-line inspection systems that critically monitor and control the process of manufacturing sterile medical device packaging materials. In this paper, attention is focused on various aspects of the design of an ultraviolet fluorescence indication sensing system that would be used in the on-line detection, sizing, and automatic isolation of defects in the adhesive-coated sterile medical device packaging material Tyvek? A spectrofluorometric analysis to determine the operating parameters of the system, construction of a two-channel prototype, analog and digital signal-conditioning and -processing techniques, quantitative experiments to determine sensor resolution and sensitivity requirements, and the design of a multielement sensor array to span the Tyvek web are some of the salient aspects of this paper.

Keywords: fluorometry, image processing, material, medical packaging materials, nondestructive testing, optical inspection, quality control, spectrofluorometry, ultraviolet radiation.

INTRODUCTION

Medical devices and medicine need to be absolutely sterile when being used or administered, Imperfect packaging leads to nonsterile medical equipment such as syringes, heart pumps, etc. If such a device is used on humans, serious postoperational complications could result that, in extreme cases, could prove to be fatal. Even if the contaminated packaging is discovered before being used, it has to be condemned, causing great wastage. Heat-seal-coated Tyvek® spun-bonded polyolefin is a packaging material used extensively by the sterile medical device packaging industry because of its strength and reliability. Tyvek serves as a semiporous "breather" window on a nonporous package that contains the medical device. When the package is placed in a controlled ethylene oxide environment, the Tyvek window allows the chemical to permeate through it and sterilize the device. The sterility of the device is then maintained because the Tyvek window is impervious to bacteria and viruses. However, a poor bond between the adhesive-coated Tyvek and the nonporous plastic former would allow bacteria to enter, posing a serious threat to the functionality of the packaging. This poor bond is often due to nonuniform or defective application of adhesive coating to Tyvek, causing patches of missed coating, known as skips, on the Tyvek sheets. It is hence vital to have an on-line inspection system that can make an early detection of defects in coated Tyvek sheets immediately after the coating process and to establish a reliable inspection protocol.

Ultraviolet (UV) radiation is being used in many fields of technology, including germicidal in hospitals, EPROM (erasable programmable read-only memory) erasing, treatment of skin disorders, and quality control of farm produce; it was first introduced in the field of nondestructive testing (NDT) for fluorescent penetrant inspection and magnetic particle testing.[1,2] Possibilities of automated scanning systems and computer-controlled crack-detection systems employing UV radiation were reported at the beginning of the eighties.[3,4] The need for better UV sources and sensors as well as improved standards[5–7] is making UV NDT applications in several other fields extremely attractive.

PRESENT METHODS OF INSPECTION

The present method of inspection of coated Tyvek for detecting skips is visual and is carried out both on-line and off-line. The adhesive coatings used on the Tyvek web possess an inherent fluorescence property from the adhesive's chemical composition, which is special to this adhesive used at Paper Manufacturers Co. Other tagging techniques using chromophores, which provide the adhesive a fluorescence property, can be found in literature? The coating fluoresces in the blue region when excited by a source of UV light (Figure 1). An absence of blue fluorescence in a certain area of the web as noted by the visual inspector denotes a skip. Such an inspection method suffers from the following major drawbacks.

(1) Visual on-line inspection 'of coated Tyvek performed immediately after coating is extremely unreliable, owing to the fact that the coated Tyvek web travels past the inspector at nominal speeds of 10 ft/s and the skips of interest are small. Also, the rated speed of the coating machine is around 20 ft/s. Causing the machine to operate at a fraction of this rated speed drastically reduces manufacturing efficiency. Moreover, because the skips are visible only-under UV excitation, fatigue suffered by the human eye causes a steady deterioration in inspection quality. As a result, smaller skips cannot be detected at an early stage, causing an enormous waste of work hours, machine time, and material.

(2) Visual inspection, when performed off-line on cut sheets' of Tyvek, is more reliable, but is tedious and time consuming, causing a bottleneck in the manufacturing process;

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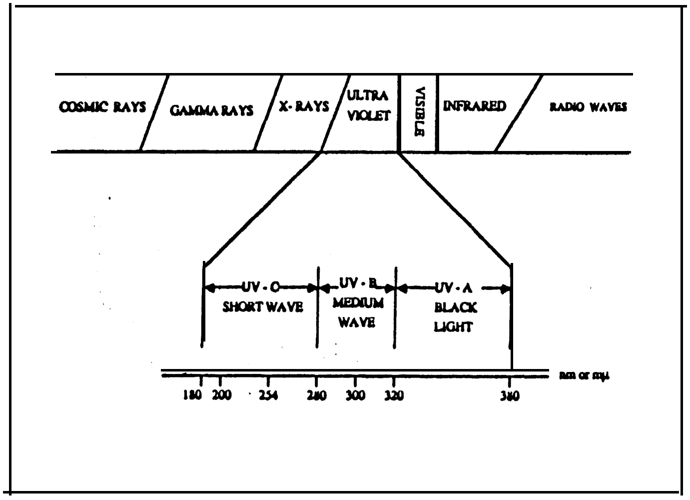

Figure 1—The electromagnetic spectrum and the UV region of interest. [7]

Therefore, a better inspection technique, using sensors to detect the fluorescence property of the adhesive coating, would considerably increase the efficiency of on-line inspection. A computer-aided data-acquisition and digital signal-processing system could be incorporated to achieve on-line monitoring, detection, sizing, classification, and automatic isolation of defects in the adhesive-coated Tyvek. Feedback and feedforward loops could also be introduced to correct such parameters as web speed, air-knife pressure, and drying temperature and to activate a host of emergency protocols, including spray gun markers to isolate defectively coated portions and/or alarm relays. Such a system employing an array of UV sensors was designed at Drexel University and when implemented at the manufacturing plant could result in reliable quality control, cost-effective manufacturing, increased speed of coating, reduced manpower requirements, less machine down time during the defect marking and other procedures, better resolution in defect detection, possible implementation of feedback controls, and defect information archival retrieval. Finally, state-of-the-art procedures build confidence, hence better sales.

Vital design aspects of such a design that are discussed in the following pages include spectrofluorometric analyses of uncoated Tyvek polyolefin sheet, coating formula, and coated Tyvek, respectively, to determine optimum front-end parameters, design of two-channel prototype, quantitative analyses using an on-line simulation system for system calibration, design of sensor array to span the Tyvek web, and final optimization, which includes element-matching based on a common noise floor, threshold selection, and feedback consideration.

SPECTROFLUOROMETRIC ANALYSES

To efficiently employ the same fluorescence detection principle used in visual inspection through an automatic sensing system, front-end parameters such as excitation and detection wavelengths and bandwidths, excitation intensity, and detection sensitivity need to be critically determined. At the chosen operating point, to ensure maximum sensitivity, the emission intensity of the adhesive coating material should be as different as possible from the emission intensity of the uncoated Tyvek. Subtle variations in coating thickness should cause corresponding variations in emission intensity. To choose such an operating point, fluorescence spectrum curves of the adhesive coating as well as those of coated and uncoated Tyvek are required. This gives rise to the need for spectrofluorometric analyses of uncoated Tyvek, coating formula, and coated Tyvek.

Spectrofluorometric analysis tests were carried out using a Hitachi-Perkin-Elmer spectrofluorometer (Figure 2). The spectrofluorometer consists basically of the following.

(a) A broadband xenon light source with a flat response curve from 220 to 780 nm effectively covering our band of interest. The power supply to the light source is regulated to ± 0.1 percent.

(b) The white light produced by the xenon source is led through a 10 nm slit into a tunable monochromator. The monochromator converts the 10 nm beam of white light into a 10 nm beam of light of any desired wavelength from 220 to 780 nm. This beam is then made incident on a transparent quartz cuvette containing the specimen.

(c) Another 10 nm slit leads the fluorescent light from the specimen to a tunable spectrometer. The spectrometer gives an analog intensity reading in $\mu W/cm^2$ for any wavelength from 220 to 780 nm. In the emission scan mode, it gives the fluorescence spectral response of the specimen. This response can be plotted on an xy recorder, with emission wavelength on the x-axis and emission intensity on the y-axis. For the present analysis, every Tyvek specimen was excited at 250, 300, and 350 nm, and a spectral curve was obtained in each case.

Experimental Details

In conventional fluorometric studies, the specimen is in the form of a solution. In our study, the problem of having to test a planar solid specimen was overcome by use of a 90 degree spectrometer. The Tyvek specimen was placed on the diagonal of the cuvette. The angle of incidence was 45 degrees. Fluorescence was measured at a 45 degree angle. A real factory situation, where reflected as well as fluorescent light would be encountered, was thus created.

As a model analysis, it would be informative to go through the curves obtained for the following as a set: (1) uncoated Tyvek, (2) a solution of the coating formula, and (3) Tyvek coated with the coating formula.

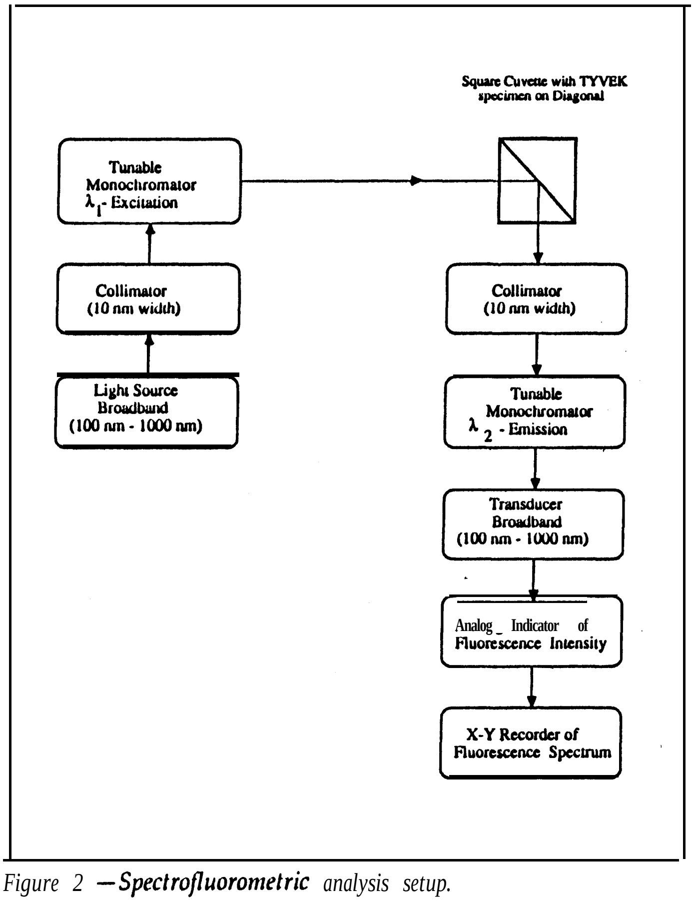

Figure 2 —Spectrofluorometric analysis setup.

Graph 1 (Figure 3) shows the response curve of uncoated Tyvek excited by a UV source of wavelength 300 nm. A good percentage of the incident light is reflected, and this appears in the spectral response curve as a band with peak wavelength 300 nm. Toward the right, we see that the uncoated Tyvek fluoresces at a band of center wavelength 580 nm.

Graph 2 (Figure 4) shows the response curve of a coating solution. Again, a lot of light is reflected, appearing as a band with peak wavelength 300 nm. In addition, the coating formula fluoresces in a region of center wavelength 410 nm. The wavelengths corresponding to the upper and lower 3 dB amplitudes are 390 and 450 nm, respectively, and the bandwidth wavelength is 60 run.

Graph 3 (Figure 5) shows the curve for Tyvek coated with the coating formula. Band 1, with center wavelength 300 nm, is obviously due to reflection. Band 2, with center wavelength **410 nm,** apparently is due to the coating, and band 3, with center wavelength 580 nm, apparently is due to Tyvek. The peak amplitude of band 3, however, has noticeably reduced. This can be accurately assumed to be due to the presence of a coating over the Tyvek **sheet.**

Fluorescence spectra for excitation wavelengths of 250 and 350 nm, which were also produced, are not shown here. From the **fluorescence** spectra shown, it could be easily seen that, for the same excitation wavelength of 300 nm, the coated Tyvek **sheet** fluoresces at a center wavelength of 410 run because of the coating, whereas the uncoated Tyvek sheet fluoresces at a wavelength of 580 nm. Thus, using a sensor that is critically sensitive at a center wavelength of 410 nm, it is possible to differentiate between skips and coated Tyvek. Furthermore, a ratio of the peak amplitudes of band 2 and band 3 could possibly give an indication of the thickness of the coating.

The principal results of the spectrofluorometry, which yield the front-end parameters, are summarized as optimum excitation wavelength, 300 nm; optimum excitation intensity, 2500 μW/cm² at 15 cm; optimum detection wavelength, 405 nm; and optimum detection range, **10–1900 μW/cm².** These results

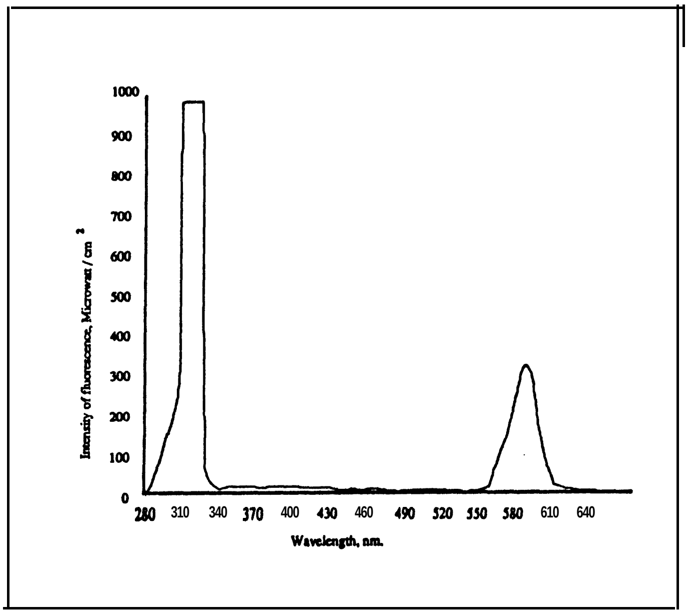

*Figure **3—Spectrometric** response **from** uncoated Tyvek when excited by 300 nm radiation.*

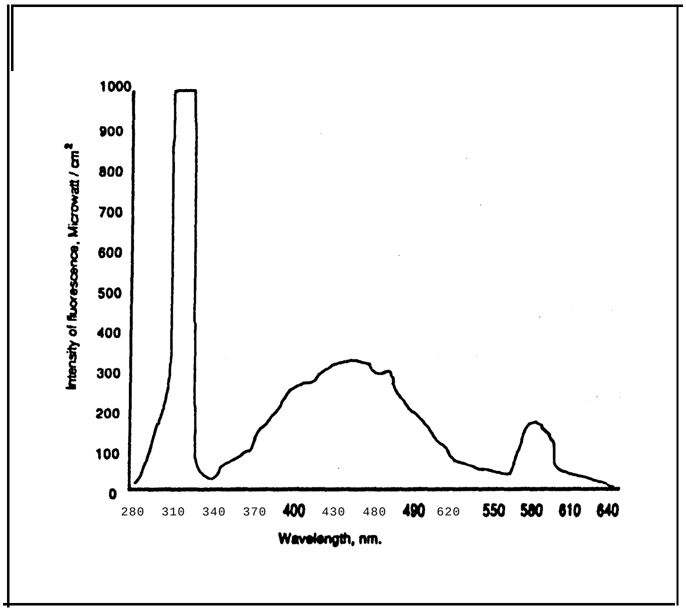

*Figure S-Spectrum obtained **from** coated Tyvek at 300 nm excitation.*

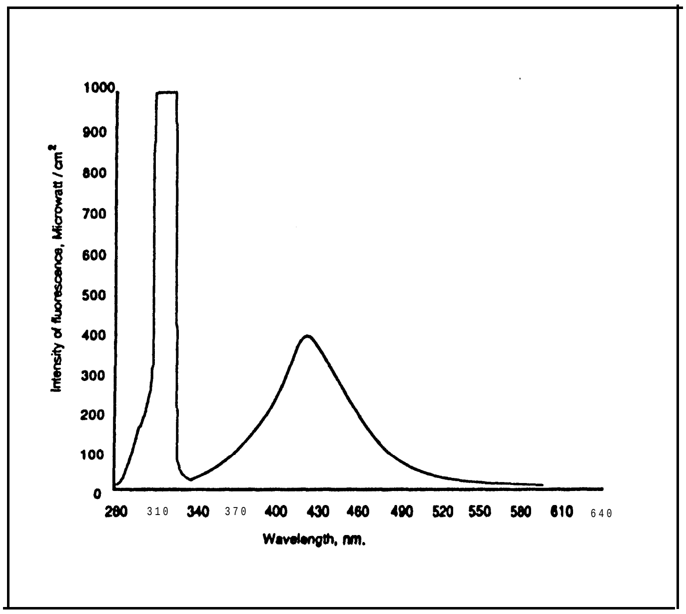

Figure 4-Spectral response of the coating solution when subjected to 300 nm excitation.

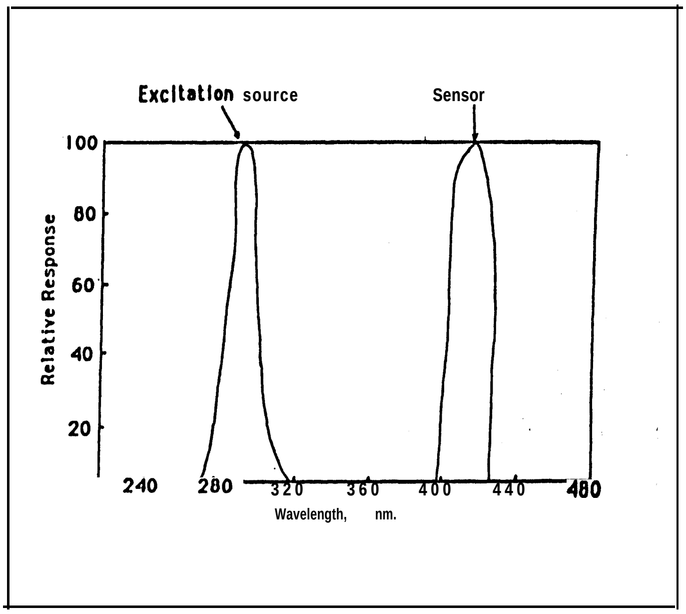

*Figure **6—Selected** responses for the excitation source and the receiving sensor.*

are biased by factors like commercial availability, price of components, etc. Figure 6 shows the spectral response curves of excitation source and sensor.

CONSTRUCTION OF A TWO-CHANNEL PROTOTYPE

The schematic diagram of the computer-assisted **UV** data-acquisition system is shown in Figure 7, The excitation source consisted of a fluorescent tube lamp using suitable filters with a peak wavelength of 302 nm and a 3 dB bandwidth of 10 nm. With a 15 min warmup, the lamps produce an intensity of 2500 $\mu W/cm^2$ at a distance of 15 cm from the geometric center of the lamp surface. The active sensor element, made of a special semiconductor material, had a center wavelength of 405 nm with a 3 dB bandwidth of 20 nm. Salient features include signal-conditioning hardware, which among other things provides linear amplification and ambient light compensation to offset quiescent readings due to external light. An adaptive digital filter algorithm to reduce any environmental/line noise was incorporated into the software, taking into account the speed of the web, so as to stay clear of filtering off small skip signals whose pulse width and frequency content vary according to the speed. The unfiltered raw signal from a typical skip in the coated Tyvek is shown in Figure 8. Any suitable threshold as indicated in the graph should discriminate the defective zones in a go/no-go inspection protocol. Position and velocity information were gathered from an optical encoder that had a linear resolution of 0.025 in. (0.64 mm). A PDP 11-23 computer with an ADAC 30 kHz, 16-channel, ND converter in its Q-bus was used to collect, store, and process data. Other peripherals included a hard disc that was used as a mass data storage device, a graphics terminal, and a graphics printer.

QUANTITATIVE ANALYSIS FOR SYSTEM CALIBRATION

An on-line simulation system that was a scaled-down version of the actual coating machine was used to determine the following.

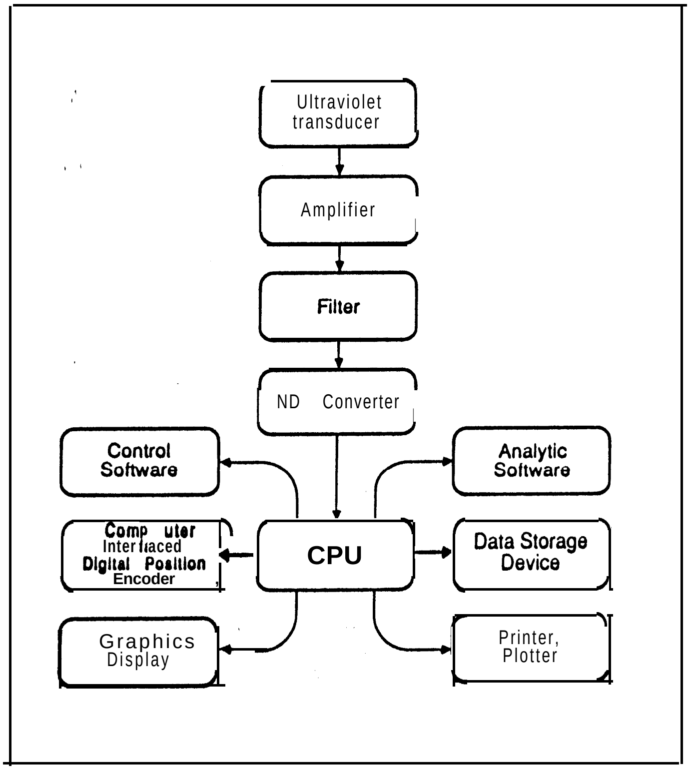

Figure 7—Schematic representation of the two-channel prototype system implemented at the manufacturing plant.

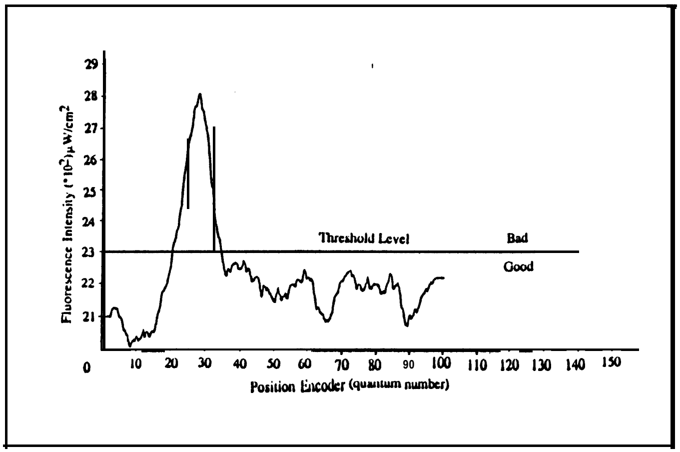

Figure 8-A typical result showing the sensitivity of the sensors to skips in the coated Tyvek.

(a) Transducer resolution, lateral and polar, at known speed: Calibration Tyvek specimens with tailor-made skips (portions with missing coating) (Figure 9) were used to determine transducer resolution capabilities; it was concluded that skips of widths down to 0.25 in. (6.4 mm) could be reliably detected at web speeds of 6 ft/s with transducer distance of 1.5 in. (38 mm) from the specimen. A sample result is shown in Figure **10.**

(b) Transducer time-response characteristics: The time-response characteristics of the sensor were slowed down by negligible amounts due to the lag network in the amplifier-filter circuit. It was found more than adequate for the web speeds of 10 ft/s commonly used.

The two-channel prototype as implemented on the factory floor adhesive-coating machine can be seen in Figure **11.**

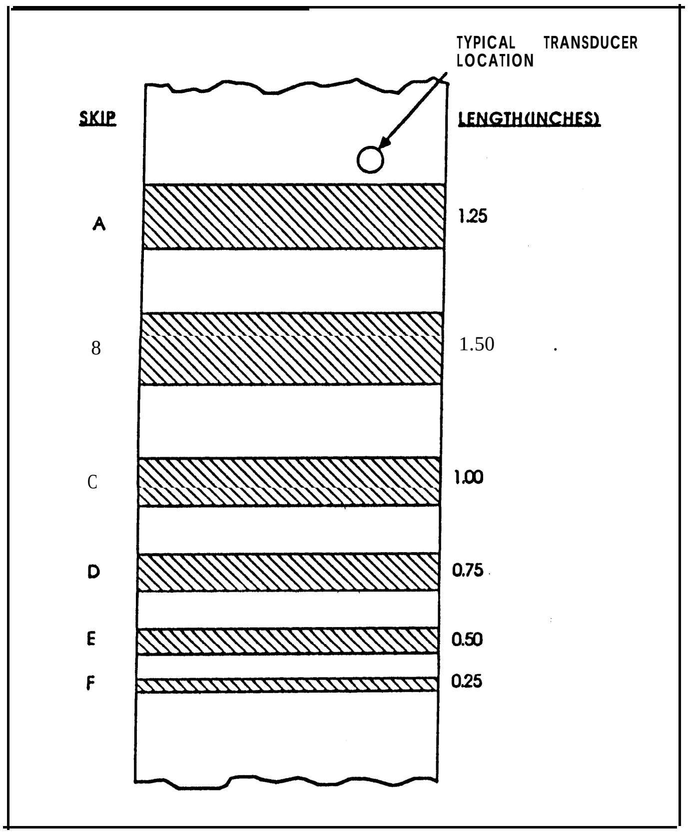

Figure 9—Diagram of the skip calibration Tyvek sheet used. 1 in. = 2.54 cm.

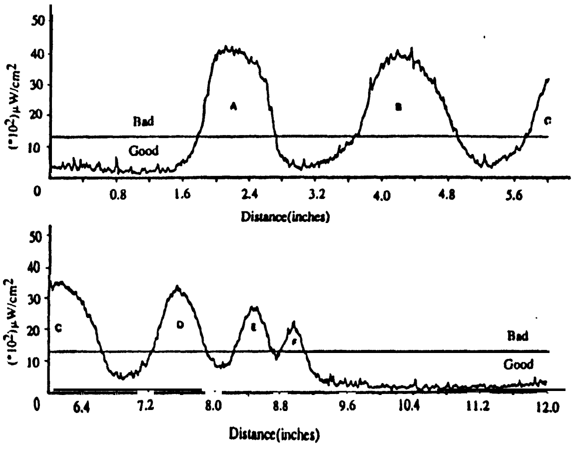

Figure 10— Result obtained from the calibration sheet, showing excellent resolution of defect identification. 1 in. = 2.54 cm.

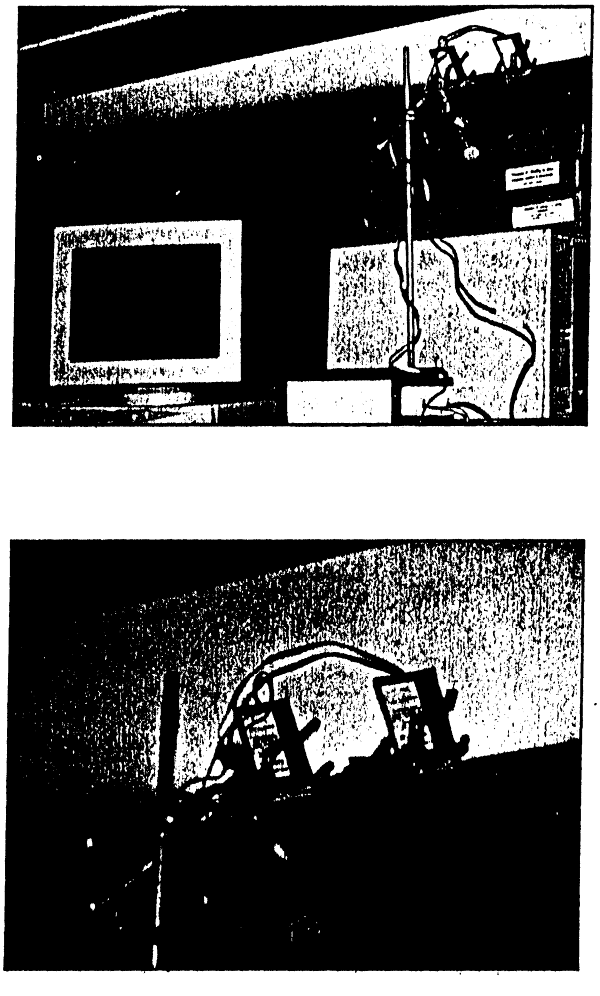

Figure 11 —Photographs of the two-channel, UV prototype system: (a) as implemented on the on-line coating machine, (b) as the UV sensors monitoring the coating fluoresce.

DESIGN OF SENSOR ARRAY TO SPAN THE ENTIRE TYVEK WEB

In designing a sensor array that would serve as the front end of the on-line inspection system, the following design requirements were taken note of: minimum size defect that needs to be resolved, Tyvek web size that needs to be covered, and budget constraints.

Using data obtained from quantitative experiments conducted using a single sensor element and a calibration skip specimen, the following performance parameters were taken into account: resolution (lateral) capabilities and sensitivity.

The following design parameters were arrived at: distance between sensor face and Tyvek roller, width of web segment covered by each element, number of elements in array, and cost of building and erecting sensor array.

Assumptions include the following: (1) the minimum skip width that needs to be resolved is 0.5 in. (12.7 mm). (2) The width of the Tyvek web is 54 in. (137 cm). (3) The price of the array is to be kept within reasonable limits.

Overall design considerations are outlined next. First, the individual array element has a Lambertian (cosine) response profile.[5] Second, the intensity of fluorescence from a skip element, impinging on the sensor face, is inversely proportional to the square of the distance separating them; i.e., if I, is the intensity recorded by the sensor from a skip surface element at a distance a from its face, then the intensity I_b recorded from the same skip surface element at a distance b from its face, keeping the angle of impingement constant, would be given by

$$I_b = I_a \times (a^2/b^2).$$

Third, the component of the skip element of area A that faces the sensor is given by $A \cos \phi$.

From trigonometry, we see that the fluorescence recording from a skip element at location c would be only $\cos^4 \phi$ times the recording from a similar skip at location a. From Figure 10, showing the sensor response to a rectangular calibration skip of width 0.5 in. (12.7 mm), we see that the best threshold would be at -6 dB from the maximum intensity. Therefore, the maximum permissible angle of impingement for reliable coverage would be given by $\cos^4 \phi$ = 0.5 (-6 dB), which yields a ϕ value of 33 degrees. With the sensor array at an optimum distance of 1.5 in. (38 mm) from the web, the width covered by each point on the sensor face = 2 × 0.93 in. (23.6 mm), or 186 in. (47.2 mm), Taking into account the 0.5 by 0.5 in. (12.7 by 12.7 mm) area covered by the sensor face, the 6 dB spatial width of every sensor element is approximately 2 in. (5 cm) and the width of the web is 54 in. (137 cm). Therefore, the total number of elements required is 54/2, or 27 elements.

If we choose a 24-element array, the angle that will have to be covered by each sensor element for the same web width would be 2.25 in. (57.2 mm) which converts to a – 7 dB spatial width; this is tolerable, although it still causes a deterioration of array sensitivity. The complete system is visualized as in Figure 12, where an ink squirter is proposed for marking the defect locations. Several add-ons such as optical storage devices for information archival, feedback controls for automa ted monitoring of all manufacturing parameters, etc. can be integrated into the system at a later stage.

ACCOMPLISHMENTS

Some of the pertinent accomplishments of this project can be enumerated as shown below:

- The extension of UV technology into quality monitoring of adhesive coatings was demonstrated.
- The potential of nontraditional NDE techniques was well established.
- Use of spectrofluorometric analysis in determining critical source and sensor parameters was shown.
- A prototype two-channel UV monitoring system was developed and successfully tested both at the research facilities and on the on-line coating machine on the factory floor.

- On the basis of several parametric studies, a complete 24-channel system was designed and many suggestions have been made.

LIMITATIONS AND FUTURE DIRECTIONS

One of the primary limitations of the current technique for a continuous manufacturing operation is the difficulty in obtaining narrow-band UV filters that do not degrade with time. The use of glass interference filters is a possibility that could avoid the periodic replacement and recalibration procedure required with the commonly available interference filters. It has also been observed that the amplitude of the reflected signal from the coating is sensitive to the presence or absence of the coating. Hence, future work in trying to use this reflectance property for detection of skips in coatings is recommended. A microprocessor-based real-time system programmed to activate abnormality protocols -including the activation of alarms, spray guns to isolate defective sections of Tyvek web, expert algorithms to microevaluate individual skips for size and macroevaluate the entire bale for coat weight-is being built for possible commissioning at the manufacturing plant. Parameters like web speed, air-knife pressure, drying temperature, etc. could then be studied for their effects on the system. Adhesive coating properties like strength, cling resistance, and coat-weight consistency could also be evaluated to study the possibility of incorporating corrective feedback control in the system.

Acknowledgments

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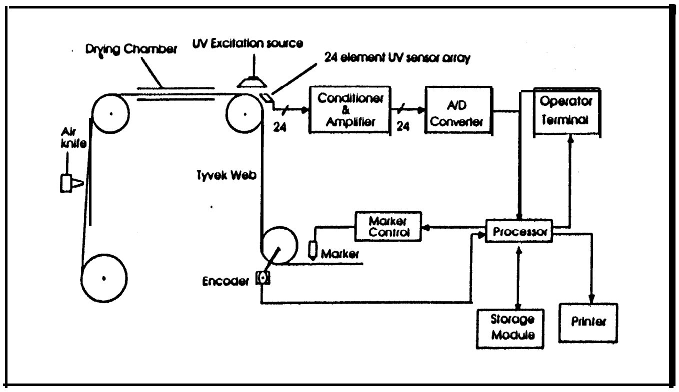

Figure 32 — Diagram envisioning the final 24-sensor array system with a spy gun used as marker.

Authors

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Krishnan Balasubramaniam is a Ph.D. graduate from Drexel University, Philadelphia, PA, with extensive experience in the field of nondestructive evaluation. He worked on NDE problems in anisotropic and layered media for four years during his Ph.D. thesis research. He also has extensive working knowledge in signal processing and feature-based analysis, which find application for signal enhancement and defect characterization in modern materials. Balasubramaniam has more than 30 publications in the field of NDE and has presented his work in both national and international meetings. He has reviewed technical publications for the *Journal of Adhesion*, *Materials Evaluation*, and the *Journal of Engineering and Materials Technology*. He has consulted for many companies including General Electric, Teledyne, Air Products, Grumman Aerospace, Sea Land, etc. and has handled research programs at Drexel University sponsored by Electric Power Research Institute, Naval Research Laboratory, Aerospatiale (France), and Nortec.

Joseph L. Rose, the Soffa Mechanical Engineering Professor at Drexel University, Philadelphia, PA, is the director of a center on IMPAQT (Intelligent Manufacturing, Processing and Quality Technologies). He is a consultant with such companies as General Electric Co., FBS, Inc., Staveley NDT Technologies, and the United Nations Industrial Development Organization. He has received five special awards, including the 1973 ASNT award for best paper, 1982 Drexel University Research Achievement, 1986 Lindback Teaching, and 1986 ASNT Tutorial Citation. He is author of four patents, one textbook, and over 150 articles on ultrasonic NDE, wave mechanics, medical ultrasound, weld inspection, adhesive bonding, and composite material inspection. Rose is a member of ASME, IEEE, ASNT, SEM, SME, ASM, and ASA.

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Authors

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Gina R. Caudill was born in Caracas, Venezuela. She is a materials engineer at the Naval Aviation Depot (NADEP), Norfolk, VA. Having worked in the NDI/T field for the last six years, using the five major methods for applications on naval aircraft and engines, Caudill has had a unique opportunity to bring her past experience to the NADEI? She has worked as a quality assurance coordinator for the Jonathan Corporation Shipyard at Norfolk, VA. As a metallurgical engineer in the Materials and Process Engineering Div. at McDonnell Douglas Aircraft Co., Tulsa, OK, she worked with Navy and Air Force aircraft. Caudill attended the Colorado School of Mines, Golden, and received a B.S. degree in metallurgical engineering from the South Dakota School of Mines and Technology, Rapid City, where she was recognized by *Who's Who Among Students in American Universities and Colleges*.