Verifying UV Exposure in 3-D Curing Systems

R. W. Stowe, Fusion UV Systems, Inc., USA

Abstract

As larger and more complex objects are candidates for UV-curable coatings, the challenges of exposing curable surfaces to adequate UV energy become greater. It is desirable to position UV lamps for the most effective exposure, and the least wasted energy. Because complex parts differ from one another, and paint line organization varies, lamp configurations unique to each line or part type may be necessary. Techniques of radiometric verification of UV exposure to all complex curable surface areas are explored, including radiochromic films.

Introduction

3-D Processing presents some new and different problems for radiometry. Parts have complex surfaces, so the irradiance levels will vary by location. For optimized lamp positioning and process verification, this could require irradiance and energy measurements at almost every point on the surface. The motion can range from the straight-through linear travel of a paint line past a fixed set of lamps, to compound motion of chain-on-edge conveyors, to combinations of part motion and limited lamp motion, and to totally robotically-controlled motion of lamps themselves. The exposure (irradiance profile) at any point will result from the combined effects of part geometry, relative surface velocity, and lamp configuration.

Steps in the 3-D design process:

1. The coating is characterized in its response to UV Exposure variables – this yields the maximum and minimum exposure required by the coating. This step is done with flat, linear processing – in the lab. Radiometry is used to *quantify* the exposure specifications (irradiance, profile, wavelength, and temperature) and to evaluate the optimum or minimum exposure required for a photo-curable material to develop its ideal properties. The exposure conditions must be within the range achievable by a production system.

2. The mechanics of the line are identified – degrees of motion, surface velocities, lamp organization, total power, etc., and lamps are positioned for maximum effectiveness.

3. Radiometry is used to verify the process design. Dry parts are instrumented with radiometers (or dosimeters) to verify that the exposure is within specified limits on all surfaces. The spectral exposure (wavelength distribution) must be the same as used in the development phase (step 1). It is often difficult to use the same instruments that were used in the laboratory. This raises serious issues of measurement with different instruments.

4. Finally, radiometry is used to monitor the process over time.

The most important principle of effective radiometry is that the measurements must be <u>relevant to the process</u> or, in other words, must be related to the development of the physical properties of the final product. By thoroughly understanding the lamp-chemistry-application interactions, more precise and useful specifications can be determined for <u>what</u> to measure in

the design of a process and for the establishment of meaningful limits that can be applied to process monitoring. In addition, data from radiometers must be communicated in a consistent and uniform way. This facilitates the duplication of the UV exposure conditions which produce the desired curing result, and is also important in the event that problem-solving communication between R&D, production, QC, or suppliers is necessary.

A wide variety of radiometric instruments is now available for measuring the radiant characteristics of industrial and laboratory UV lamps and curing systems. Relating these characteristics to the performance of a UV-cured product depends on how well the selected parameters match the critical factors of the cure process. Because of the significant differences in measurement equipment, the specific instrument(s) used to report data must be clearly identified in order to specify or reproduce the required cure (exposure) conditions.

UV Exposure: Irradiance, Spectral Distribution and Energy

There are four key factors of UV exposure that affect the curing and the consequent performance of the UV curable material. Simply stated, these are the minimum exposure parameters that are required to sufficiently define the process:⁽¹⁾

- **irradiance** -- either peak or profile of radiant power arriving at a surface, measured in W/cm² or mW/cm²;
- spectral distribution relative radiant power versus wavelength in nanometers (nm);
- time (or 'speed') energy is the time-integral of irradiance measured in J/cm² or mJ/cm², and
- **infrared** (IR) or heat usually observed by the temperature rise of the substrate, °F or C. (A non-contacting optical thermometer is recommended for surface temperature measurement).

Irradiance data must <u>always</u> include identification of the <u>wavelength range</u> to which it applies. This is one of the most common omissions in radiometry. When irradiance is measured in any specific range of wavelengths, it is called *"effective irradiance."*⁽²⁾ When this wavelength range is clearly understood, the term *"irradiance"* is sufficient. ("Intensity" is not a technically defined term, but is commonly but improperly used to mean irradiance). Peak irradiance has a distinct effect and benefit on speed and depth of cure.⁽³⁾ Irradiance levels in 3-D curing are typically much lower than in flat linear curing.

UV Effective Energy is sometimes loosely (but incorrectly) referred to as "dose," For an exposure in which irradiance is not constant, such as rising then falling, it is the time-integral of irradiance. This is the total UV energy to which a surface is exposed as it travels past a lamp or a sequence of lamps. Effective energy density incorporates irradiance profile, the wavelength range of interest ($\lambda_1 \rightarrow \lambda_2$) and time:

$$E_{(\lambda_1 \to \lambda_2)} =_{t_0} \int^{t_1} I_{(\lambda_1 \to \lambda_2)} dt$$

As with irradiance, when the wavelength range is clearly stated, and it is clear that the meaning is "per unit area," this term can be simply abbreviated as "energy."

Information about irradiance or of the entire exposure *profile* is important to the design.

The fact that different irradiance profiles can produce different physical properties in most UVcurable materials is the reason that profile information is needed in the process design stage. The exposure *profile* is characteristic of any lamp design and, in multi-lamp 3-D applications, the lamp positioning and organization.

A measurement of total UV energy is a <u>composite</u> of irradiance profile and velocity, but information about neither irradiance, profile, nor time can be extracted from it. Consequently, data on energy alone is less important to design, but can be useful in monitoring or control.

Radiometric Instruments and Devices

In selecting radiometric instruments, there is a variety of choices of types. Usually, an important factor is simply if the instrument or device is compatible with the process equipment. Another important determination is whether the instrument measures the proper exposure parameter.

Radiometers measure *irradiance* (usually watts/cm²) at a point, but over a uniquely defined wavelength band. Differences in detectors, filters, construction, and principles of operation result in the fact that different narrow-band radiometers give different results when measuring broad-band sources. A radiometer from one manufacturer can report significantly different UV data from another instrument from a different manufacturer. This is because instruments have different *responsivity*, or wavelength sensitivity. Also, instruments differ in their spatial sensitivity (angle of view), although most have diffusers to give them an approximate cosine response. As a practical matter, many users prefer to compare data from instruments only of the same type.

Dosimeters measure accumulated energy at a surface (watt-seconds/cm² or joules/cm²), also over some uniquely defined wavelength band. There are electronic and chemical types. Many electronic integrating radiometers will also calculate energy. Because this is the only measurement that incorporates *time* of exposure, it tends to be commonly used.

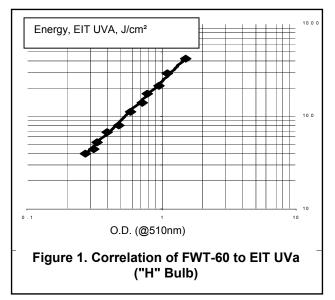
"Mapping" Radiometers Some of the most dramatic adaptations of radiometers for UV processing are sampling radiometers with on-board memory. After a test exposure, the instrument is connected to a device -- either a computer or a dedicated processor -- to display the entire exposure profile. These instruments can also calculate peak irradiance and energy. Single-band and multiple-band instruments are available.⁽⁴⁾ Since these record the "history" of a pass under lamps, they can provide data on the irradiance profile of each lamp in rows of lamps. Relating the time scale to distance requires only the knowledge of the precise speed of the measurement.

Spectroradiometers are very narrow-band instruments, essentially responding to spectral irradiance, and are highly wavelength-specific -- some with resolution as fine as $\frac{1}{2}$ nanometer. These instruments -- actually miniature monochromators -- can be valuable when there is a need to evaluate irradiance in a selected wavelength band of interest, but they don't measure time-integrated energy. Recent developments in these instruments include the ability to *select* a specific wavelength band for easier evaluation of the spectral distribution of a lamp output or spectral irradiance.⁽⁵⁾

Radiochromic dosimeters are tabs that attach to a test surface and respond to total time-integrated energy by changing color or by changing optical density. Depending on the chemistry of the detector, it can change permanently or only temporarily. These photochromic detectors typically respond to a wide range of UV wavelengths. Tabs or tapes that are interpreted by eye or by comparison to a printed color chart are considered less accurate and less repeatable than films read by instruments (colorimeters or densitometers).

They can be very handy, especially for 3-D objects, as a number of them can be placed about the object to measure and compare the energy delivered to any part of the surface. For flat curing, tabs and strips have the obvious advantage that they can be attached to a flat web or sheet and can survive transit through nips, rollers, and the like, without damage. They are inexpensive and easy to apply.

A drawback to radiochromic films is that they generally respond to and record accumulated energy only. In a multiple lamp system, they cannot distinguish the individual exposures of successive lamps. Commercial radiochromic films are not wavelength-specific. In fact, very little spectral responsivity data is available. Radiochromic chemistries tend to respond to short UV wavelengths, typically from 200 up to 300 or 350 nm. Some preparation has to be done in order to correlate the results of these films with either radiometer measurements, or physical properties, or both. Figure 1 illustrates the correlation of tabs whose optical density (at 510 nm) has been correlated specifically to an EIT PowerPuck[®] radiometer.



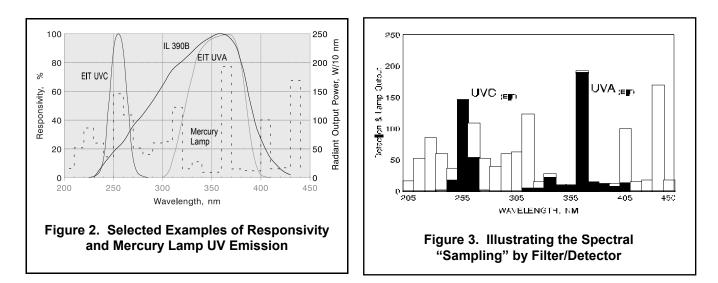
This type of correlation must be done for each specific exposure (type of bulb and spectral distribution). Once done, the correlation can make quick work of multiple measurements.

This suggests that these can be very effective for use in process monitoring or in evaluation of configurations in process design. Radiochromic films can be helpful in the design of a system in the specific task of physical arrangement of lamps in, for example, surface curing of 3D objects. With more development in the area of responsivity and spectral calibration, radiochromic coatings and films could become a useful process control tool.

Responsivity

Typically, the generally accepted UV range designations are: UVC 200-280 nm UVB 280-315 nm UVA 315-400 nm A recent addition to these ranges is "UVV" (400-450 nm), owing to interest in longer-

wavelength curing. It should not be confused with the designation "VUV" or Vacuum UV (100-200 nm). The amplitude of response of a detector to different wavelengths is referred to as *responsivity*. The design of the instrument, cell type and filter results in a singular response curve. The net response curve, in percent of maximum response, is called its *relative spectral responsivity*. Examples of response ranges of two commercial instruments is shown in Figure 2.



Spectral responsivity is the characteristic that differentiates wide-band radiometers from narrow-band radiometers. It can be easily seen, from Figure 2, that any of these yield very different measurements when exposed to the <u>same lamp</u>! Further, it should be noted that, at best, the data reported for any band is a *sampling* of the spectral power in that band. This is illustrated in Figure 3.

The manufacturer's designations for the band of the examples illustrated in Figure 2 are:

EIT, Inc.	EIT UVC ⁽⁶⁾	240-260 nm	(50%)
International Light	IL 390B ⁽⁷⁾	250-400 nm	(10%)
EIT, Inc.	EIT UVA ⁽⁶⁾	320-390 nm	(50%)

All instrument manufacturers provide the responsivity data for their instruments. It should be noted if the manufacturer uses the 50% or the 10% response for the designation of bandwidth (both are illustrated in Figure 2).

Measurements Outside of The "Band of Interest" or Correlating Different Radiometers

Either of these can be a particular problem when the material exposure specifications or the lab development measurements were made with different instruments. Dynamic (traveling) instruments typically used in the laboratory with flat samples may not be easily attached to complex surfaces of the production parts. It is not unusual to see these instruments taped to production parts in an effort to acquire measurements in full scale.

The use of radiometers with different responsivities to measure complex light sources is

a classic technical problem. A correlation can be made between different instruments, but it will be valid <u>only</u> if the measurements are made under <u>exactly</u> the same lamp and spectral distribution. Even then, differences in calibration, and spatial response can cause inaccuracies. There is no easy way to correlate different radiometers (responsivity) for different lamps (spectral emission). Simply proportioning the measurements is not valid.

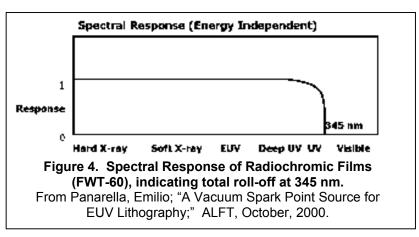
Another common difficulty is evaluating out-of-band data. For example, If the actual spectral range of interest is in the "UVC," but the instrument used measures only in the "UVA," is it possible to deduce the energy in the "UVC" range? This can be done, but requires very specific information on the spectral irradiance from a lamp and the precise responsivity of the instrument. Figure 2 illustrates the principle, which involves creating a transfer function derived by mapping the active radiometer's response curve on the spectral irradiance, and proportioning to the measurement data from the active instrument. In this way, the effective irradiance in another defined wavelength band can be calculated. Because spectral irradiance is determined by a combination of the lamp's spectral emission and reflectivity, it is emission must be either calculated from manufacturer's data or measured with a spectroradiometer.

Consequently, it is common practice to use instruments from the same manufacturer that have the same responsivity, spatial response, and calibration.

Some Limitations

Few commercial radiometers accurately respond in the 200-240 nm range. This is primarily due to limitations in filter materials used with photo-detectors, and to internal scattering effects in spectroradiometers.

Radiochromic detectors are very responsive to shortwavelength UV, but are rarely calibrated for responsivity in *any* wavelength band -- they typically require correlation to a radiometer. The roll-off (of long wavelengths) should be identified, particularly if the range of exposure of interest is in the UVA or UVV ranges.



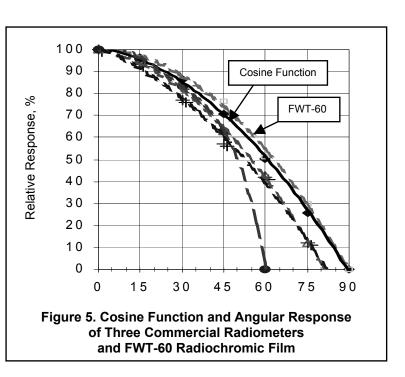
Specific Concerns for 3D Measurements

Reliable radiometry requires an understanding of the errors and sources of variation in radiometers as well as how laboratory radiometric measurements are correlated with production measurements and control.⁽⁸⁾ 3-D processing invariably involves dynamic exposure -- either the part(s) or the lamp(s) -- or both -- are moving. Exposure and radiometry are affected by surface contour and the surface velocity through a field of complex exposure. There are a few radiometer characteristics that can affect the accuracy and validity of surface measurements.

Owing to the fact that large-part 3-D curing is generally accomplished in the far-field of UV lamps, irradiance levels will be far below those encountered in flat, linear processing. Multiple lamps will result in complex divergence patterns of radiation arriving at any point. Finally, a surface may be present an aspect with respect to the source from 90°, which is ideal, to 0°.

For these reasons, radiometers used in 3-D UV exposure must have (1) consistent cosine response, (2) circular symmetry, and (3) a low irradiance threshold.

Cosine Response The angular response of a radiometer is the "weighting" it gives to arriving rays, depending on the angle of incidence. When this weighting is proportional to the cosine of the angle of incidence, $(0^{\circ}$ is perpendicular to the surface), the radiometer is said to have "cosine response." Although arbitrary, there are two reasons for a UV process radiometer to have cosine response: (1) it is an approximation of the "weighting" that occurs naturally in the curing of a film, and (2) it is geometrically defined and reproducible. Figure 5 shows the cosine function compared to three commercial radiometers and FWT-60 radiochromic ("radiachromic") film.



In near-field linear processing (flat curing), the majority of the UV flux is nearly perpendicular to the surface and, in both axes of a tubular lamp, diminishes to nearly zero at $\pm 45^{\circ}$. Consequently any deviation from cosine response causes only small errors. But, in 3-D processing, complex surfaces are not uniformly situated perpendicularly to the source, and any part of the surface may be oriented so that the <u>only</u> flux it receives arrives at an oblique angle, so serious cosine deviation can result in measurement error.

Circular Symmetry The construction of a few radiometers is not symmetrical -- in other words, they do not yield the same measurement for rays arriving from the "north, south, east and west." Although not large, the variation in readings can be as much as 10%. Most dynamic radiometers do not have asymmetry problems, but this is a source of major error with radiometric probes. Combined with cosine response, this characterizes the uniformity of response to rays arriving from any point in a hemispherical space.

Threshold Many battery-powered radiometers conserve power by delaying measurements until they sense a minimum irradiance level -- or threshold. Often, 3-D exposure is in the very low irradiance range -- especially on the "hard-to-reach' surfaces -- and can be in the 50-100 mW/cm² range. A radiometer with a 50 mW/cm² threshold would be subject to serious error.

Users of any radiometer should know its (1) wavelength band or bands, (2) dynamic range, in watts/cm², (3) capacity for recording energy, in joules/cm², (4) sampling rate, if it is a sampling type, and if it reports *instantaneous* peak irradiance or *average* peak irradiance, (5) threshold, and (6) its spatial response.

Conclusion

Radiometry is a powerful analytical tool for UV curing process design, process verification, and invaluable as a QC tool for process monitoring. . It is important to identify the key exposure parameters that have the most significant effect on the performance of the end product. In 3-D processing, radiometry provides the very important step of verifying adequate exposure *before any wet product is run*

For process *design*, it is desirable to evaluate several exposure parameters in order to optimize a process. To evaluate the effects on the physical properties of the final cured product, correlation with exposure variables is essential. These variables can be expressed in terms of irradiance profile, spectral distribution, total energy, and infrared energy (or temperature). Multi-band radiometers, mapping radiometers (to evaluate profile), and spectroradiometers can record information on a significant number of these parameters. In addition to facilitating the optimization process, these measurements are used to determine the operating limits for production process control.

Radiometers are important to process *verification*. Once designed and optimized, the production configuration of parts, motion, and lamps must be verified, typically by attaching instruments or sensors to critical surfaces and recording irradiance profiles and/or energy for each measurement point at the uncoated part passes through the exposure zone. Subsequent monitoring of the process in production may be limited to "surveillance" on only a few key parameters -- those which, when out of pre-determined limits, will affect the result. From process design, these critical parameters were identified. Relatively inexpensive, simple and rugged tools and methods can be used in production monitoring. These may be on-line monitors, dosimeter tabs, single band radiometers, and the like. Ultimately, these measurements must be correlated with measurements of the optimized process from the process design parameters.

Selecting a method of measurement or a particular radiometer should be based on the specific process and the identification of the key exposure variables which have the greatest effect on the process. Care should be taken to avoid errors resulting from inappropriate band selection or from intrinsic deficiencies in the measuring instrument(s).

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