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An integrating sphere-based ultraviolet exposure chamber design for the photodegradation of polymeric materials

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Abstract

The primary method for obtaining laboratory weathering data for a wide range of commercial polymer products including coatings, textiles, elastomers, plastics and polymeric composites is through the use of ultraviolet radiation exposure chambers (UV-chambers). Although numerous improvements have been made in the design of UV-chambers over the last 80 years, the repeatability and reproducibility of the exposure results from these chambers have remained elusive. This lack of reproducibility and repeatability is attributed to systematic errors in their design, operation, and control which, in turn, has prevented comparisons of the performance of competing construction materials exposed in the same environment, comparisons of the performance of the same material exposed in different laboratories, and the comparison of field and laboratory results. The paper re-examines current UV-chamber designs, discusses possible sources of systematic error associated with these chambers, and describes an innovative UV-chamber design having a basis in integrating sphere technology which greatly reduces the magnitude of these errors.

1. Introduction

Ultraviolet radiation exposure chambers (UV-chambers) are the primary means for generating laboratory weathering data for a wide range of commercial products including coatings, elastomers, plastics and polymeric composites, (collectively these products will be termed *construction materials*) [1]. Over the years, numerous technical improvements have been implemented in the design, construction, and control of these UV-chambers. However, the repeatability and reproducibility[a] of the exposure results obtained from these chambers have remained elusive [2–5].

The repeatability and reproducibility of experimental results are affected by material response variability and experimental and systematic errors. Experimental errors and material variability, which occur in all experiments, are random in nature and can be compensated for through the use of proper experimental designs.

Systematic errors, on the other hand, are uncompensated, non-random sources of error which bias experimental results. Common sources of systematic errors associated with existing UV-chambers include human/machine interactions, high specimen temperatures, non-uniform irradiance over the surface of a specimen, and temporal changes in exposure conditions. The mission of any investigator is to identify, isolate, and reduce these sources of systematic errors. This can be accomplished by standardizing procedures, making changes in existing exposure equipment, or by circumventing these difficulties through alternate UV-chamber designs.

In this paper, known sources of systematic errors are discussed and an innovative UV-chamber, having a basis in integrating sphere technology, which may be capable of reducing the magnitude of these systematic errors, is presented.

2. Current UV-chambers

Commercially available UV-chambers began to appear ca. 1920. Atlas Electric Devices introduced its

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[a] See terminology appendix for definitions.

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carbon arc "Fade-O-Meter" in 1918; several years later, Nelson [7] published and provided preliminary exposure results for a UV-chamber design using a mercury arc lamp in 1922; and Buttolph [8] patented several modifications to Nelson's UV-chamber in 1924. UV-chambers containing fluorescent lamps were introduced at a later date.

UV-chambers basically consist of a UV-light source(s) and a specimen rack. The two most popular UV-sources for photodegrading materials have been the xenon arc and fluorescent lamps [9]. The popularity of carbon and mercury arcs has greatly diminished over the last few decades due to the instability and high maintenance of carbon arcs and due to the "unnatural" wavelengths present in mercury arc lamps.

Both the xenon arc and fluorescent lights are gas discharge tubes and are usually line sources. A gas discharge tube contains an inert gas (e.g. mercury or xenon) within an enclosed glass cylinder which is sealed on both ends by tungsten electrodes. When an electrical potential is imposed between the two electrodes, a plasma is generated between the two electrodes. Emittance from gas discharge tubes is considered to be diffuse.

Most commercial xenon arc UV-chambers are similar in construction to the design proposed by Nelson in 1922 [7]; that is, the line source is vertically positioned in the center of the UV-chamber while specimens are rotated on a rack around the axis of the line source. Fluorescent lamp UV-chambers are constructed somewhat differently due to the high slenderness ratio of the fluorescent tube. In fluorescent UV-chambers, the lamps are positioned horizontally in the center of the UV-chamber with specimens placed in a row along either side of the lamps.

Numerous modifications in UV-chamber designs have been made over the last 80 years. These modifications have been aimed at improving the repeatability and reproducibility of exposure results and include (1) the identification of a more temporally stable ultraviolet light source, (2) the identification of spectral radiant power distribution which more closely approximates the maximum solar ultraviolet radiant power [10–13], and (3) the re-design of the exposure racks within a chamber to improve the spatial irradiance uniformity over the dimensions of a specimen and among specimens. Specific examples of improvements include (1) identification of cut-off filter combinations to remove radiation below 290 nm; (2) the introduction of photopic sensors and feedback-control devices for minimizing temporal changes in the radiant power [14–16] and (3) the introduction of the three-tier exposure racks in xenon arc UV-chambers.

These changes have greatly reduced the variability in exposure results, but they have not resolved issues related to the lack of repeatability or reproducibility of exposure results [5,6]. Hence, further investigation into possible sources of systematic errors is needed.

3. Systematic errors affecting reproducibility and repeatability of current UV-chambers

Common sources of systematic error found in current UV-chambers include human/machine interactions, unnatural exposure conditions, non-uniform irradiance over the dimensions of a specimen and among specimens, the inability to accurately and precisely measure ultraviolet radiation dose, and temporal changes in exposure conditions.

3.1. Human/machine interactions

Systematic errors attributable to the interaction of humans and machines include those related to the operation, maintenance, and calibration of the UV-chamber. Errors due to human/machine interactions can be greatly reduced by standardizing the operation, maintenance, and calibration protocols. Committee ASTM G3 has published several standard practices for operating xenon arc [17] and fluorescent tube UV-chambers [18]. Fischer [6] and Fischer and Ketola [19] have recently reviewed the results of a round-robin testing in which these standard practices have been applied.

3.2. Unnatural exposure conditions

3.2.1. Specimen temperature

Xenon arc lamps are almost always operated at a higher current density flux than are mercury arc lamps because of the smaller cross-sectional area of the xenon atom relative to that of the mercury atom [20]. This high current density flux makes the xenon arc a very "hot source" [21,22]; that is, xenon arcs emit a substantial amount of energy in the visible and infrared regions. As such, the temperature of exposed specimens tends to be high relative to the temperature of similar specimens observed outdoors. Panel temperatures for opaque polymeric materials approaching 60°C have been reported in xenon arc UV-chamber studies (see, for example, Martin et al. [23] and Clark et al. [24]).

For specimens simultaneously exposed in a xenon arc UV-chamber, the temperature of the specimens can vary over a wide range depending on the specimen's spectral absorptivity, which is related to its color. Fischer and Ketola [19] exposed panels made from the same material, but differing in color in a xenon arc weatherometer. The observed panel temperature range for these materials was as high as 30°C. It is known that degradation kinetics is highly temperature dependent; hence, it is not surprising that such a high temperature range makes a comparison of the performance of materials exposed to nominally identical exposure conditions difficult. Preininger [25] and the Association of Automobile Industries and its Working Group [4], for example, have concluded that the largest contributor to the lack of repeatability and reproducibility of xenon arc UV-chambers is the lack of temperature control.

Improved temperature control of UV-chambers can be achieved by removing the primary source of thermal energy, visible and infrared radiation, while maintaining the photolytically effective ultraviolet radiation. This can be accomplished by introducing a heat controlling optical element (e.g. a dichroic mirror) between the light source and the specimen. Dichroic mirrors, for example, are designed to transmit (or reflect) ultraviolet radiation through (or off) the dichroic mirror and onto the specimens while reflecting (or transmitting) the visible and infrared portions to a heat sink. Examples of UV-chamber designs incorporating a dichroic mirror in their construction include those proposed by Berger [26] and Klippert [27]. Alternate heat-controlling optical elements are discussed by Rabek [28].

3.2.2. Optimal wavelength radiation

Ultraviolet radiation below 290 nm does not reach habitable portions of the earth's surface [29,30] and, thus, the presence of radiation below 290 nm may photolytically degrade materials via "unnatural" chemistry [31–33]. Extensive efforts have been made by equipment manufacturers in identifying light sources and light source/cut-off filter combinations having a spectral radiant energy distribution closely approximating the maximum solar ultraviolet radiant energy spectrum [12,13,57]. Although not a perfect match, UVA-340 fluorescent lamps and xenon arc lamps equipped with borosilicate/borosilicate filters appear to be a close approximation to the maximum solar ultraviolet energy spectrum [13].

An alternative approach is to abandon efforts to produce a stable light polychromatic light source which emulates the maximum solar ultraviolet energy spectrum, and, instead, to irradiate specimens with narrow bandwidth radiation. This can be accomplished by placing filters (cut-off or interference filters) in front of the exposed material. Examples of materials degradation studies using this strategy include those described in Buttolph [8], Suga [34], and Trubiroha [35]. This practice has been adopted by the medical and biological research communities [9] and should be given more scrutiny by the materials community.

3.3. Spatial irradiance uniformity

Ensuring spatial irradiance uniformity over the dimensions of a specimen and from specimen-to-specimen is a prime consideration in designing any optical system. Spatial uniformity is needed to determine the spectral ultraviolet radiation dosage received by a specimen. Spectral ultraviolet radiation dosage must be known in order to compare the performance of constructionmaterials [36,37].

Spatial irradiance uniformity is difficult to attain in current UV-chambers due, in part, to the larger surface area over which uniformity must be controlled. Factors affecting irradiance uniformity include reflectances from the specimen and walls of the chamber and physical limitations imposed by the optical system (e.g. the geometry of the light source and the dimensions of the specimens). (Many pigment particles are highly reflective to ultraviolet radiation. See, for example, Wilcock and Soller [38] and Koller [39].)

Spatial irradiance uniformity is often improved by modeling radiative transfer within a UV-chamber. Models, which closely approximate current UV-chamber construction, include those published by Jacob and Dranoff [40], Matsuura and Smith [41], Zolner and Williams [42], Irazoqui et al. [43], and Quarderer and Kadlec [44]. Radiant energy models for other promising chamber designs include the model derived by Cassano and Smith [45]. In general, predictions from the models fit irradiance measurements within the chamber rather well. From these models, the parameters controlling irradiance uniformity include the position, number, and length of the line source(s), the size and geometry of the specimens, and the distance between the line source and specimens. Typical model assumptions include:

1. A single line source located in the center of a cylindrical chamber where the cylindrical chamber acts as a specimen.
2. The length of the line source and cylindrical chamber are equal.
3. The line source is an ideal source; that is, the radiant intensity emitted from each incremental length of the line source is constant and invariant.
4. The line source is a Lambertian radiator; that is, the radiant intensity emitted from each incremental length of the line source is perfectly diffuse and obeys Lambert's law.
5. Reflection from the walls of the UV-chamber is negligible.

Several of these assumptions are violated in current UV-chamber designs. Assumption 1 does not hold for fluorescent UV-chambers, which usually contain a number of fluorescent lights. Assumption 2 does not hold for xenon arc equipped UV-chambers, since the specimen racks and specimens often extend beyond the length of the line source. Quarderer and Kadlec [44] have made irradiance predictions beyond the ends of the line source. Assumption 3 is a good approximation for AC powered line sources, but it is not a good assumption for DC powered line sources. For both AC and DC powered sources, the radiant intensity is higher at the cathode than it is at the anode. For DC powered line sources, the ratio of the emittance from the cathode to the anode can exceed 70 [44,46–48]; whereas, for AC powered lamps, this ratio is closer to two [48]. This difference in radiant intensity may explain why the maximum of the radiant energy profile for DC-powered line sources seldom coincides with the middle of the line source [see, for example, the radiant intensity profiles published in Martin et al. [23] and Clark and Harrison [24]. Finally, the contribution to the total ultraviolet irradiance of light reflecting off the walls of a UV-chamber and off specimens (assumption 5) does not appear to be addressed in current UV-chamber designs.

Radiant energy measurements for fluorescent UV-chambers have been made by Fischer et al. [5] and Fedor and Brennan [16] and have been made for xenon arc UV-chambers by Coblentz et al. [49], Martin et al. [23], Clark and Harrison [24], and Fischer [6]. Comparison of these measurements with predictions from the mathematical models provides insight into possible limitations in current UV-chamber design.

Fischer [6], for example, observed a 30% change in irradiance and in degradation for specimens located at the center and at the ends of the fluorescent lamps; the model proposed by Irazoqui et al. [43] predicted a 40% change. Thus, uniform irradiance over the length of a fluorescent lamp may be physically impossible to achieve.

The three-tier specimen rack design for xenon arc UV-chambers provides the designer with much more freedom to follow the contour of the theoretical radiant energy profile from the light source. Theoretical irradiance variations along the length of a specimen for a new light source and a three tier rack design are less than 2%. Variations in spatial irradiance over the width are also of concern. Two-dimensional radiant power mappings for xenon arc UV chambers, like the mapping provided by Fischer [6] for a fluorescent UV-chamber, have not been found in the literature. Capron and Crowder [50] mapped the spectral radiant flux in the plane perpendicular to the axis of rotation of the xenon arc, however, and concluded that the horizontal distribution of radiation is not uniform in spectral radiant intensity. The fixed rack design for xenon arc lamps is, of course, a significant advance over cylindrical and two tier rack designs. The goodness of the strategy, however, is based on the premise that the spectral radiant flux from nominally identical xenon arc lamps is constant and does not change as a light source ages. Experimental support in the literature for these premises is lacking.

3.4. Temporal variation

The radiant power output from an arc source is notoriously unstable over time. This temporal instability can be attributed to both equipment variables and changes in the light source as it ages (light source plus the filters).

The major equipment variable affecting radiant power output is a change in the electrical current density over time. Radiant intensity from an arc source is related to current density through a power law function ($E=a\ D^b$, where E is radiant intensity, D is current density, and a and b are empirical constants) [44,49,51–53]. The higher the current density, the higher the temperature of the plasma and, thus, the greater the radiant power. Also, the higher the current density, the higher the proportion of the total radiant power emitted in the ultraviolet region [52,53]. Thus, temporal stability of a lamp depends heavily on the stability of the lamp power source.

The optical properties of the arc lamps, filters (in the case of xenon arc lamps), and phosphors (in the case of fluorescent lamps) change with time through an aging process. Temporal changes in the arc light sources have been studied by Coblentz et al. [49], Buttolph [54], Martin et al. [23], Schäfer [22], Clark and Harrison [24], Mullen et al. [55], and Forbes et al. [56]. The tungsten electrodes of all arc sources are designed to operate near the melting temperature of tungsten [20]. Thus, when the lamp is on, tungsten is sputtered off the electrodes and deposited onto the interior walls of the glass cylinder [57]. These deposits reduce the spectral transmittance properties and eventually cause the glass envelope to devitrify and crack [52]. In commercial xenon arc chambers, efforts have been made to minimize this effect by placing a metallic sleeve around the anode.

The glass enclosure surrounding the arc also ages. This can occur whenever the arc plasma touches the enclosure causing it to melt and to deposit silicon onto the interior surface of the glass. The width of a xenon arc is known to thicken with an increase in current density to the lamp [58]. Increasing the current density to the arc is the most common strategy used in stabilizing radiant intensity as an arc source ages.

The quartz and other glass filters surrounding a xenon arc also age through a process called solarization [49]. Solarization is the reduction in transmittance of a filter resulting from the exposure to short wavelength UV radiation and to high temperatures. Decreases in transmittance are usually rapid at first followed by a more gradual decrease in transmission. The greatest transmission loss occurs at wavelengths less than 365 nm [49,57,59]. The effects of solarization may be minimized by pre-aging the filters prior to inserting the specimens into the UV-chamber [57].

Total and spectral irradiance control has been greatly improved through the use of photo-feedback and control devices [14–16]. The radiant intensity of the light source is often controlled at a specific wavelength or over a wavelength band. Decreases in total or spectral radiant intensity are compensated by increasing the current density to the arc source. Increasing the current density seldom results in a uniform change at all wavelengths, however. Instead, the spectral radiant intensity at short ultraviolet wavelengths ($\lambda < 350$ nm) exhibits a much greater change than it does at wavelengths longer than 350 nm. Therefore, correcting the radiant intensity at a particular wavelength or wavelength band does not return the spectral radiant intensity of the light source to its original spectrum; that is, the spectral output from a light source is temporally unstable. Thus, instead of trying to control spectral radiant intensity from the lamp system, it may be necessary to monitor the spectral radiant intensity output of the lamp using a spectroradiometer.

4. Integrating sphere based UV-chamber

A UV-chamber design based on integrating sphere technology has been built (Fig. 1).

Integrating sphere theory and technology are well-established [60–62] and integrating spheres are commonly used in making total reflectance or total transmittance measurements on materials and as a uniform light source. In this application, the integrating sphere is used as a uniform light source.

An integrating sphere is a hollow spherical chamber which has an inner surface coated with a highly diffuse reflecting coating either in the form of a coating or a monolithic wall material; that is, the surface is Lambertian. Currently, the most highly reflective, Lambertian material is prepared from pressed polytetrafluorethylene powder and bulk fluoropolymers, both having reflectance values greater than 98% at ultraviolet wavelengths longer than 280 nm [63].

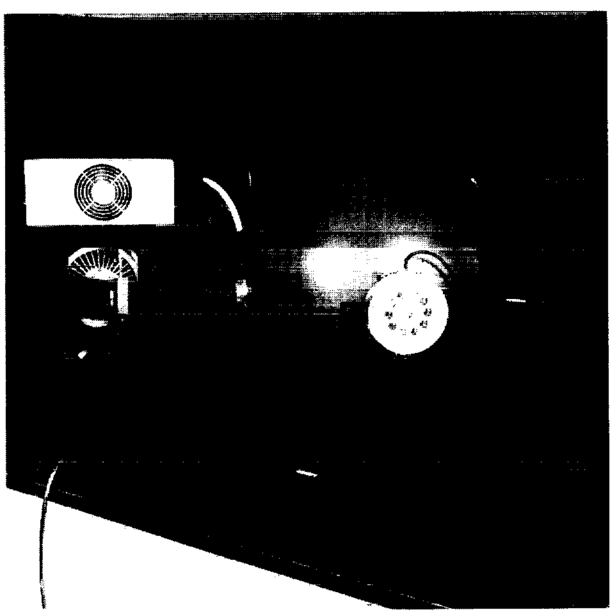

Fig. 1. Integrating sphere UV-chamber with two apertures. Exposure cells are mounted over apertures.

When radiation is admitted into the interior of an integrating sphere, the radiation is diffusely reflected from its interior surface. After multiple reflections, the radiation within the sphere becomes spatially integrated. It is highly uniform and has an increased irradiance that is directly proportional to the total radiant flux introduced into the sphere. This is very advantageous for monitoring the radiant energy flux within and exiting from the sphere since this flux is spatially uniform. Thus, a fiber optic detector, inserted through the shell of the sphere out of the line of first strike from the light source and connected to a spectroradiometer, provides an excellent measure of the spectral radiance within the sphere.

Assuming proper design, the radiation exiting an aperture machined through the shell of an integrating sphere is also diffuse and uniform over the dimensions of the aperture. Moreover, if multiple apertures are machined through the shell of the sphere, the light exiting each aperture is also diffuse, uniform, and has the same spectral radiance. Thus, an integrating sphere UV-chamber design immediately resolves the major source of systematic errors in current UV-chamber design; that is, uniform irradiance across the dimensions of a specimen and from specimen to specimen.

This design appears to be capable of mitigating most, if not all, of the systematic errors from the sources described above. Spectral radiant energy measurements were made in the center of two 14 cm radii apertures for the sphere shown in Fig. 1. The diameter of the sphere is 0.3 m. These spectra are overlaid in Fig. 2. Note that no discernible difference in the spectral output is apparent.

A rule of thumb in integrating sphere technology is that approximately 5% of the interior surface area of an integrating sphere can be opened without significantly affecting light integration. Since the interior surface area of a sphere increases by the square of its radius, the surface area allocated to apertures (and, thus, specimen exposure area) also increases by the square of the radius of the sphere.

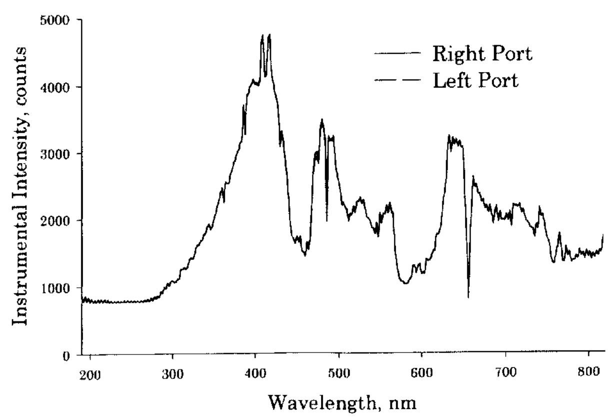

Fig. 2. Spectral emittance from two different apertures overlaid on top of one another.

Since the light exiting a sphere is spatially uniform, positioning a specimen at an aperture would expose it to uniform, diffuse radiation. Obviously, photons reflected from the surface of the exposed specimens should soon be spatially re-integrated within the sphere. However, possible errors resulting from these reflections will be assessed.

In many situations, it would be advantageous to uniformly irradiate specimens at a distance from the integrating sphere as shown in Fig. 3. This would be important, for example, if one wanted to expose specimens in a multiplicity of independently controlled exposure environments. For example, it may be advantageous to expose the specimen(s) at one aperture at 60°C, 95% relative humidity, polychromatic light; whereas, at another aperture, the specimen could be exposed at 50°C, 95% relative humidity, and 290 nm radiation. The capability to apply mechanical stresses to the specimen(s) while simultaneously exposing it to UV radiation can also be achieved with the integrating sphere-based chamber. Other unique exposure environments can be achieved through the construction of independent exposure chambers, including freeze/thaw conditions and rain. Uniform spectral irradiance of the specimens within each exposure chamber can be achieved through the use of nonimaging optics [64], such as depicted in Fig. 3.

With respect to systematic errors, integrating spheres appear to mitigate most or all of the systematic errors observed in current chamber designs. Uniform irradiance exposure across the dimensions of a specimen and from specimen to specimen is assured by the integrating sphere physics. Other advantages of an integrating sphere UV-chamber include the following:

1. Simplicity in design and easy accessibility to the light source, exposure cells and specimens:

   The light source, exposure cells, and specimens are located on the exterior of the integrating sphere and, thus, they are readily accessible while an experiment is on-going and for maintenance of the UV-chamber. This accessibility should greatly reduce problems associated with human/machine interactions. A standard protocol for operating such a UV-chamber, however, must be prepared.

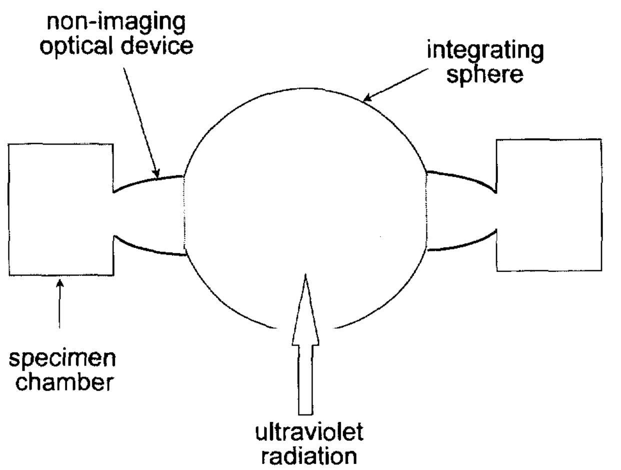

Fig. 3. Integrating sphere equipped with non-imaging optics and external exposure cells.

2. Removal of visible and infrared radiation from the radiant flux

   High specimen exposure temperatures can be minimized by removing most of the visible and infrared portions of the radiant energy flux emitted by the light source prior to entering the sphere. For example, radiation from a 1000 W xenon arc is used as the light source for the integrating sphere shown in Fig. 1. By removing most of the visible and infrared radiation emitted by the light, the temperature within the chamber can easily be maintained at slightly above room temperature. Thus, panels of different color are exposed at the same temperature.
3. Removal of radiation below 290 nm.

   By positioning a cut-off filter after the dichroic mirror, radiation below 290 nm can be removed from the radiant flux. Moreover, interference or cut-off filters can be positioned in front of each specimen so that each specimen can be uniquely irradiated using any combination of wavelengths.
4. 4. Spatial irradiance uniformity.

   As a uniform radiation source, an integrating sphere is capable of minimizing errors due to non-uniform irradiance over the dimensions of a specimen and from specimen-to-specimen. Spatial irradiance uniformity does not depend on the light source, the age of the light source, or batch to batch variability, but, instead, it is controlled by the physics of integrating spheres.
5. Temporal irradiance monitoring and control.

   Temporal changes in the spectral radiant intensity of the light source can not be controlled. Through the use of the integrating sphere, however, temporal variations can be easily spectroradiometrically monitored. This is possible because the radiant power within the integrating sphere remains uniform and, thus, changes in the radiant power can be easily monitored.
6. Experimental flexibility.

   Finally, integrating sphere provide the opportunity to simultaneously and independently expose a multiplicity of specimens each to its own exposure environment. This can be achieved by positioning specimens in individual exposure cells and uniformly irradiating the specimens by projecting a uniform, diffuse beam from apertures machined into the integrating sphere. The environmental and operating conditions within each exposure cell can be uniquely selected.

5. Conclusions

UV-chambers play an important role in comparing or predicting the performance of construction materials and determining the effect of different weathering factors on the performance of a construction material.

Although significant modifications have been made in current UV-chamber designs, controlling the systematic errors and thus the repeatability and reproducibility of these chambers has remained elusive.

An integrating sphere UV-chamber design has been proposed which appears to be capable of greatly mitigating known sources of systematic errors. These systematic error sources include human/machine interactions, high specimen temperature, inclusion of short wavelength radiation, non-uniform spatial irradiance over the dimensions of a specimen and from specimen-to-specimen, and temporal changes in radiant flux. Preliminary evaluations of an integrating sphere based UV-chamber are very promising.

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Appendix: Terminology

Current density: the current flowing to or from a unit area of an electrode surface. The current per unit cross-section area of a conductor. Also known as electric current density.

Dichroic mirrors: mirrors that are capable of efficiently separating polychromatic radiation into two parts. Dichroic mirrors are made to have a very high reflectance (more than 90%) for a broad spectral region and a high transmittance (over 98%) for the remainder of the spectrum. The absorbance of these mirrors is insignificant. They offer many technical advantages over the pellicle mirror and filter system used in one-shot color cameras and for color television because of the high efficiency of light utilization.

Dose (of optical radiation of a specified spectral distribution): term used in photochemistry, phototherapy, and photobiology for the quantity radiant exposure falling on a unit surface area of an object. Unit J m^{-2}.

Lambertian surface: a surface or radiator which has radiance which is not dependent on angle is called Lambertian. Flux leaving a Lambertian surface is perfectly diffuse; that is, the radiance is constant regardless of viewing angle.

Line source: an idealized source of light consisting of an infinitely long line from which light is emitted with uniform intensity.

Maximum solar ultraviolet radiant energy spectrum: the solar radiation spectrum observed on the equator, at sea level, at noon on a clear day during the summer solstice.

Repeatability: the precision of a method expressed in terms of the agreement attainable between measurements made by a single operator using the same apparatus and techniques.

Reproducibility: the precision of a method expressed in terms of the agreement expected between measurements made in different laboratories using similar apparatuses and the same procedure.

Solarization: the reduction in transmittance of a filter resulting from the exposure to short wavelength UV radiation and high temperatures.

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