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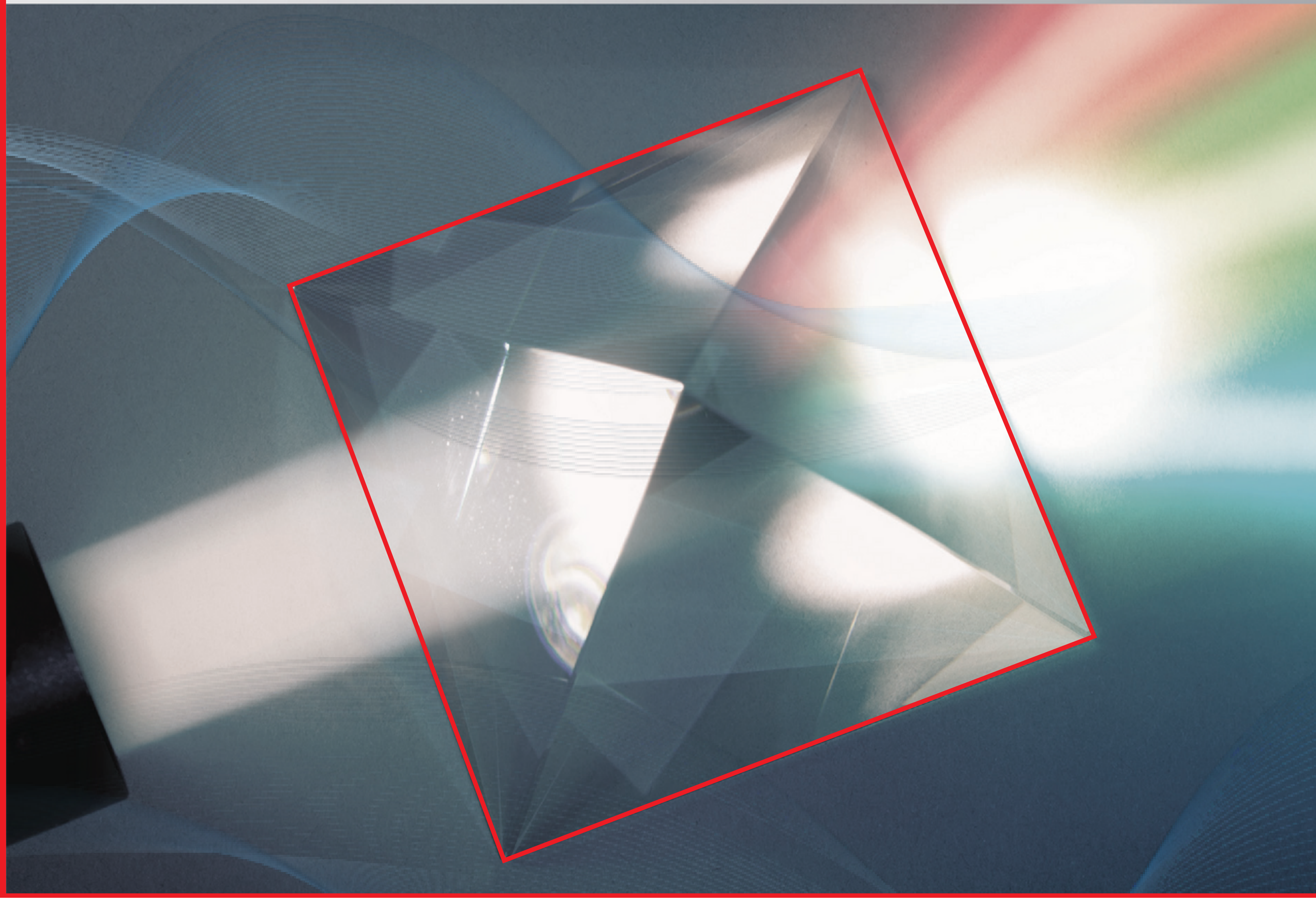
LOCTITE®

Light Cure Adhesive Technology Guide



Henkel

CONTENTS

LIGHT CURE ADHESIVE TECHNOLOGY GUIDE

INTRODUCTION

Virtually all adhesives, sealants and coatings require a curing or hardening process. This process may be as simple as the cooling of a hot melt adhesive or the evaporation of solvent from a contact cement, or it can be more complex and involve a chemical reaction, as is the case with epoxy, anaerobic and light curing adhesives.

Shortly after the commercialization of UV (ultraviolet) light sources over three decades ago, LOCTITE® pioneered the development of light curing adhesives, sealants and coatings for industrial applications. These are materials which cure or harden (polymerize) when exposed to light of appropriate wavelength and intensity. Originally, these systems responded only to UV light, but, due to advances in light cure chemistry, response to suitable visible light is now also common and often advantageous. In some applications, it is even necessary. Although "light cure" or "photocure" more accurately describe the chemistry, since light other than UV is used, the term "UV cure" is deeply ingrained, and will probably continue to be used to describe both UV and visible light cure systems.

Advantages

- The primary advantage of light cure processing is speed of cure. Depending on the product and system, cures can be achieved within seconds. Other benefits include:
- One-part systems – no need to measure or mix; no pot life concerns
- No solvents
- Very fast room temperature cure
- Cure on demand – facilitates parts alignment
- Requires less equipment space and energy than ovens
- Allows testing of parts soon after assembly – eliminates the possibility of large quantities of work-in-process scrap
- In-line production

Limitations

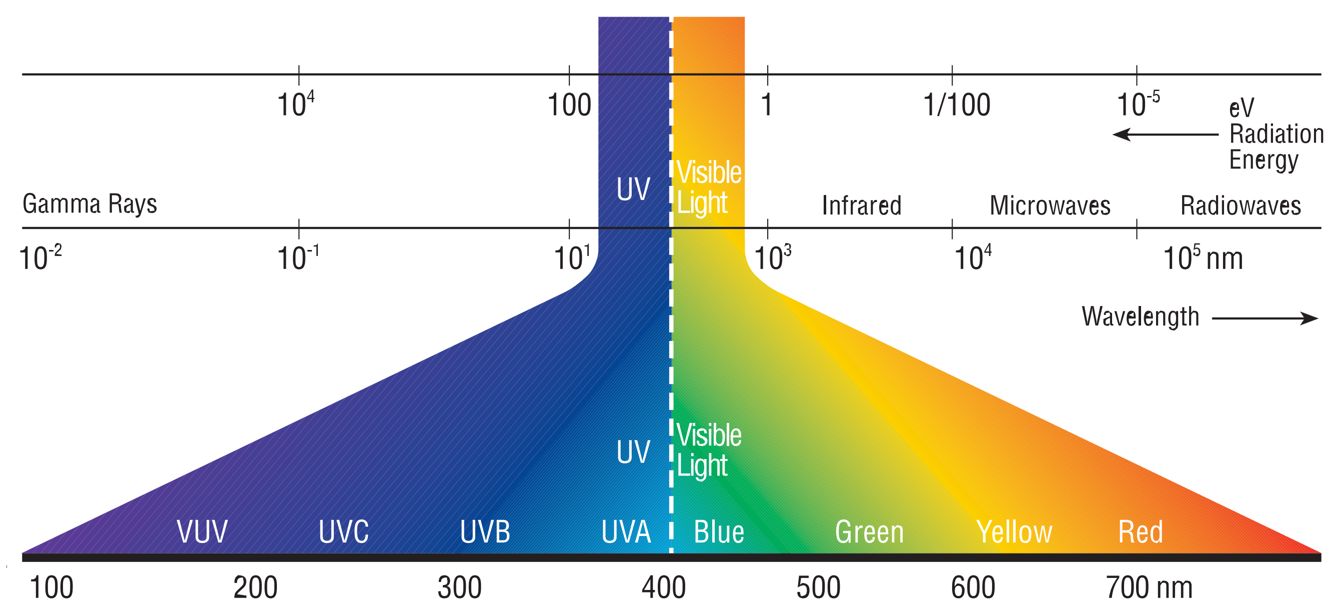
The primary limitation of a light cure system is that the material to be cured must be exposed to suitable light — both in type (wavelength or spectral distribution) and intensity. This means that for an adhesive application to be successful, one substrate must transmit appropriate light. Also, assemblies with shaded or shadowed areas (areas not exposed to light) will require a secondary cure mechanism.

BASIC PRINCIPLES

The Electromagnetic Spectrum

Energy is needed to initiate a chemical reaction. UV and visible light are examples of *radiant energy* – energy transmitted from a high-temperature source to a receiving body without the use of any matter in between. UV and visible light actually comprise a very small portion of the *electromagnetic spectrum*, which characterizes all wavelengths of radiant energy found on earth. UV light extends from 100 to 400 nm (nanometers) and visible light from 400 to about 760 nm in wavelength. A nanometer is one billionth of a meter (10 to 9 meter) or 10 angstroms. Light in the UV or visible region, which is capable of interacting with matter to produce chemical reactions, is also referred to as *actinic light.*

The Electromagnetic Spectrum



The diagram above illustrates the types of radiant energies and associated wavelengths which make up the electromagnetic spectrum.

TYPES OF LIGHT

UV Light

As indicated in the diagram on page 4, UV light is subdivided into four groups based on wavelength/energy and ability to interact with matter:

UVA (315 to 400 nm) Used for UV polymerization reactions; also for fluorescent inspection purposes.

UVB (280 to 315 nm) Used with UVA for polymerization and, since it is the most energetic region of natural sunlight, for accelerated light aging of materials. UVB light is responsible for suntanning.

UVC (200 to 280 nm) Used for rapid surface cure of UV inks and lacquers; also for sterilization and germicidal applications.

VUV (200 to 100 nm) Vacuum UV can only be used in a vacuum and is, therefore, of minor commercial importance.

Visible Light

Visible light is also subdivided, with different colors being associated with different wavelengths, as shown in the diagram of the electromagnetic spectrum.

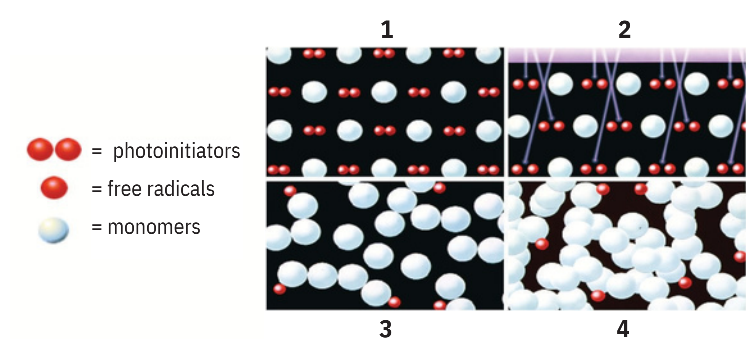
Vis (400 to 480 nm) Wavelengths used for visible light cure are in the violet-to-blue region (400 to 480 nm). Henkel's Indigo® Visible Light Cure adhesive products cure in the pure visible spectrum above 450 nm.

Commercial Use of Radiant Energy

Radiant energy found in nature is generally diffuse. In order to make radiant energy economically and commercially useful, people must be able to generate it at will and be able to intensify during its generation. Designing equipment that efficiently and economically produces suitably intense radiation for industrial use, whether UV, X-ray or any other type, is a continuing challenge.

Light-Induced Polymerization

In order for a light cure adhesive to react to UV or visible light, a chemical called a photo initiator must be present in the formulation. The figure below illustrates the sequence of a typical light curing reaction. The uncured adhesive is shown in figure 1 where the white spheres represent adhesive monomer and the double red spheres represent photo-initiators. When the liquid adhesive is exposed to the appropriate light energy, the photo-initiators fracture into free radicals (single red spheres) as shown in figure 2. These free radicals then initiate the curing or polymerization process shown in figure 3, leading to the extremely rapid development of the long polymer chains that make up the cured adhesive as shown in figure 4.



CHEMISTRY

Types of Light Cure Adhesive Chemistries

Light cure products used in industry today are based on three major types of polymerization chemistries: free radical, anionic and cationic.

Free Radical

Acrylic

The majority of commercial light cure products are of the free-radical acrylic type. Upon exposure to suitable light, extremely rapid polymerization occurs. Polymerization stops as soon as the light is removed. Free-radical acrylic systems are subject to oxygen inhibition, which means that oxygen in the air prevents the molecules at the surface from polymerizing, leaving a wet or tacky surface. This phenomenon can be minimized through proper selection of product, equipment and optimization of the process. Free-radical acrylic systems are the most versatile of all light cure systems because many different types of monomers and oligomers are available for the formulator to obtain different properties.

Silicone

Light cure silicones are another type of free radical system. Henkel has developed two types of proprietary silicones that cure upon exposure to light — silicones that cure completely upon light exposure and dual cure silicones. Dual cure silicones cure partially upon exposure to UV light, followed by a secondary moisture cure mechanism that completes the cure, cures shadowed areas and provides enhanced adhesion. Both systems require high-intensity UV light for curing. Like acrylics, light cure silicones are inhibited by oxygen. Light cure silicones provide outstanding low-temperature flexibility, excellent electrical properties and a high degree of water resistance.

Hybrid

Hybrid light cure systems utilize a combination of standard epoxy chemistry, e.g., a two-part mix epoxy/hardener, and free-radical acrylic chemistry. Acrylic-monomer and photoinitiator are added to one of the epoxy components. After mixing the two parts, exposure to light causes the product to gel firmly, resulting in a fast fixture. This feature enhances productivity. Subsequent cure of the epoxy at room temperature or by heat generates properties typical of epoxies.

Anionic

Light cure cyanoacrylates utilize an anionic dual-cure mechanism. Polymerization is effected by both UV/visible light and by the basic surface. Light cure cyanoacrylates offer the substrate versatility of conventional cyanoacrylates, including adhesion to elastomers and polyolefins (with primer), with the added benefits of low blooming, no oxygen inhibition, excellent cure in areas not exposed to light and the ability to cure through large gaps.

Cationic

Cationic systems contain epoxy and/or vinyl ether materials along with other components to modify properties. Because a fairly limited variety of monomers and oligomers are available for use in these systems, versatility in tailoring properties is not as great as with acrylic systems. Unlike free radical systems, some cure continues after the light source is removed, but this is minimal and often requires a thermal bump, or heating, to be effective. However, polymerization will not propogate into shadowed areas. The photoinitiators used in cationic systems can be somewhat toxic and their residues can be corrosive, e.g., to delicate platings used in the electronics industry. Reactivity of cationics may be decreased by exposure to high humidity. Cationic systems are not subject to oxygen inhibition and, therefore, exhibit excellent surface cure. Cationics also typically offer high thermal resistance and low outgassing.

Pre-activated cationic systems provide similar benefits and limitations as the cationic systems. The difference with these materials is that the system is exposed prior to assembly to start the reaction. Once the assembly is mated, the reaction continues to a thermoset material. The benefit is that you can cure through opaque parts or in areas that will not see exposure.

EQUIPMENT

Basic Equipment Components

Basic equipment components of commercial light cure systems include the following:

- Light source (bulb technology or light-emitting diode (LED)
- Irradiator (lamp housing and reflector assembly)
- Power supply and electrical controls
- Shielding and cooling
- Conveyor and/or auxiliary equipment

Light sources, reflectors and cooling options will be discussed in the following sections.

The Light Source

The heart of a light cure system is, of course, the light source or light-emitting diode (LED). Many types of light sources are available and the choice of a particular light source depends both on technical needs and on economic constraints. The two most important characteristics, which determine how fast and effectively a light source will cure a product, are the spectral *output* and *intensity*.

Spectral Output

Spectral output is defined as intensity of light at each wavelength over the range of wavelengths emitted by the light source. *For the most effective cure, this pattern of output must be matched to the pattern of absorption of the photoinitiator in the product.* This principle forms the basis of all successful light cure applications. Spectral output determines whether a light source is suitable for a particular application and how effectively the light source will cure the product. Lamp engineering and modifications of the material inside the lamp (called *doping*) enable shifting of the spectral output to other wavelengths. Similarly, LED light sources are designed with peak intensity at specific wavelengths. Figures 1 to 7, on the pages which follow, illustrate spectral output characteristics of various light sources commonly used in industry.

Since there are certain absorption maxima common to many adhesives, there is often a choice of light sources for a given product. Major absorption areas of importance are 254 nm, (surface cure), 365 nm, (the "workhorse" cure wavelength, also effective in promoting depth cure) and 400 to 436 nm, (currently used where visible photoinitiators absorb). Visible light cure is required for UV-opaque substrates and for deeper cure.

Because measurement of spectral output is not practical in an industrial environment, spectral output of the type of light source in the equipment must be known. The supplier's literature is the best source for this information. *Henkel Technical Data Sheets* provide information on recommended light sources for each product.

Intensity

Light source intensity is defined as the overall power of the light source and is most often designated in watts. Also called power density, intensity refers to total light-source output across the entire electromagnetic spectrum. Light-source intensity generally affects the rate of cure of a particular product, since it is *one* of the factors determining the amount of light that actually reaches the product, e.g., the light which initiates the chemical reaction. Other factors affecting the quantity of light reaching the product are discussed on pages 15 and 16.

It is important to distinguish between basic lamp intensity and intensity or amount of light at the working surface. The quantity of light at the working surface is defined in either intensity units or energy units. Light *intensity* at the product surface, described by the term *irradiance*, is a measure of momentary exposure and is most often quantified in *milliwatts/cm2*. Light energy at the surface is a measure of cumulative intensity exposure (intensity x time), also called *dose*, quantified as *millijoules/cm2*, and is simply:

mW/cm2 x seconds = mJ/cm2

Variations in use of these terms are summarized below:

Term	Abbreviation
microWatts/cm2	μW/cm2
milliWatts/cm2	mW/cm2
Watts/cm2	W/cm2
Joules/cm2	J/cm2
milliJoules/cm2	mJ/cm2

One mW/cm2 equals one thousand μW/cm2; one joule is one thousand millijoules.

Henkel light cure products are often referred to as high- or low-intensity (also called high- or low-energy) curing products. Generally speaking, a low-energy product will respond well to roughly 25 to 30 mW/cm2 intensity, occasionally less, while high-intensity cure refers to the 80-100 mW/cm2 region and above.

Measurement of irradiance (light intensity at the working surface) is essential in order to optimize cure conditions and maintain consistency in cure results. Fortunately, easy-to-use equipment is available for this measurement. Henkel offers radiometers in different regions of the electromagnetic spectrum and for different types of equipment.

Industrial Light Sources

LED

LED technology has been widely used throughout the industry because of the numerous benefits an LED can provide. An LED provides a very narrow spectral output, which can minimize excessive exposure to other wavelengths. LED technology provides instant on/off capability and lasts much longer than a conventional bulb (i.e., mercury arc, electrodeless, etc.). Typically, an LED can last over 10,000 hours, when most conventional electrode bulb technology fails after 2,000 hours at best. In addition, the LED technology does not generate significant heat and uses less power than conventional bulbs. Although there are other wavelengths, the spectral output of a 405 nm LED is shown in Figure 1.

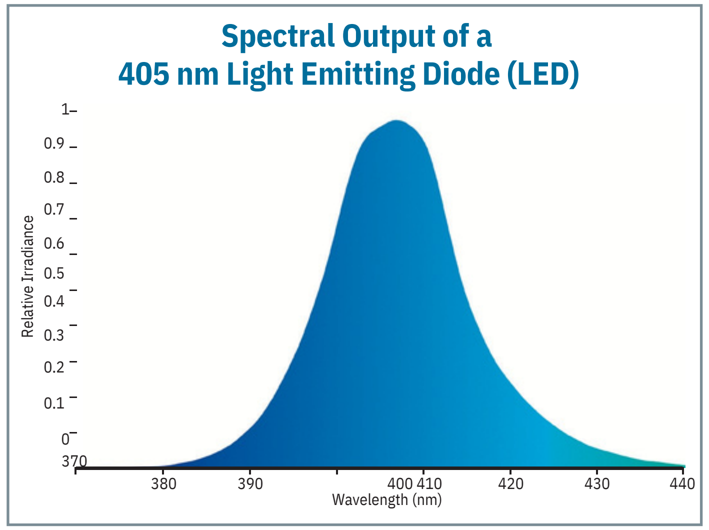


Figure 1

Henkel has spot, line and flood systems that utilize LED technology.

Medium-Pressure, Mercury Vapor Lamps with Electrodes

These lamps, also called mercury arc lamps, are the workhorses of the light-curing industry. They combine cure efficiency with design versatility and moderate cost. An electrode-type, medium-pressure, mercury vapor lamp consists of three major components:

- a quartz glass sleeve
- fill material, typically an inert gas, and a small amount of liquid mercury
- electrodes sealed into the ends of the quartz sleeve

A high voltage is applied to the lamp across the electrode terminals. The voltage field ionizes the inert gas and produces heat to vaporize the mercury, which creates a pressure of about two atmospheres. This pressure is needed to maintain a balance between the required high intensity, while maintaining the spectral output essentially in the UV region. The mercury vapor arc, which is created, exhibits the unique property of high emission of light in the ultraviolet region of the spectrum. In particular, intense emissions occur in the 240 to 270 nm and 350 to 380 nm areas, which is where typical UV photoinitiators absorb. This intense light beyond the violet region of the visible spectrum has sufficient energy to interact with photoinitiators and cause their fragmentation, which initiates polymerization. Some visible light and infrared radiation are also generated. Figure 2 presents the spectral output of a typical medium-pressure, mercury, vapor lamp.

Electrode-type, mercury vapor lamps are available in sizes ranging from a few inches to about 80 inches in length and at various power levels, ranging from 100 watts/inch to 600 watts/inch. The most popular lamps are in the 200- to 300-watt/inch range. Intensity delivery on a part is high and can be in the 100- to 200-mW/cm2 range.

Warm Up and Cool Down

One disadvantage of mercury vapor lamps is that they require a warm-up and cool-down time. They cannot be turned on and off instantly.

Intensity Deterioration

The intensity of output of a mercury vapor lamp with electrodes deteriorates with use. After 1,000 hours of use, the output will be reduced by 15% to 25%. It is important to note that because lamp deterioration is seldom catastrophic, it is not easily perceptible. Regular monitoring of lamp intensity at the working distance is extremely important.

Henkel has flood systems that utilize medium-pressure, mercury vapor lamps with electrodes.

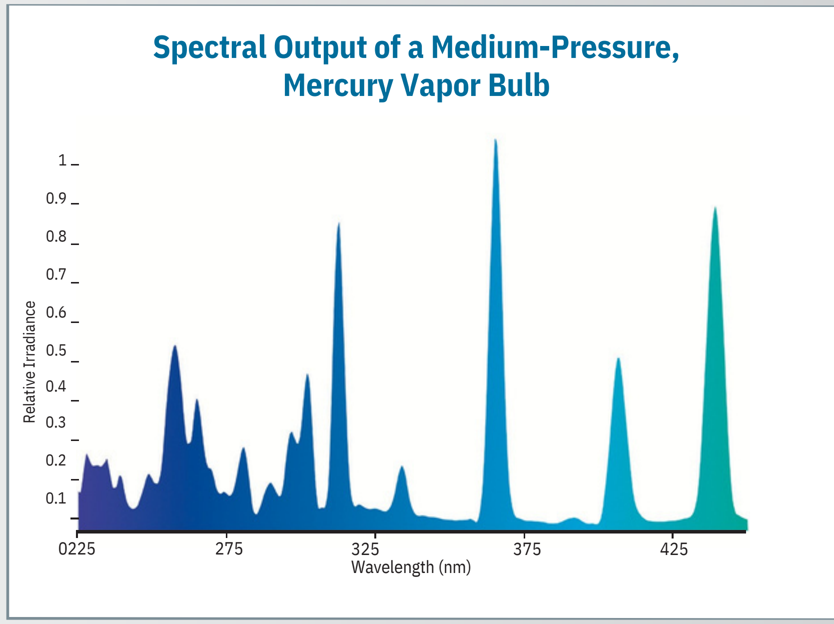


Figure 2

High-Pressure, Mercury Vapor Lamps

Mercury lamps, which can be spherical in shape, feature portability and fast startup time, but have electrodes. High-pressure lamps are typically used in wand-systems. The energy is delivered via a liquid light guide to the tip of the wand. Figure 3 shows the emission spectrum of this source.

Henkel also provides systems with high-pressure, mercury vapor lamps, with intensity of up to 20 W/cm2.

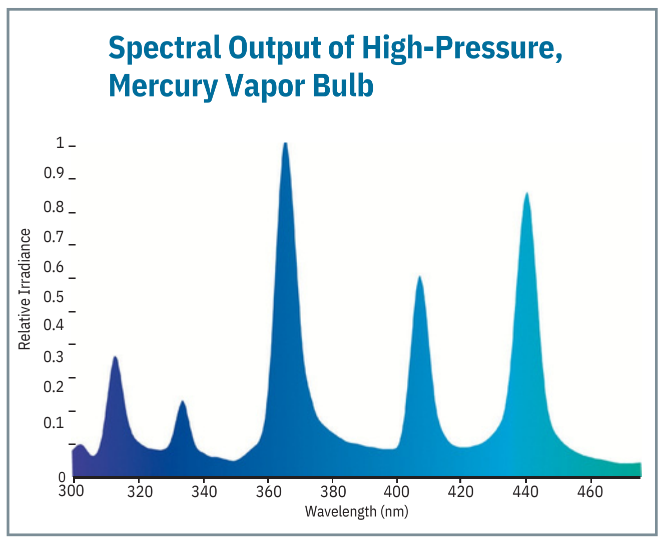


Figure 3

Metal Halide Lamps

Metal halide lamps are a type of medium-pressure, mercury arc lamp (with electrodes) in which the spectral output is modified by addition of metal halides to the bulb contents. The most common dopant is an iron halide, which enhances output in the UV region. Gallium or indium halides are used to shift emission maxima to the visible region. Henkel offers a choice of two types of metal halide lamps. The metal halide system is a low-to-medium-energy unit, delivering 20-to-50-mW/cm2 intensity at the part surface, depending on distance from the source.

Figure 4 illustrates spectral output of the UV bulb, which is iron doped; Figure 5 illustrates output of the visible bulb, which is indium doped. Henkel metal halide-cure equipment utilizes advanced light-filtering technology that enhances cure response

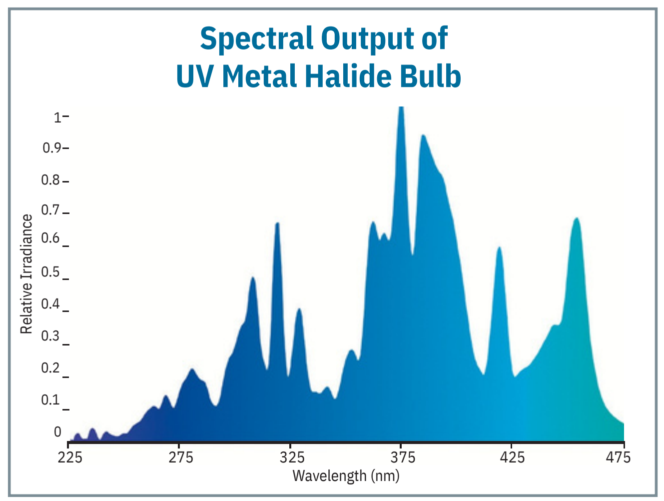


Figure 4

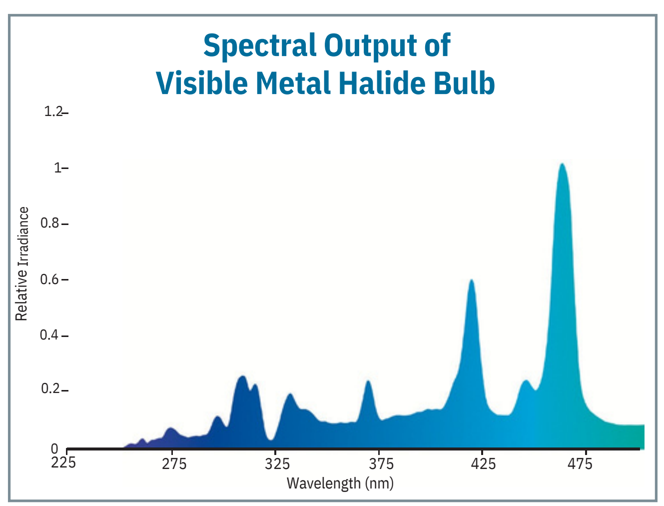


Figure 5

Electrodeless, Mercury Vapor Lamps

The electrodeless, mercury vapor lamp is a unique type of medium-pressure lamp. In this design, the mercury in a quartz sleeve is vaporized by microwave energy rather than by the electrode process. Major advantages over lamps with electrodes include rapid on-off capability, much longer bulb life (guaranteed for 8,000 hours) and bulbs with a smaller diameter which, in combination with reflector design, results in higher efficiency. With electrodeless, mercury vapor lamps, a very high intensity can be generated at a working surface, well in excess of 2,000 mW/cm2, enabling very high cure speed.

Metal halide doping (adding a metal halide to the contents of a lamp) is used to change the spectral output of these bulbs. Spectral output of the three most popular electrodeless bulbs are shown in Figures 6a-c. The H bulb output is most similar to a conventional medium-pressure, mercury electrode-type bulb output. In the V bulb, output has been shifted strongly into the visible region, while the D bulb has been tailored to exhibit characteristics of both bulbs. The D bulb is often used to achieve good cure depth. A new H+ bulb exhibits enhanced emission at shorter wavelengths, effective in promoting surface cure.

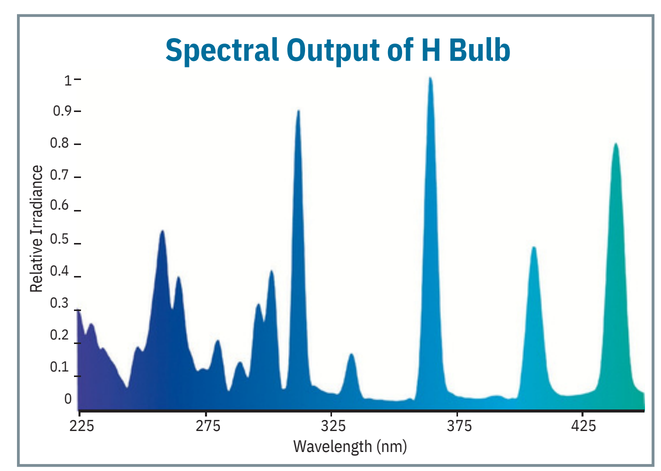


Figure 6a

Disadvantages of electrodeless bulbs include significantly higher cost not only for the bulb, but also for the entire system, and limited bulb-length sizes, only six inches or ten inches. Length is limited by the size of magnetrons needed for microwave generation, but units may be placed in tandem to effectively increase cure area.

Henkel can supply electrodeless, mercury vapor lamp technology.

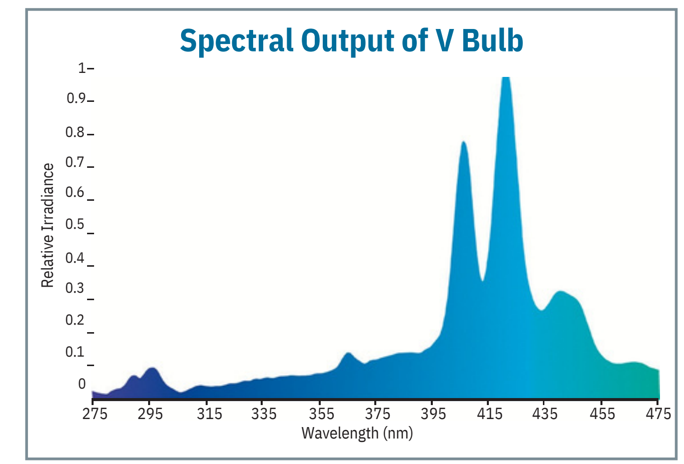


Figure 6b

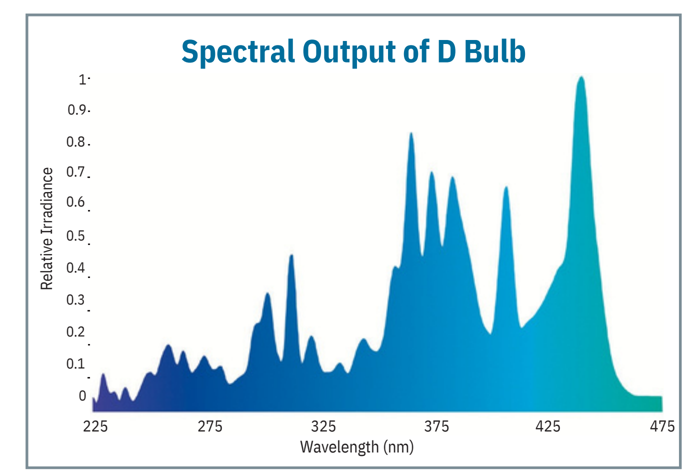


Figure 6c

EQUIPMENT

Blacklight Lamps

Blacklight lamps are a type of fluorescent lamp designed to emit in the UVA region. Since intensity is quite low, about 6 to 10 mW/cm2, they have limited use in a production environment, although they can be used for quality-control purposes or with materials that cure under very-low-energy radiation. Spectral output maximizes in the 320-to-400-nm region (see Figure 7).

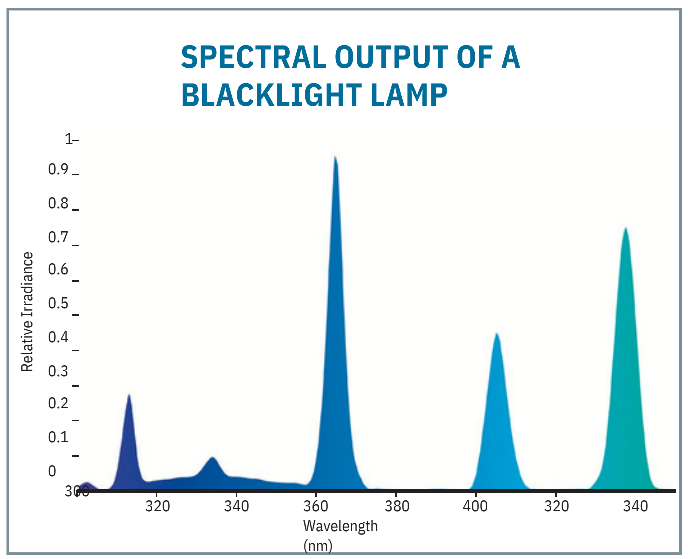


Figure 7

Other Light Sources

A pulsed xenon or flash xenon lamp utilizes xenon gas and no mercury to generate UV, visible and infrared radiation. This equipment, often used for curing of fiber-optic coatings, utilizes an energy storage capacitor to trigger the lamp system 120 times per second, with 6 to 10 microsecond pulses, to yield bursts of very high energy and power peaks, as high as one million watts. Advantages are known to be very fast cure rates and degree of cure, excellent depth of penetration, low heat buildup and instant on-off capability. A wide variety of lamp configurations is possible, although high cost—higher than electrodeless systems—is a disadvantage.

Laser light sources are also used industrially. Several laser sources are available, including argon, krypton and helium/cadmium. This technology is often used for rapid production of prototypes (stereolithography) operations. While laser sources produce very fast cure, cost and safety are two major obstacles.

The Reflector

Depending on end-use application, it may be desirable to concentrate or focus the radiant energy on the target substrate (achieved via an elliptical reflector) or to diffuse or flood the light to produce an even distribution of light intensity over a surface (achieved via a parabolic reflector). Parabolic reflectors radiate light over the broadest possible cure area, but with the lowest light intensity at the substrate. Focused elliptical reflectors offer the highest possible light intensity, but the smallest exposure area. Also, a focused light generates significantly more heat, which may adversely affect the substrate. When using focused light, part positioning becomes critical since intensity drops off sharply as the distance from the focal point increases. An alternative is *defocused* light. This offers reasonably high intensities and somewhat broad cure areas by using focused light and positioning the substrate beyond the focal point.

Basic reflector designs and lighting options are illustrated in Figure 8.

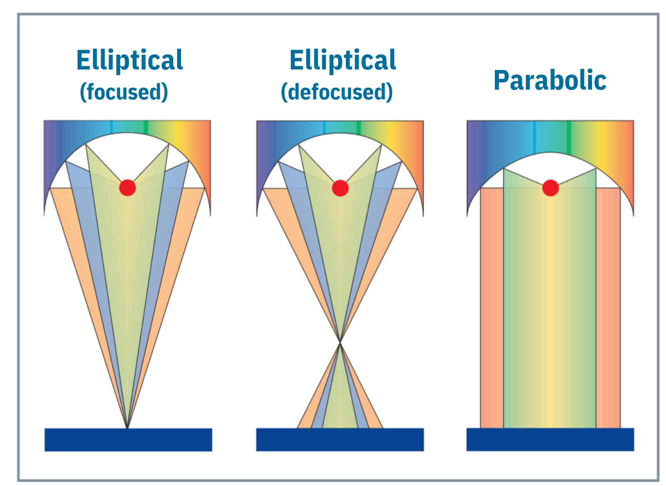
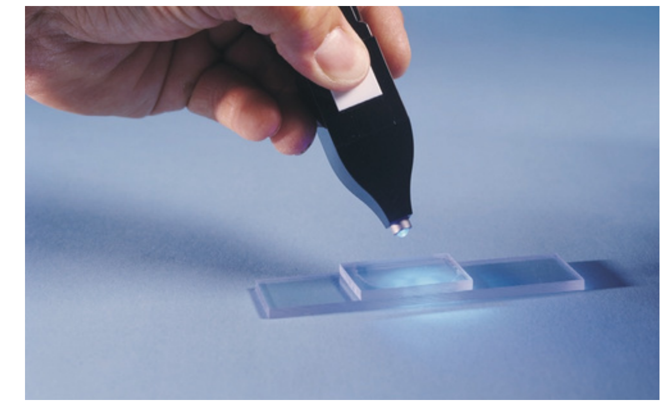


Figure 8



Cooling

Even the most tailored mercury-vapor, ultraviolet light produces some infrared or heat energy. The heat generated must be removed, not only to protect the lamp, but also to prevent damage if thermally-sensitive parts are being processed. Often, cooling by circulation of air through vents in back of the reflector is sufficient. However, additional cooling systems are available, including:

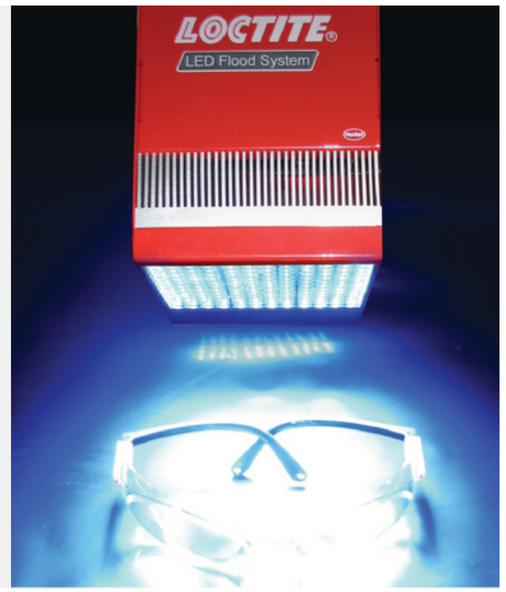
- Water cooling of the lamp housing or reflector
- Bowing refrigerated air between polished quartz plates above the work surface
- Using a dichroic reflector or cold mirror

Cooling of a lamp cannot be indiscriminate. The quartz sleeve of the lamp must be maintained at a temperature from 600 to 800°C. Below 600°C, the mercury will condense and the lamp will lose its arc; above 800°C, the quartz sleeve will start to deteriorate. Lamp terminals on electrode-style lamps must be kept below 150°C to prevent damage to the seals. Although cooling lamps is critical to the success of the bulb vaporizing the mercury, the efficiency of the LED is also affected by heat and, also, must be actively cooled to provide high irradiance.

Four Major Factors must be taken into consideration when designing a successful light cure application. These factors influence both product selection and overall efficiency of the manufacturing process. They are:

- Spectral output /photoinitiator match
- Light intensity on product
- Transmission properties of substrate
- Cure properties needed (depth cure, surface cure, speed, etc.)

General guidelines for optimizing each of these parameters to achieve reliably the full cure of a product in an economically viable manner are presented in this section.



Spectral Output/Photoinitiator Match

One of the most important considerations in achieving optimum cure is to match the spectral output of the light source to the absorbancy characteristics of the photoinitiator system in the material to be cured. This is accomplished by coordinating light-source choice with product chemistry. As previously discussed, a variety of light sources with differing spectral outputs are available.

Different photoinitiators have different absorption characteristics. The more intense a light source's emission at the wavelengths absorbed by a product's photoinitiator, the more efficient the cure. Often, several light sources will cure a product with some degree of effectiveness, but one source may enhance particular properties required, e.g., surface cure or depth cure. While measurement of spectral output is not practical on-line, the equipment manufacturer will supply output diagrams of lamps used. Henkel Technical Data Sheets utilize Henkel equipment to generate data for each product.

Light Intensity on the Product

Factors Determining Light Intensity

Overall intensity of light on the product, *irradiance,* is a major factor influencing the speed of cure –the more intense the light, the faster the cure speed.

Irradiance is a Function of:

- Light source power
- Distance from light source to product
- Type of reflector
- Lamp age
- Transmission characteristics of substrate

Light Source Power

As previously noted, light-source power is rated in watts per inch of length, also called power density. The mercury vapor arc lamps most commonly used in commercial light curing systems are available in power densities ranging from 100 to 600 watts/in., but the two most widely used are 200 and 300 watts/in. This value is total light-source output across the entire electromagnetic spectrum, including visible and infrared radiation, and does not indicate intensity at any particular wavelength.

Distance from Light Source to Product

As with any source of light, the intensity at a surface decreases as the distance from the source increases. The distance-intensity relationship is significantly influenced by reflector design. In order for the measurement of the light-source intensity to be meaningful, readings must be taken at the working distance from the light source and in the area where the adhesive is located during the curing process.

Type of Reflector

As discussed on page 13, reflector design effects intensity of light at the working surface. It is important to maintain a consistent working distance between the source and the product to be cured in order to keep the influence of the reflector optimized.

Lamp Age

The usable life of a mercury arc lamp depends on several factors, including whether it has or does not have electrodes, number of starts, operating time per start, temperature in the electrode area of lamps with electrodes and power density. Lamps with electrodes are expected to last 1,000 hours with loss of no more than 15% to 25% of original intensity. Lamps without electrodes boast a 8,000-hour-life. The best source for information on aging characteristics of specific lamps is the lamp or equipment manufacturer. Regular monitoring of lamp intensity at the working distance is extremely important. Equipment for monitoring intensity is readily available and is suited for use in an industrial environment.

Visible LEDs will last for over 10,000 hours without degradation, which provides a very robust curing system.

KEYS TO SUCCESS

Transmission Properties of Substrate

For effective photocure, light of suitable intensity and spectral distribution must reach the product. For a coating or potting application, once factors determining light intensity described on page 15 are known, effective cure of a product can be predicted. However, for an *adhesive* application in which the light must pass through a substrate before it reaches the product, one other critical factor must be considered – the substrate transmittance characteristics.

Even though a material appears colorless and transparent to the eye, it may actually block out light needed for a UV cure. For example, while most clear glass-types transmit UV light, Flint F Glass (D.E.D.F. 5901) and polyvinyl butyral-laminated safety glass filter out all light of 365 nm wavelength, which is needed for UV cure. Also, two basic types of visually clear polycarbonate (P/C) are common in industry today: UV-transmitting, typically used in medical device applications and UV-absorbing, used in auto headlamp and other outdoor applications. UV-transmitting P/C passes about 70% of 365nm light, but UV-absorbing P/C passes only about 3%, not enough to cure products, containing only UV photoinitiator. Other types of clear plastics may also filter UV light. Visible light-cure products usually offer the solution to achieving cure through clear UV-filtering substrates, and with new visible light curing technology, it is often possible to cure through clear-tinted or clear-colored substrates also.

Color and gloss of the bottom substrate can affect cure speed. The reflected light from a white or glossy-bottom surface may speed cure, while a black-bottom substrate may slow cure.

Measuring a substrate's transmission characteristics is easily accomplished even in a production environment and should be performed before specifying a product. Radiometers, which detect light at 365 nm and at 400 nm, should be used to determine if either a UV or visible cure is required. The substrate is exposed to the light and intensity measurements, which the adhesive will be exposed to during curing. Intensity, without the substrate on top of the radiometer, should also be measured to determine light-filtering characteristics of the substrate.

Cure Properties Needed

A further consideration involves selecting or adjusting options available in order to most effectively achieve the curing and cured product properties required for the specific use. These requirements include targeted cure speed, depth of cure (CTD) and surface cure.

Cure Speed

Cure speed depends both on the composition of the product and on light intensity. Any factor that reduces light intensity on a product will reduce cure speed. For example, if the speed of a conveyor belt is increased to reduce cycle time, incomplete cure may occur due to decreased time of exposure to light. However, it is important to note that problems may also occur if the product cures too fast. One concern is high heat generation. Excessive heat may adversely affect thermally sensitive parts, produce cracking in the cured product or cause fogging due to volatilization of the normally non-volatile components in the product. A more subtle, but significant, negative effect of too rapid a cure is the possibility of reduced adhesion to the substrate.

Depth of Cure

Cure depth is enhanced by longer wavelengths, e.g., greater than 350 nm. For very large cure depths, visible light cure systems are recommended. CTD is a function of product chemistry, wavelength, intensity and time of exposure, this information can be found in the Henkel Technical Data Sheets for each Henkel product. Cure depth may be enhanced — or cure in shadowed areas may be achieved — by use of dual cure systems, which are formulations containing a secondary cure mechanism (heat, moisture or activator).

Surface Cure

Surface cure, on the other hand, is enhanced by exposure to shorter, more energetic wavelengths. Surface cure may not be important in an adhesive application, except if there is a large fillet, but is obviously important for coating, tacking or potting applications. As previously indicated, acrylic systems are subject to oxygen inhibition, which adversely affects surface cure. Making the atmosphere inert with nitrogen is effective in overcoming this problem and is used in the coating industry, but is not practical for most industrial applications.

The best solution to this problem is careful selection of product and cure equipment parameters and use of high energy, particularly at 254 nm. Both medium-pressure, mercury arc lamps and the electrodeless H and H+ bulb emit strongly at 254 nm.

Measuring Light Intensity/Energy

The successful use of light-curing products is very much dependant on light intensity or irradiance at the part surface. Thus, it is critical to measure and monitor this characteristic regularly. For best practice, light should be measured under actual production conditions and, if appropriate, through the substrate being assembled. Radiometers are designed to measure the appropriate wavelengths needed for polymerization, such as industry standards 365 nm and 400 nm, enabling easy on-line monitoring. Keep in mind that even if intensity readings at a specific wavelength are similar between two light sources, cure efficiency may not be the same, due to the potential differences in the entire spectral distribution from the light sources. However, measuring at one wavelength is an excellent means of determining optimum irradiance for a particular application and for monitoring the performance of the light source to ensure consistent cure results.

Light energy is another measurement that is important to help a customer understand the interaction of light and the curing adhesive for proper polymer development. Light energy, which is also commonly called *dose,* measures the light intensity at the work surface (irradiance) multiplied by time (expressed in mJ/cm2 or J/ cm2).

Dose = Irradiance (mW/cm2) * time(s).

Dose is also measured at a specific wavelength so one can determine the energy at the desired wavelength important for matching light absorption of the photoinitiator across different light sources.

Broad Versus Narrow

There are two types of radiometers (broad range and specific wavelength) on the market. If using a single wavelength radiometer, which measures the wavelength at which your adhesive cures, you can accurately compare two different pieces of lightcure technology equipment at that wavelength. If you are using a broad-range radiometer, you have the potential to overexpose or underexpose the adhesive. The reason is that bulbs and LEDs have different spectral outputs. So, if you are measuring a bulb that has the highest peak at 365 nm, the broad range will pick up this maximum peak. However, if you have a bulb that has its highest peak at 325 nm, the broad range will peak up this peak and not the 365 nm peak, which is where the adhesive cures. To elevate this, testing, along with broad range measurements is critical when comparing bulbs to ensure that the irradiance of the appropriate wavelength is being transmitted to the adhesive.

When Is the Product Cured?

Users of lightcure adhesives have desired an easy process to verify that the adhesive has been sufficiently cured to address concerns that the appropriate wavelength of light of sufficient intensity has been correctly applied for the needed duration of time. Off-line techniques exist, such as the destructive testing of parts or analytical testing of cured adhesive samples. An on-line technology (called AssureCure®) also exists that rapidly confirms the degree of cure of a light cure acrylic. The AssureCure® system is easily integrated into existing or new assembly production lines, is non-destructive to parts and readings can be taken in as little as 20 milliseconds.

Additional Off-line Destructive Testing Techniques

- Bonding: strength test to destruction or to a proof load
- Coating: peel or cross-hatch adhesion tests
- Potting: cross-section material and test hardness through depth
- Tacking: strength to pull off

Once cure conditions have been tuned to satisfaction, all variables should be closely controlled and regularly monitored to insure consistent results.

SAFETY

Hazards to personnel

Hazards to personnel involved in UV and visible light curing are of four types:

- Ultraviolet emission
- Ozone exposure
- High voltage and temperature
- Lamp handling

Ultraviolet Emission

The primary concern of ultraviolet light emission is the potential of radiation burns due to exposure to a high-intensity UV light. The energy associated with non-ionizing UV light is unable to penetrate into the body and interact with tissue, and so it does not produce the dramatic physiological effects associated with ionizing atomic radiation. However, exposure to high-intensity UV and visible light (which contains UV light) can cause severe damage to skin and eyes. The ultimate effects of these injuries are determined by both dose rate and length of exposure. Biological damage is most prevalent at exposures to wavelengths below 325 nm (UVB and UVC regions). Although the primary emission band of a medium-pressure mercury vapor lamp is at 365 nm, other strong emissions occur at 313 and 254 nm, requiring this popular light source to be treated with great caution.

UV Burns to the Skin and Eyes

Unlike thermal burns, which are felt immediately, UV burns take several hours to be felt. Even short exposure to high-intensity UVB and UVC light can cause severe burns to the skin and eyes. Skin burns generally appear as a reddening 3 to 4 hours after exposure and maximize about 12 hours post-exposure. It takes 2 to 7 days for the reddening to start to decrease. UV burns of the eyes will cause the cornea to blister and peel, giving the victim the sensation of having sand in the eye – hence the terms "ground glass eyeball" and "welder's flash." This type of injury usually becomes evident 6 to 12 hours after exposure, but could surface as soon as 2 hours or as late as 24 hours after exposure. Symptoms can be very painful, but are usually temporary and recovery is typically within 48 hours. Exposure to the longer wavelength UVA may also contribute to the formation of cataracts.

Protective Equipment

Skin and eyes should always be shielded for maximum personal safety. Protective equipment includes appropriate goggles, face shields, gloves, clothing and UV-opaque shielding material. Exposure to first-bounce radiation should also be avoided.

Exposure Limits

Because of these potentially serious effects on skin and eyes, allowable exposure limits have been established by both the American Conference of Governmental Industrial Hygienists (ACGIH) and the National Institute for Occupational Safety and Health (NIOSH). The NIOSH standards for unprotected eyes and skin for an eight-hour day are:

- <0.2 µW/cm2 maximum total effective exposure to wavelengths in the 200 to 315 nm range
- 1.0 mW/cm2 maximum total effective exposure to wavelengths in the 315 to 400 nm range

A standard radiometer can be used to monitor UV levels in various areas.

LED Benefit

LEDs in the visible spectrum can eliminate all of the above issues because they do not emit in the UV region. However, LEDs in the UV or in the low visible spectrum (405 nm) range will, of course, emit UV energy, and protection of the skin and eyes is still required for the end user.

Ozone Exposure

Ozone is a colorless gas with a strong odor which is present at sea level in 0.05 ppm concentration. At 20 times that concentration (1 ppm), ozone can be irritating to the eyes and respiratory tract and cause headaches. Ozone is generated when very short UV wavelengths (184 nm), passing through the pure quartz sleeve of UV lamps, interact with oxygen. Ozone production is most prevalent during lamp startup. OSHA regulations require ozone concentrations to be maintained below 0.1 ppm.

The easiest way of protecting workers from high levels of ozone is venting the area to the outside, where ozone rapidly decomposes into oxygen. In well-engineered UV-cure systems, no additional equipment is needed since this is accomplished via the air flow used for cooling. Another way is to eliminate generation of ozone by preventing production of short-wave energy, either through addition of oxides to the quartz or by selecting a lamp that has been doped to change its spectral response to higher wavelengths.

Simple and inexpensive equipment is available for monitoring ozone levels. The most widely used consists of a hand pump, fitted with a glass vial containing a chemical solution, which changes color upon exposure to ozone. The color change can be converted to ppm using a chart supplied with the system. This type of equipment is available from any major safety equipment supplier.

High Voltage and High Temperature

Suitable enclosures should be used for high-voltage components and commonsense precautions regarding the extremely high bulb temperature should be observed. Be sure to unplug the system before replacing the bulb.

Lamp Handling

Lamps should be handled with cotton gloves, never with bare hands, since fingerprints can contaminate the quartz and lower bulb efficiency and useful life. Breakage can be a hazard, since the mercury in the bulb, although small in quantity, is poisonous. It is a good idea to invest in a mercury-spill, cleanup kit. Be sure to unplug the system before replacing the bulb.

As with many other industrial processes, safety precautions must be observed in using UV-curing technology. With operator education and good equipment design and control, hazards can be minimized and a dependable cost-effective production process can be achieved.

LED Benefits

Since LED light sources do not use quartz sleeves, they do not produce ozone.

FREQUENTLY ASKED QUESTIONS

Which light source should I use to cure a UV/visible light-curing product?

The first consideration in selecting a light source is the transmission properties of the substrate through which the light must pass (if an adhesive application). This can be determined at the plant if suitable equipment is available or by the Henkel Application Engineering Group. If the substrate transmits both UV and visible light, then there are many source options available. Choice of light source then depends on the equipment specified. Of course, cure speed will be faster with higher intensity (mercury arc and electrodeless) systems.

If the substrate transmits only visible light, you have to rely on the visible photoinitiator in the product and should not use the UV metal halide bulb. Electrodeless V or D bulbs, LEDs or the visible halide lamp are primary choices. Mercury arc and electrodeless H bulbs also work because they emit considerable light in the visible range of the spectrum.

Before final selection of a light source, other requirements of the product should be taken into consideration, especially cure through depth (CTD) and surface cure. These two properties are especially important for potting applications. If very large CTD is needed, an LED or electrodeless system with either the D or V bulb is recommended. If surface cure is needed, a bulb that emits strongly in the short range UV region is recommended, e.g., a mercury arc source or a electrodeless H or H+ bulb. These sources will emit enough visible light to cure a visible/UV product even through a UV opaque substrate.

Remember, the above recommendations apply only to products that contain both UV and visible photoinitiators.

What is the best way to match a light source to the adhesive selected?

First, determine the photoinitiator system in the adhesive: UV-only or UV visible. Then, determine transmission characteristics of the substrate. If the substrate transmits visible light, but not UV light (that is, is UV-opaque), you cannot use a product that contains only a UV photoinitiator; a visible light photoinitiator must be present in the product. If the substrate transmits both UV and visible light, any source will cure a product, although the main concern is speed of cure, which is related to light intensity at the part surface. Factors influencing light intensity are discussed in detail on pages 8 and 9.

I inherited a light source. How do I know if it will work with the adhesive I have selected?

Try to obtain information on the light source, including type of bulb. Measure irradiance at the proposed working surface in UV and visible ranges, both with no substrate present and through the part substrate. Use a radiometer that measures at 365 nm and 400 nm.

What is the difference between the visible technology and the Indigo® technology?

Visible technology refers to products that cure in the low visible region of the electromagnetic spectrum (i.e., 405 nm). Henkel's Indigo® product line cures at higher visible wavelengths >450 nm. These adhesive chemistries are coupled with Henkel's Indigo® brand of equipment, which includes LEDS and bulb type systems.

The light curing adhesive I have used for months is suddenly not working properly. Parts are falling apart right off the assembly line. What should I check? Is it the adhesive or the light?

There are three elements in any application: the part, the product and the process. Check all three.

The Part

Make sure the transmission characteristics of the substrate or the substrate supplier has not changed. These characteristics should have been measured when the application was first designed. If a UV-opaque plastic was inadvertently substituted for a UV-transparent plastic, a change not easily evident to the eye may prevent the product from curing under the conditions originally established.

The Product

Light-curing adhesives typically exhibit extremely consistent cure characteristics. Henkel adhesives, for example, are subject to rigorous quality-control testing. It is rarely a problem with the adhesive so long as the same adhesive is being used as was originally specified. Make sure there has been no inadvertent change in product identity, packaging or storage conditions.

The Process

Historically, changes in process conditions are the most likely cause of assembly failures. Check simple factors first. Has the process speed been increased? Are the parts in the proper orientation? Then measure the intensity of the light at the working surface using a radiometer with probe(s) of the appropriate wavelength, 365 and/or 400 nm. Irradiance is a critically important process condition and should have been specified when the application was designed.

Remember that the intensity of a light source declines slowly over time and, thus, is not immediately perceptible. In addition, contamination to light guides and reflectors requires periodic cleaning to ensure proper intensity. If the intensity is not as specified after cleaning, change to a new bulb and recheck the intensity. If it still is not right, check the reflector system, the distance to part and similar factors.

How can I be assured that the adhesive is fully cured? Is there a scientific method?

Yes, an on-line technology (called AssureCure) exists that rapidly confirms the degree of cure of a light cure acrylic. The AssureCure® system is easily integrated into existing or new assembly production lines, is non-destructive to parts and readings can be taken in as little as 20 milliseconds. Additional confirmation that the curing process has been completed can be attained via off-line, destructive testing and/or analytical testing of the cured adhesive.

FREQUENTLY ASKED QUESTIONS

How many joules does it take to cure the product selected?

As discussed earlier, a joule (1000 millijoules) is a cumulative amount of energy, determined by multiplying irradiance by time:

mW/cm2 x seconds = millijoules/cm2

It is not possible to answer the question without specifying other variables, especially depth of product to be cured. In a way, the question can only be accurately answered empirically. If a suitable cure is obtained in 10 seconds at 100 mW/cm2, this means one joule (1000 mJ) is needed. If suitable cure is obtained in 20 seconds at 30 mW/cm2, 0.6 joule is needed. These are very typical cure conditions for our products. Generally speaking, our newer products will cure with one joule or less energy in a typical application.

Can LOCTITE® products be cured in EB (electron beam) systems?

Theoretically, yes. Due to the nature of the energy in an EB system, free-radical cures are effected even without a photoinitiator. However, while EB is an effective way to cure a coating, cure of an adhesive is generally not practical since an extremely high-voltage, electron beam accelerator would be needed to generate the energy required to penetrate a typical substrate. In addition, the several-hundred-thousand-dollar capital investment and minimal versatility of the system are disadvantages.

Will gamma rays (used in gamma ray sterilization) cure LOCTITE® products?

Considering their location in the electromagnetic spectrum, gamma rays could theoretically cure our light-curing products, given sufficient dosages. Moreover, there is empirical evidence that demonstrates that cure can be effected by gamma rays. Systematic studies have not been conducted to determine the exact conditions needed, or whether complete cure is possible or practical. One additional word of caution: for certain plastic substrates, including polycarbonate, it is not a good idea to partially cure a product and complete the cure later, e.g., during gamma-ray sterilization, because the uncured portion of the product may cause stress cracking in the polycarbonate, given sufficient time of contact and stress in the substrate.

How do I select the best light cure chemistry and product for my application?

The first step in selecting the best chemistry and product is identification of the substrate(s). Next, determine the performance specifications for the assembly. This will further narrow the field of choices by defining product properties such as temperature resistance, adhesive strength, flexibility, toughness, etc. If this is an adhesive application, knowledge of bond geometry is important to determine gap cure requirements and need for cure in shadowed areas. Processing conditions, e.g., production speed, will further help determine the appropriate chemistry and product. Once these factors are known, Henkel engineers can recommend suitable products.