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Quantitative Confirmation of the Degree of Cure of a Light Cure Acrylic Adhesive

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Abstract

Approximately forty years ago, the adhesive industry introduced acrylic-based adhesives that cured or solidified on exposure to UV light. Concurrently, UV light cure equipment became commercially available. This early UV technology offered manufacturers distinct advantages over traditional adhesive technologies such as cyanoacrylates and epoxies, including very rapid cure rates, adhesion to a variety of substrates, the ability to fill large gaps, and easy automation.

Light cure adhesives are used in a wide range of industries from medical needles to LCD displays and automotive headlamps. The technology enabled manufacturers to accelerate their production processes while producing aesthetically pleasing, clear bondlines.

The use of light cure acrylic technology requires that the bondline be accessible to the light source selected. Hence, opaque joints or areas of an assembly that are shielded from light will not cure. In addition, insufficient irradiance (the amount of light energy delivered to the liquid adhesive) caused by the selection of an inappropriate light source, and/or the degradation of a qualified light source can result in uncured or partially cured adhesive.

Users of light cure acrylic technology have long desired a quantifiable in-line process to verify that the adhesive has been sufficiently cured. The recently developed Loctite® AssureCure®[1] technology provides such a solution where confidence in cure is critical to a bonded assembly.

Light Cure Technology

Light cure acrylic adhesives must be exposed to light of sufficient surface intensity and the appropriate wavelength to activate the curing mechanism. The wavelengths of light are found in the electromagnetic spectrum (Figure 1) which categorizes radiant energy by wavelength.. Light cure acrylic adhesives are typically cured using ultraviolet and/or visible wavelengths of light in the range of 200 to 500nm.

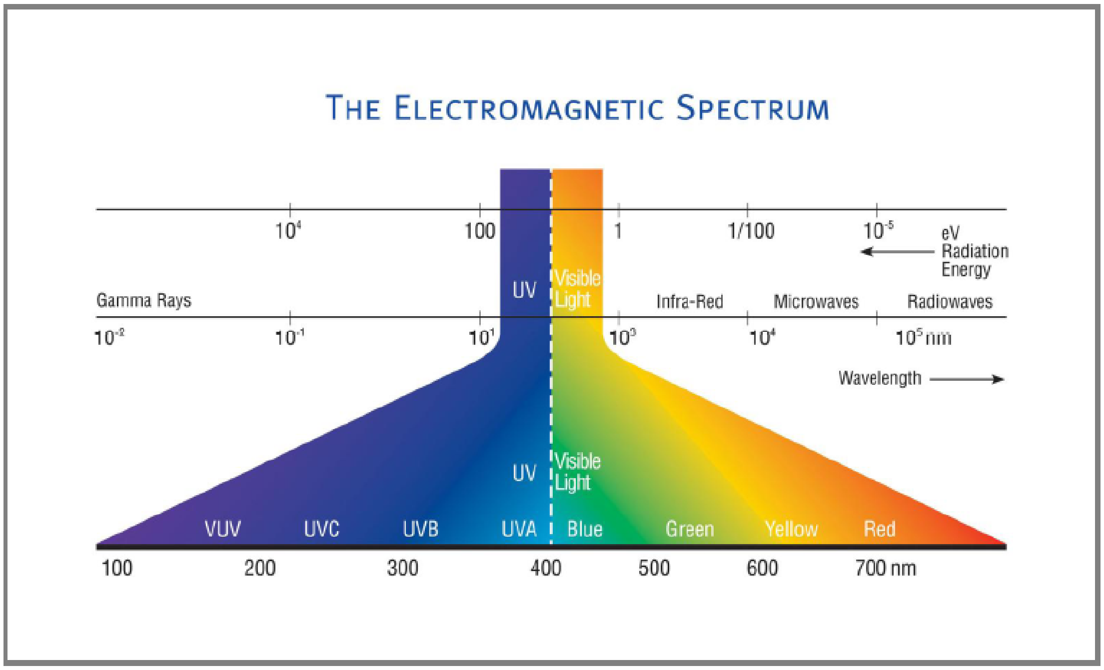

Figure (1): The Electromagnetic Spectrum

Figure 2 illustrates the sequence of events of a typical light curing reaction. All liquid light cure adhesives contain photoinitiators which are shown as double red spheres. When the liquid adhesive is exposed to light of an appropriate wavelength and intensity the photoinitiators absorb the light energy and fragment into reactive free radicals. These free radicals react with the monomers, shown as single white spheres, initiating an extremely rapid polymerization process.

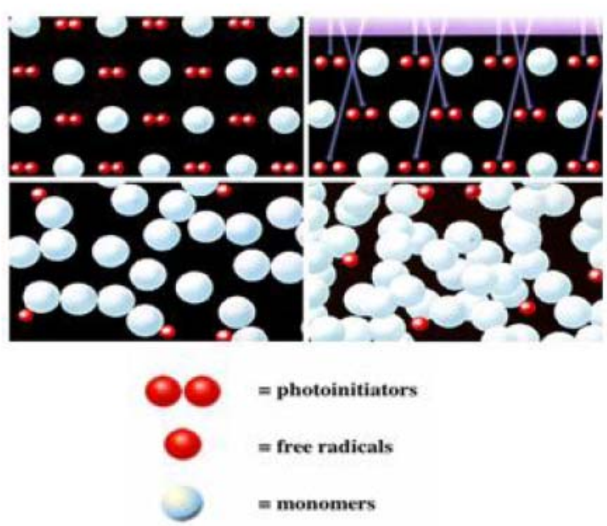

Figure (2): Typical Light Cure Schematic

Table 1 in the appendix shows the major benefits and considerations of traditional light cure adhesives.

Beyond the need for light to reach the adhesive, a primary drawback is the inability to confirm the curing process has properly occurred. This concern has been verified in surveys to light cure acrylic users who employ a number of techniques attempting to address this shortcoming via the mechanisms noted below in Table 2 below[2].

Adhesive Detection Mechanism	Adhesive Prescence	Non-Destructive Testing (No scrap)	In-Line Process	Confirmation of Cure
Fluorescence Adhesive	YES	YES	YES	NO
Color Changing Adhesive	YES	YES	YES	NO
Physical/Destructive Testing	YES	NO	NO	NO
Analytical Testing	YES	NO	NO	YES
AssureCure	YES	YES	YES	YES

Table (2): Current Cure Confirmation Methods

Adhesives with fluorescent detection can only confirm that the adhesive is present and in what location.

Color changing adhesives can only confirm that the adhesive has been exposed to light, but is not directly linked to free radical generation or the curing reaction.

A physical or destructive testing process can confirm that curing of the adhesive has occurred, yet the procedure is not directly integrated on-line, involves a time lag that cannot immediately stop the manufacture of defective assemblies, and the value of the tested parts are lost as scrap.

Analytical testing of the adhesive can confirm that an acceptable level of cure has been reached, but cannot be integrated on-line for immediate quality verification. Likewise such a process results in lost parts to testing and cannot prevent a build up of defective parts if incomplete curing continues.

The AssureCure system outlined below provides the manufacturer with in on-line, non-destructive process that confirms an acceptable level of cure has been achieved.

AssureCure System Basics

AssureCure is designed to be used directly on a manufacturing line to quantitatively determine that a light cure acrylic adhesive has cured. The AssureCure system combines a newly developed adhesive with detection equipment and a software package.

The system is designed to optically measure changes during polymerization. By applying a special algorithm to this measurement, the degree of cure is obtained without destructive testing. The measurement is obtained in as little as 20 milliseconds.

The AssureCure system is comprised of a fiber optic light source and detection equipment. The fiber optic light source momentarily illuminates the adhesive and the response is analyzed by the detection system.

By applying a specially developed algorithm to the response signal, a direct correlation to the adhesives degree of cure is obtained. The data is collected, analyzed, and can be displayed on an existing PC or PLC using the AssureCure software package. Results can be displayed numerically, corresponding to the degree of cure, or as a pass / fail measurement.

The process of integrating an AssureCure system on a production line starts by first producing correlation curves for the specific application of interest. One correlation curve, shown in Figure 3, compares graphically the adhesive's degree of cure, based on FTIR measurements, versus the output from the AssureCure system. Figure 3 shows an excellent correlation between the AssureCure system response on the right scale, and an FTIR3 analysis of the adhesive over varying light exposure times. As the measurement from the AssureCure detection increases, the % cure or % conversion measured with FTIR also increases.

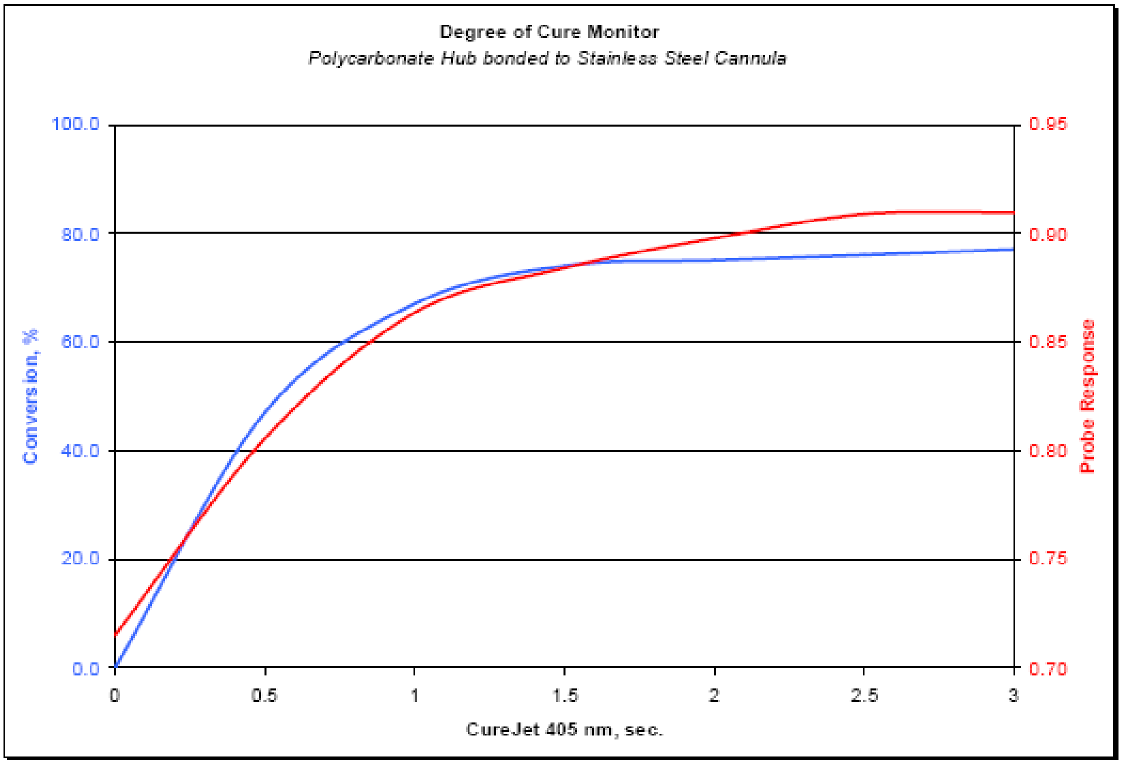

Figure (3): Percent of cure (conversion %) from FTIR analysis versus AssureCure probe response over varying light exposure time intervals

The AssureCure system response also directly correlates to the strength build of the adhesive. Figure 4 shows the correlation curve of needle pull strengths versus the AssureCure output over various time intervals of light exposure. As the pull strength of the polycarbonate hub to the stainless steel cannula increases, the output from the AssureCure system also increases, showing excellent correlation. The AssureCure system provides assurance that the adhesive has cured completely and reached the desired strength for the target application.

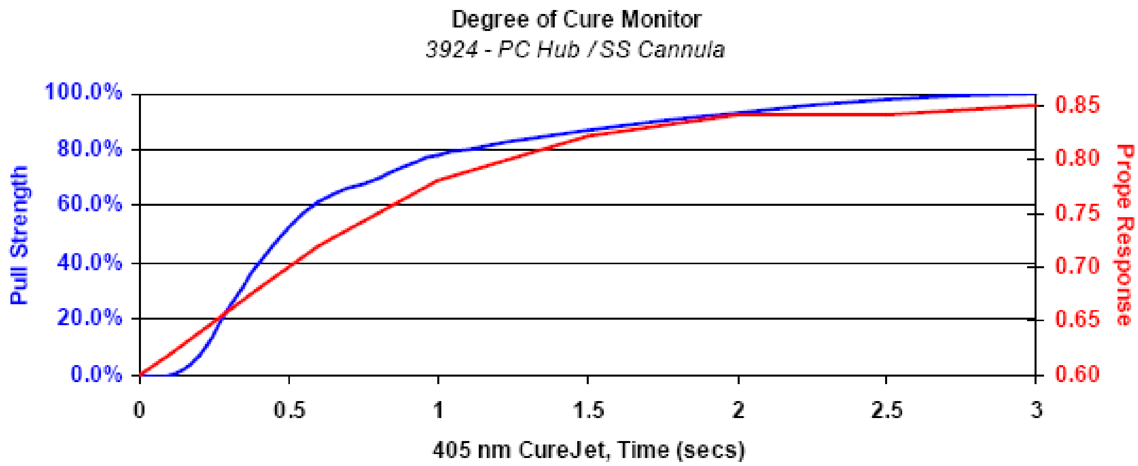

Figure (4): Correlation between needle pull strength and AssureCure response over varying light exposure time intervals

In the manufacture of biomedical devices the biocompatibility compliance of the medical device is essential to ensure that the assemblies being produced are safe for use in the field. Cytotoxicity testing determines the biological reactivity of a mammalian cell culture in response to a test article. The response to the cell monolayer is evaluated under a microscope and given a grade from 0-4 depending on the biological reactivity (cellular degeneration and malformation). A result of grade 0 indicates that there was no reactivity during the testing where as a grade 4 indicates that there was a severe amount of reactivity. Figure 5 shows another correlation curve that compares the AssureCure output to the biocompatibility of a bonded assembly. As the Cytotoxicity of the bonded sample improves from a grade 4 (severe reactivity) to a grade 0 (no reactivity) indicating the progression of the curing process, the AssureCure output indicates a concurrent progression of the curing process. This inverse correlation offers biomedical device manufacturers assurance that their bonded assemblies have achieved an acceptably low level of cytotoxicity.

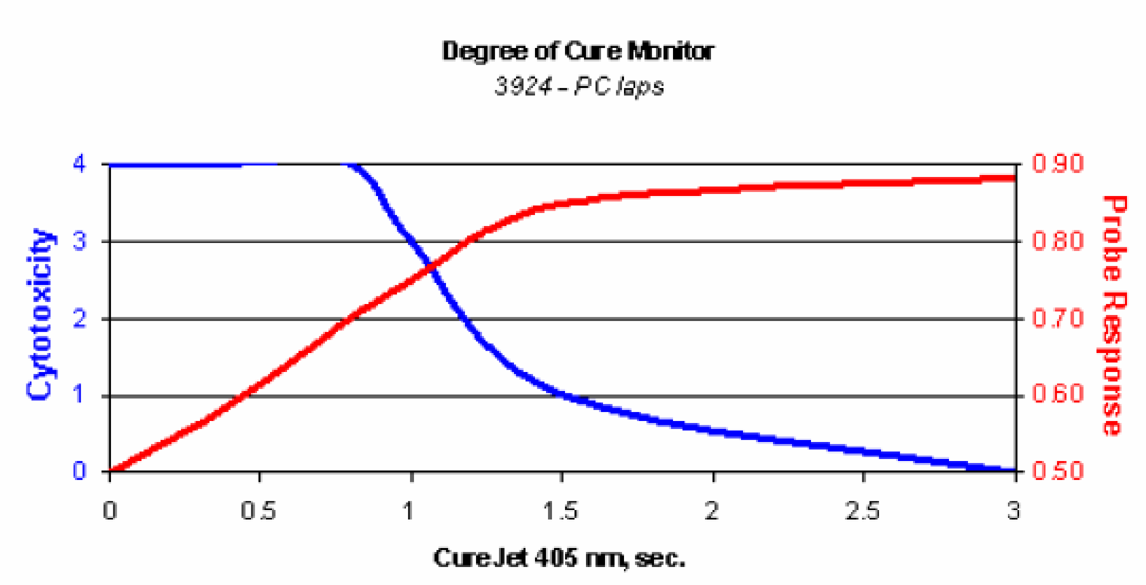

Figure 6: Correlation between cytotoxicity and probe response

Once integrated into a production line, the AssureCure system provides a reliable, quantitative measurement of the degree of each of the parts. The data obtained from the AssureCure system has shown an excellent correlation to % conversion using FTIR analysis, performance properties of the adhesive, and biocompatibility compliance. The AssureCure system can be installed online and its small footprint allows for installation into existing production lines. The AssureCure system also provides manufacturers an immediate quality check on 100% of their bonded assemblies. This on-line quality assessment provides the ability to eliminate the buildup of defective product waiting for off line quality checks.

Conclusions

The AssureCure system provides end users with a reliable, on-line, non-destructive method to confirm that the adhesive used to bond their components has cured. This system offers significant value to manufacturers at high financial risk if their products fail in the field causing damages to their product assembly, a manufacturer's brand equity, the end customer's property, and potentially to the end customers themselves.

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Benefits	Considerations
Manufacturing Process	
Very fast cure speeds (5 to 30 secs) facilitate high speed manufacturing rates.	Confirmation of Cure - No conclusive verification that light exposure was adequate to yield full cure
Cure on Demand - adhesive remains liquid allowing for part alignment, followed by rapid curing exactly when needed	Capital Expense - the purchase of adhesive dispensing and light curing equipment is required
Ease of Quality Control - rapid full cure permits immediate performance testing	
Reduced product staging/work in process – free up floor space and increases asset utilization	
Low labor input - both the dispensing and curing process are easily automated to reduce labor costs and increase product consistency	
Product Design and Performance	
High bond strengths on many materials - including metals, plastics, and glass allowing for wide lattitude in choice of substrate material.	Light Transmission – light must reach the adhesive
Aesthetic appeal - vast improvement over fasteners and other mechanical assembly methods	
Miniaturization - very precise dispensing and curing is easily accommodated	
Environmental, Health & Safety	
Little to no VOC's or emissions - minimal IR/Ozone emissions with UV wavelength adhesives and no emissions with visible wavelength adhesives	Eye/Skin Protection - Protective measures are required for UV wavelength adhesives
Safe and Non-Toxic - adhesive formulations are non-toxic and safe to handle. Exposure to visible wavelength lights yield no damage to skin or eyes.	Venting of Ozone – may be required for higher intensity UV light sources

Table (1): Benefits and Considerations of Light Cure Adhesives

1 – Loctite AssureCure is a registered trademark of the Henkel Corporation
2 – Henkel Corporation survey to light cure acrylic adhesive users