

## Photo-Oxidation and Photoprotection of the Surface Resin of a Glass Fiber-Epoxy Composite

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### Synopsis

The rapid photo-oxidation of the surface epoxy resin of a commercial seven-ply laminate (Scotchply 1009-26) is due principally to the epoxy novolac resin component. The photo-oxidation rate of this resin is eight times that of the other component, a bisphenol A epoxy resin. This rate depends on the conditions of cure, and photo-initiation occurs in part through aromatic carbonyl groups formed by oxidation of the methylene linkages of the novolac at the cure temperature (160–180°C). Inhibition of this thermal oxidation by vacuum cure or a chain-terminating antioxidant increases the photostability. Photoprotection of thin resin sections by the UV stabilizer 2-hydroxy-4-isooctoxybenzophenone and an epoxidized analog is assessed.

### INTRODUCTION

The surface of a glass fiber-epoxy composite is rapidly degraded on outdoor exposure unless it is protected by a surface gel coat, paint, or a UV-absorbing protective film.<sup>1</sup> This degradation is characterized by a loss of the surface resin layer and blooming of the glass fiber. Fundamental studies of this process have been limited by the uncertainty in epoxy resin composition and the intractable nature of the cured composite that prevents the use of conventional techniques of polymer analysis.

Recently, studies have been made of a commercial seven-ply glass-epoxy laminate of known composition (3M-Scotchply 1009-26) using sensitive techniques of surface analysis such as laser pyrolysis GC-MS,<sup>2</sup> ESCA, Fourier transform IRS-IR spectroscopy,<sup>3</sup> and torsional pendulum analysis.<sup>4</sup> These studies of samples exposed at field sites have shown that degradation is most severe in hot, wet climates with high total solar radiation dose and suggest that the surface resin degrades by photo-oxidation to produce low molecular weight segments that are washed from the surface.

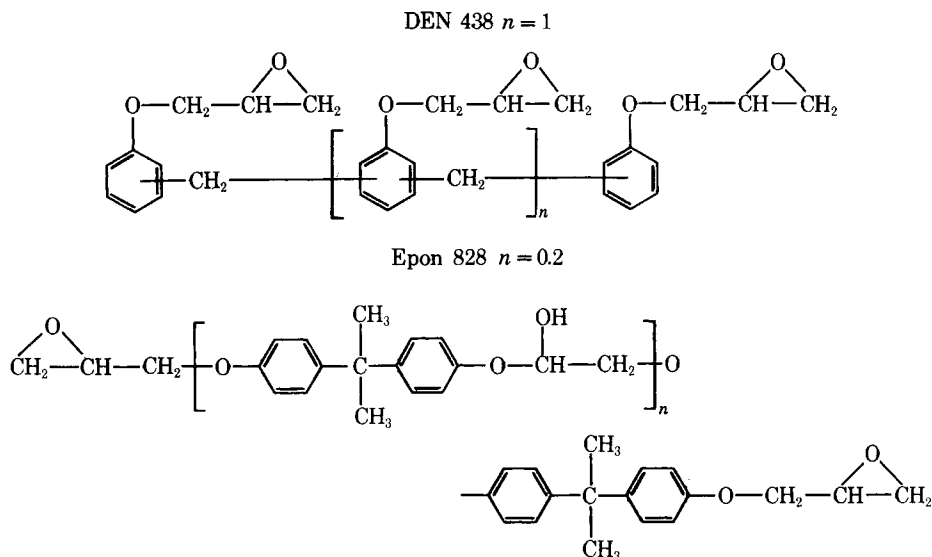
In this investigation, the photo-oxidation process of the resin and its components are studied to determine the initiation process, the rate of oxidation, and the way this can be inhibited. Results on thin films of resin are related to those from the surface resin layer of the composite.

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## EXPERIMENTAL

## Materials

The fiber glass-epoxy resin system, Scotchply 1009-26, is a seven-ply laminate (alternating 0° and 90°) of 73% Owens-Corning E-glass and 27% of a mixed epoxy resin. The 1009 resin composition is 67% by weight of a trifunctional epoxy novolac (DEN 438—Dow Chemical Company) and 33% by weight of a diglycidyl ether of bisphenol A (Epon 828—Shell Chemical Company) with 3% by weight of boron trifluoride-monoethylamine complex curing agent. The resin composition is summarized below (where  $n$  equals 0.2 for Epon 828 and 1.0 for DEN 438):



The laminates were cured under a pressure of 50 psi at 164°C for 45 min and postcured for 4 hr at 177°C in a forced-air oven. Thin films of the resin components were prepared under identical conditions but without the application of pressure. All stabilizer systems were commercial materials and used as obtained. The UV-absorbing epoxy (UVE) evaluated as a surface screener was synthesized by condensing 2,2',4,4'-tetrahydroxybenzophenone with excess epichlorohydrin.<sup>5</sup>

The product was a viscous liquid that crosslinked on heating. Excess epichlorohydrin was removed by vacuum distillation, the salt formed precipitated in acetone, and the resin extracted with ethanol. UVE showed an ultraviolet absorption spectrum characteristic of 2-hydroxy-4-alkoxybenzophenones, and IR data confirmed that at least one of the hydroxyl groups in the 2-position was not epoxidized. GPC analysis in tetrahydrofuran indicated only one prominent condensation product; and from the  $\bar{M}_n$  determined by VPO of 352, the epoxy is presumed to be mostly diglycidyl ether with possibly some triglycidyl ether. This is confirmed by the epoxy group value of 2.1 per molecule by IR analysis.<sup>6</sup> Other glycidyl ethers of the 2-hydroxybenzophenone UV absorber class were prepared but possessed unsuitable light absorption properties.

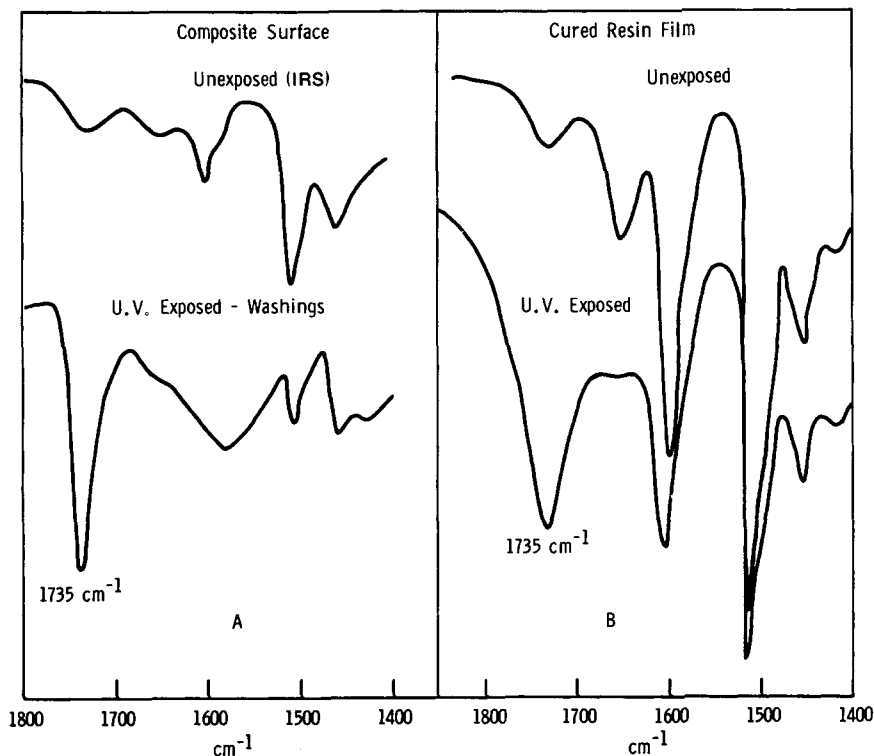


Fig. 1. (a) IRS-IR spectrum of unexposed 1009-26 composite surface and transmission IR of surface washings from composite surface exposed 4000 hr to sunlamp. (b) Transmission IR of air-cured 1009 resin film before and after 55 hr of exposure to sunlamp.

### Spectroscopic Studies of Cure and Photo-oxidation

The changes in the epoxy resins on cure were followed by transmission IR and UV spectrophotometry on thin sections heated in a forced-air oven, vacuum oven, or directly in a heated cell compartment in which the sample temperature and atmosphere could be controlled. A Fourier transform IR spectrometer (Digilab FTS-10M) with a single-pass internal reflectance attachment (Harrick Model TPMRA-P) was used for surface studies of the composite and resin slabs. The internal reflection element was KRS5, with angle of incidence equal to  $45^\circ$ . The same spectrometer was used for kinetic studies during short cure times. Thirty-two signal averages at  $4\text{ cm}^{-1}$  resolution produced excellent transmittance spectra from  $3800$  to  $450\text{ cm}^{-1}$  in less than 50 sec, while the number of signal averages was increased to 1024 for internal reflectance spectra of the composite to enhance the weak resin absorption.

Photo-oxidation studies were performed with a bank of 275-W sunlamps (General Electric CG401-E15 Code RS) that produced a dose rate as measured by a calibrated radiometer of  $4 \times 10^{-3}\text{ W/cm}^2$  (based on monochromatic radiation of 400 nm). The dose rate over the region of 300 to 350 nm, the wavelength region of maximum sensitivity of the resin, was  $1.02 \times 10^{-3}\text{ W/cm}^2$ . The sample temperature was  $35^\circ\text{C}$ .

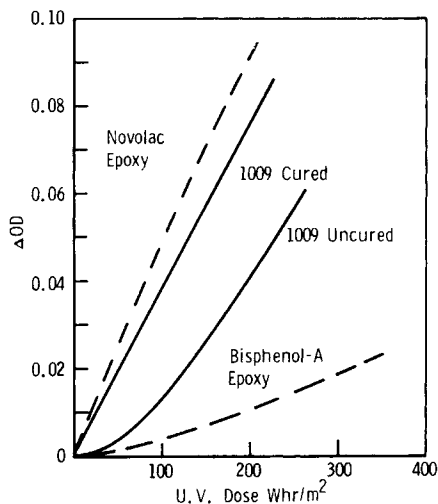


Fig. 2. Change in aliphatic carbonyl concentration measured by absorption intensity at  $1735\text{ cm}^{-1}$  ( $\Delta OD$ ) with total UV dose from 300 to 350 nm for the resin systems indicated.

## RESULTS AND DISCUSSION

### Analysis of Surface Resin of Composite

After outdoor exposure for 12 months or more, the surface layer of resin of the composite is depleted. Thermogravimetric and IR-IR studies have shown that the rate of fiber blooming is increased when the surface is exposed to water either as rain or a periodic water spray in addition to sunlight.<sup>3</sup> When the composite is exposed to solar radiation alone or a UV lamp without water spray, the resin layer can be retained for analysis.

A composite panel that had been exposed for 4000 hr under the sun lamps (total UV dose of  $41\text{ kWhr/m}^2$ ) was washed with distilled water and the organic material recovered. The IR spectrum showed a strong ester carbonyl band at  $1735\text{ cm}^{-1}$ , aliphatic hydrocarbon bands at  $2860$  and  $2930\text{ cm}^{-1}$ , an ether band at  $1250\text{ cm}^{-1}$ , and only weak aromatic bands. Mass-spectral analysis gave a highest mass of 340 for the ester and 264 for the hydrocarbon. In Figure 1(a), the IR spectrum in the region  $1800\text{--}1400\text{ cm}^{-1}$  is shown for the surface of the unexposed composite and the material recovered from the photodegraded panel. In Figure 1(b) are shown the IR spectra for a  $5\text{-}\mu\text{m}$  film of cured resin before and after UV exposure.

The prominent feature is the growth in the aliphatic ester group at  $1735\text{ cm}^{-1}$  with exposure time, and this has been used as an index of photo-oxidation. While the UV exposure times in these studies of the resin films are much less than the total dose in Figure 1(a), it is assumed that the oxidation rates measured are directly related to the rate of formation of low molecular weight oxidation products at the surface.

### Epoxy Resin Photo-oxidation

Thin air-cured sections of the 1009 resin and its components, Epon 828 and DEN 438, were exposed to UV and the change in the  $1735\text{ cm}^{-1}$  oxidation band

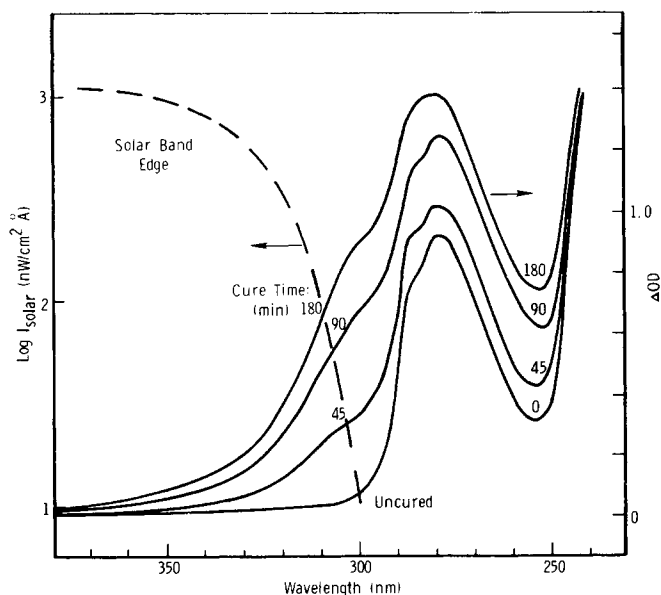


Fig. 3. Changes in the UV absorption spectrum of 1.5- $\mu\text{m}$  film of 1009 resin during cure in air at 165°C for the times shown. The reported solar spectrum in this region is also shown.

measured periodically. This is shown in Figure 2, and the observed oxidation rate for the epoxy novolac, DEN 438, is eight times that of the bisphenol A epoxy, Epon 828, and dominates the photo-oxidation of the 1009 system. Also shown is the photo-oxidation curve for an uncured film of 1009 resin of the same thickness. The oxidation kinetics are clearly different at short exposure time, and it appears that the high photo-oxidation rate of the cured epoxy novolac is related to the cure process itself. In particular, the initiation process for photo-oxidation has been changed by cure, and the possible reason for this is considered in the following section.

### Solar Radiation Absorption by the Cured Resin

In Figure 3 is shown the UV absorption spectrum of a 1.5- $\mu\text{m}$  film of 1009 resin before cure, and after cure times of 45, 90, and 180 min at 165°C. Also shown is the reported solar spectrum in this wavelength region.<sup>7</sup> The uncured resin showed negligible absorption of solar radiation, whereas on heating at 165°C for a short time, there is the formation of an intensely absorbing chromophore. The concentration of this species formed depends on the temperature and duration of cure. If the cure is carried out under vacuum, chromophore formation is minimized. When the separate epoxy resin components of 1009 were studied, it was found that only the epoxy novolac showed this band formation. Epon 828 showed no change in UV absorption after heating at 177°C for up to 6 hr.

Ultraviolet irradiation of the cured 1009 resin film for 10 min resulted in a loss of chromophore. With a fully postcured system, there is a decrease in the absorption intensity from 300 to 350 nm on irradiation, followed by an increase after several hours of further dose. On prolonged irradiation, the absorption band edge extends to 400 nm, leading to yellowing of the film.

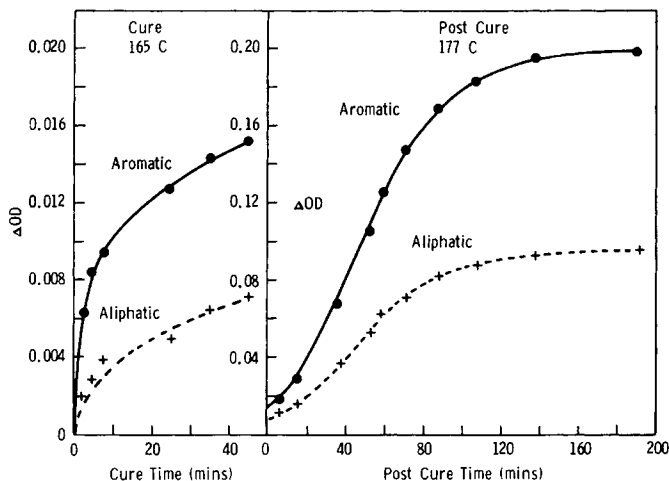


Fig. 4. Rate of formation of aromatic and aliphatic carbonyl bands in the IR during cure and postcure of 1009 resin. Note the difference in ordinates for cure and postcure.

### Changes in the IR Spectrum During Cure

During the air cure process, the principal changes in the IR spectrum are (i) a decrease in intensity of the epoxide group frequency at  $910\text{ cm}^{-1}$ ; (ii) an initial increase in OH absorption at  $3500\text{ cm}^{-1}$ ; (iii) the appearance of a strong aromatic carbonyl band at  $1655\text{ cm}^{-1}$  and a weaker aliphatic carbonyl at  $1735\text{ cm}^{-1}$ ; (iv) a change in both intensity and band position of the  $1610\text{-cm}^{-1}$  aromatic band.

Observations (i) and (ii) result from epoxide ring opening during cure and are also observed under vacuum or in an inert atmosphere. Changes (iii) and (iv) are due to an oxidation process during cure and are not observed if cure is carried out under vacuum. The oxidation bands are obvious in the spectra of both the commercial composite panels and the laboratory-cured resin films shown in Figure 1. The aromatic carbonyl band is of a lower frequency than expected for a simple aromatic ketone and is characteristic of an hydroxy-substituted benzophenone. Studies of the oxidation of an analogous phenol formaldehyde resin at temperatures from  $140^\circ$  to  $200^\circ\text{C}$  by Conley et al.<sup>8,9</sup> had shown that the methylene bridge is preferentially oxidized to give substituted benzophenones. The changes in the  $1600\text{ cm}^{-1}$  region arise from polarity changes in substituents attached to the aromatic nucleus. Replacement of methylene with carbonyl after extended oxidation produces the observed increase in intensity at  $1600\text{ cm}^{-1}$ .

The rate of growth of the oxidation bands during cure and postcure of the 1009 system is shown in Figure 4. After extended oxidation of the bisphenol A epoxy, a weak aliphatic carbonyl absorption band is observed, and it has been found that the aromatic carbonyl arises only from oxidation of the DEN 438 resin. The aromatic carbonyl arises only from oxidation of the DEN 438 resin. The intense UV absorption properties of hydroxybenzophenones explains the marked changes observed in the UV absorption spectra of the 1009 resin system on cure (Fig. 3) that were shown to arise from the epoxy novolac component of the resin on curing in air.

The rate of oxidation of the epoxy novolac was measured at temperatures from  $140^\circ$  to  $200^\circ\text{C}$  and compared with the rate of methylene bridge oxidation of

phenol-formaldehyde resins and a polybenzyl model system as reported by Conley.<sup>9</sup> Both aliphatic and aromatic carbonyl groups are formed. At 200°C, the rate constant determined by a Guggenheim analysis of data obtained as in Figure 4 was  $0.0058 \text{ min}^{-1}$ . This agrees with the reported oxidation rate of polybenzyl at this temperature ( $0.0052 \text{ min}^{-1}$ ) and a fully cured phenolic resin ( $0.0056 \text{ min}^{-1}$ ). Oxidation of the epoxy novolac at 145° and 165°C, however, produced a nonlinear Guggenheim plot, suggesting that pseudo-unimolecular oxidation kinetics are not observed.

Similar results were obtained by Conley for a partly cured phenolic resin, and the analysis of the fast component of the nonlinear plot produced a rate constant at 145°C of  $0.01 \text{ min}^{-1}$  and at 165°C of  $0.02 \text{ min}^{-1}$  for the epoxy novolac resin studied here, in close agreement with the reported rates for the partly cured phenolic resin.<sup>9</sup> The principal difference between the oxidation of the epoxy novolac resin without curing agent at 200°C and that at the lower temperatures was that the resin crosslinked at this temperature, producing an insoluble resin, while at the lower temperatures, no cure was observed. This high-temperature self-cure could result from residual unepoxidized methylol groups in the epoxy novolac that will open the epoxide ring.

The aliphatic carbonyl formation may result from extended oxidation of the methylene groups in the aliphatic ether crosslinks of the cured novolac and bisphenol epoxies. This does not produce the striking change in the light absorption property of the resin that results from the aromatic carbonyl formation.

### Inhibition of Oxidation During Cure

As indicated in the previous section, the oxidation can be largely inhibited by curing and postcuring under vacuum. Incorporation of an antioxidant in the resin before cure should inhibit aromatic and aliphatic carbonyl group formation. The efficiency of this process has been investigated for representative commercial chain-terminating and peroxide-decomposing antioxidants. A primary consideration when an additive is included in a resin formulation is any effect it may have on the rate and degree of cure. The representative antioxidants (Irganox 1010, Irganox 1076, Uvinox 3100, and DLTDP) were mixed at 1.0 wt-% with 1009 resin and the cure exotherm measured by DSC. Only Uvinox 3100 produced a change in cure kinetics and was found to produce resin cure when included in DEN 438 alone and heated to 165°C. [Irganox 1010 (Ciba-Geigy): tetrakis[methylene-3(3',5'-di-*tert*-butyl-4'-hydroxyphenyl)propionate]methane; Irganox 1067 (Ciba-Geigy): octadecyl-3-(3',5'-di-*tert*-butyl-4'-hydroxyphenyl) propionate; Uvinox 3100 (GAF): mixed mono- and disubstituted tris-nonylphenyl phosphite; Carstab DLTDP (Cincinnati Milacron): dilauryl thiodipropionate.]

It was found that the peroxide decomposing antioxidants DLTDP and Uvinox 3100 were ineffective at inhibiting oxidation at concentrations from 0.01 to 1.0 wt-%, which was chosen as a practical upper limit. Of the two chain-terminating antioxidants, only Irganox 1010 produced effective stabilization over a reasonable concentration range; and even with this stabilizer, a concentration of 0.5 to 1.0 wt-% was required to inhibit aromatic carbonyl formation through the full cure cycle. The antioxidant both increased the time to initial carbonyl group for-

mation (minimum detectible limit 0.01%) and lowered the rate of carbonyl growth.

The data were not collected over a sufficient time span to permit full kinetic analysis for all concentrations. The induction period and relative oxidation rates (compared to zero additive concentration as 1.0) are summarized in Table I for an oxidation temperature of 165°C.

### Inhibition of Photo-oxidation of 1009 Resin

The known photosensitivity of substituted benzophenones (other than 2-hydroxy substitution) in hydrocarbon matrices suggests that a prominent initiation route could be photoreduction through the first excited triplet state of the aromatic carbonyl impurity center. Suppression of thermal oxidation by either vacuum cure or addition of an effective antioxidant should lower this photo-oxidation rate. To test this, the epoxy novolac DEN 438 was cured at 165°C with 3.0 wt-% boron trifluoride complex curing agent under the following conditions: (i) in a vacuum oven; (ii) in a forced-air oven; (iii) in a forced-air oven but with 1% Irganox 1010 included as antioxidant.

The formation of aliphatic carbonyl at 1735  $\text{cm}^{-1}$  with UV dose is shown in Figure 5. Vacuum oven cure lowers the photo-oxidation rate by less than a factor of 2, and the rate observed is close to that obtained for the uncured resin (Fig. 2). Clearly, there are other initiation processes occurring in addition to the aromatic ketone sensitization. The antioxidant (iii) produces a low initial-rate photo-oxidation, but the final rate is the same as in (i). It appears that Irganox 1010, in addition to preventing aromatic carbonyl formation during cure, also inhibits the initiation process observed in the vacuum-cured sample. The increase in photo-oxidation rate after 200  $\text{Whr/m}^2$  is due either to the consumption of the antioxidant or the photolysis of the hindered phenol itself. The intrinsic photochemical instability of phenolic antioxidants has been recognized as a limiting factor in their photo-oxidative protection efficiency.<sup>10</sup>

### Photoprotection by UV Screening

The initiation of the photo-oxidation of the epoxy resin occurs only in part through the aromatic ketone impurity centers, and conventional antioxidant confer only limited protection. Screening the resin from the active wavelength region of the solar spectrum by incorporation of a UV stabilizer with a high absorption coefficient in this region has been assessed as an alternative protective

TABLE I  
Effect of Irganox 1010 on the Oxidation of Epoxy Novolac Resin  
DEN 438 in Air at 165°C

Concentration, wt-%	Induction time, min	Relative oxidation rate
0	0	1.0
0.05	0	1.0
0.10	60	1.0
0.25	220	0.25
0.50	375	0.25
1.0	750	0.05



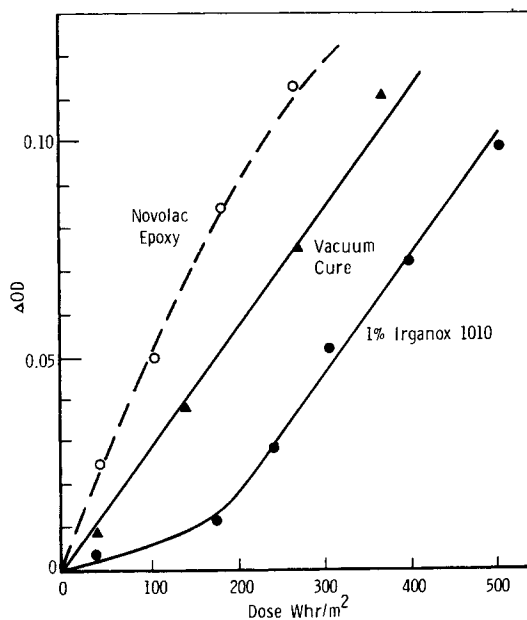


Fig. 5. Change in aliphatic carbonyl band ( $1735\text{ cm}^{-1}$ ) with UV exposure for epoxy novolac DEN 438 cured under vacuum and with 1% antioxidant (Irganox 1010).

technique. Typical UV stabilizers are the so-called UV absorbers such as 2-hydroxybenzophenones and 2-hydroxybenzotriazole derivatives which confer improved photostability to many thermoplastics at concentrations from 0.2 to 1.5 wt-%. The actual mechanism for photoprotection of these stabilizers is uncertain, largely because the observed protective efficiency in thin sections is often greater than the calculated protective efficiency on the basis of competitive UV absorption alone.

The theoretical protective efficiency declines rapidly with decrease in film thickness. This has been calculated in Figure 6 for protection of the cured 1009 epoxy resin by the UV stabilizer 2-hydroxy-4-isooctoxybenzophenone [Carstab 700L UV absorber (Cincinnati Milacron)]. This material is a liquid at room temperature that easily dissolved in the resin system and produced no change in cure kinetics as measured by DSC. The calculation was made on two separate bases: (i) Taking the measured UV spectrum of the air-cured resin over the range 300 to 350 nm; (ii) taking a limiting case of negligible radiation absorption by the cured resin as is observed when the resin is vacuum cured or contains an effective antioxidant.

The appropriate formulae and limitations have been summarized by Heller.<sup>11</sup> The protective efficiency ( $PE$ ) is defined as the residual light absorbed by the polymer photosensitive center in the presence of the stabilizer to that absorbed without the additive.  $PE$  thus varies from 0 for maximum protection to 1.0 for the unprotected resin.

The results of Figure 6 clearly show that the calculated maximum protective efficiency for a particular stabilizer concentration increases with film thickness, but does not exceed 0.8 for the maximum concentration and film thickness included in this study. Also shown are the observed experimental points for the inhibition of photo-oxidation by the UV stabilizer 2-hydroxy-4-isooctoxyben-

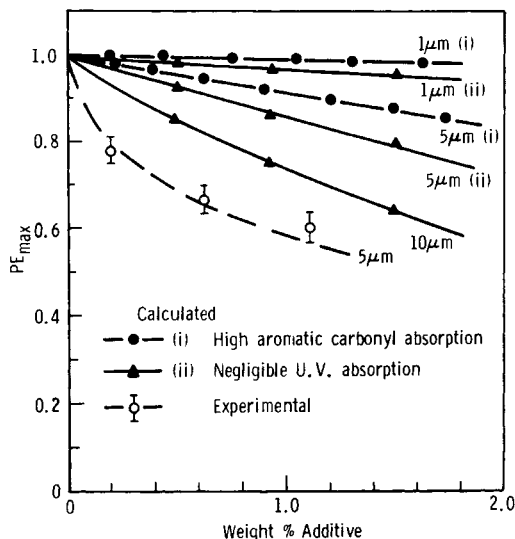


Fig. 6. Calculated and observed protection efficiency ( $PE$ ) for the UV absorber 2-hydroxy-4-isooctoxybenzophenone in 1009 resin of the thickness shown.

zophenone in 5- $\mu\text{m}$  films of air-cured 1009 resin. The UV stabilizer was found to partly inhibit the formation of the carbonyl groups during cure, but the observed protective efficiency curve still exceeds the calculated curve for negligible sensitizer absorption. This could result from some further antioxidant activity of the stabilizer during photo-oxidation or another of the secondary protective mechanisms that have been discovered for 2-hydroxybenzophenone stabilizers in polymers.<sup>12</sup> The overall protective efficiency is very poor in sections 2  $\mu\text{m}$  or less, i.e., the order of the surface layer of resin in the composite.

The limiting factor in the use of the UV absorber is frequently one of poor compatibility; and as the concentration increases above 1 or 2 wt-%, the additive may crystallize out. In addition, the screening is required to be greatest at the surface of the material to be protected, so that UV stabilizer in the bulk is wasted unless it can diffuse to the surface. For example, if the concentration of the UV stabilizer considered in the previous section could be increased to 50 wt-%, then the protective efficiency in a 1- $\mu\text{m}$  film would be 0.37 compared with 0.9 at 2 wt-%. For a 2- $\mu\text{m}$  film, the  $PE$  would be 0.2, i.e., a fivefold lifetime increase should be theoretically attainable.

This approach has been explored by synthesizing a 2-hydroxybenzophenone stabilizer with epoxide group substituents (UVE, see experimental section) and determining its screening efficiency and inherent photochemical stability. The UV absorbing epoxy could be readily cured with 3% boron trifluoride-monoethylamine complex and when cocured with Epon 828 formed a transparent, tough film. In order to assess the screening efficiency, a film of Epon 828 plus 50 wt-% UVE was cured on a UV-transparent slide. This gave an optical density at 370 nm of 1.4, equivalent to a pure UV absorbing epoxy film 1.5- $\mu\text{m}$  thick. The photo-oxidation rate of a film of air-cured 1009 resin under this coated slide compared to the photo-oxidation rate of a film under an uncoated slide gave a protective efficiency of 0.2. This assessment method was necessary because of the formation of a weak aliphatic carbonyl band in the pure UVE on extended exposure. This oxidation of the screening system possibly arises through trace

amounts of totally epoxidized tetrahydroxybenzophenone which would be photosensitive and initiate degradation. UV analysis of the GPC-fractionated polymer has failed to show this, but the inherent stability may be further improved by fractionation and purification. One further practical limitation to the use of this system is the self-cure of the resin. This is presumably catalyzed by pendent hydroxyl groups and can be followed in the early stages of room-temperature aging by broadening of the GPC curve to higher molecular weight. While low-temperature storage inhibits this, it is possible that the photoactive centers above are forming during self-cure on aging, or even on catalyzed cure.

### CONCLUSIONS

The results of this investigation suggest that the weathering stability of an epoxy resin can be affected by the conditions of cure. In particular, the methylene linkages of an epoxy novolac resin are thermally oxidized on cure and postcure in air to give aromatic carbonyl groups. These absorb strongly in the UV from 300 to 350 nm and can initiate photo-oxidation. Inhibition of thermal oxidation by curing under vacuum or including an antioxidant lowers the photo-oxidation rate. The thermal oxidation can be suppressed only by a high concentration of a chain-terminating antioxidant such as a hindered phenol, and the inherent photochemical instability of these antioxidants may then limit the photoprotection. Even when thermal oxidation is apparently inhibited, photo-oxidation of both the bisphenol A and novolac epoxies is observed. A UV stabilizer offers the best protection in this case. The normally low protective efficiency of a UV-absorbing additive can be overcome by including an epoxidized 2-hydroxybenzophenone in the surface layer of resin. High surface concentrations of the stabilizer are then possible, and the photoprotection is limited only by the inherent stability of the UV-absorbing epoxy.

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