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Photodegradation in Ballistic Laminates: Spectroscopy and Lifetime Extension

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Photodegradation in Ballistic Laminates: Spectroscopy and Lifetime Extension

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Abstract

Several years ago, we in the Materials and Process Sciences Center (Org. 1800) were asked by Dept. 9613 to study the materials aging issues which had led to the loss of ballistic protection by Armored Tractor (AT) windshields and windows. We speculated that this loss of impact strength was due to photodegradation of the polycarbonate (PC) inboard ply. We developed a spectroscopic method to identify changes in the outboard surface of the PC, and showed that the changes in the surface which occurred upon natural aging in the field could be reproduced by exposing the laminates to a simulated solar flux.

Based on these results, we recommended changes in the adhesive interlayers to filter out the ultraviolet (UV) light causing the aging problem. Working with the laminate vendor, PPG, we re-designed the laminates to implement these changes and block essentially all UV light from the inboard ply. The most recent phase of this work involved accelerated solar aging of laminates made with the new design to verify that photoaging effects have been blocked by the new materials. We report here the results of that study, and recommended follow-on work.

Contents

Introduction and Project History.....	1
Materials and Method.....	3
Results and Discussion.....	6
Conclusions.....	8
References.....	10

Tables

1 Transmittance at selected wavelengths.....	11
2 Detailed peak position data.....	11

Figures

1 Plot of wavelength vs. absorbance of laminates.....	14
2 A schematic drawing of the "seagull" reflectance accessory.....	15
3 The reflectance spectrum of PC.....	15
4 A series of absorption spectra of PC.....	16
5 Same series as in Fig. 4 with the frequency scale expanded around the carbonyl band.....	16
6 A scale expansion of two PC absorption spectra from Fig. 5.....	17
7 The carbonyl absorption bands for PC layer of unexposed and exposed ballistic laminate.....	17
8 A second example of the carbonyl absorption bands for the PC layers.....	18
9 The carbonyl absorption bands for the PC layers exposed in a solar simulator to the equivalent of about of 10yrs of sun exposure in a transport vehicle.....	18
10 The carbonyl absorption bands for the PC layers exposed in a solar simulator to the equivalent of about of 10yrs of sun exposure in a transport vehicle with no peak shift.....	19

Introduction and Project History

Several years ago, the Materials Center at Sandia (Org. 1800) was asked by Dept. 9613 to look at Armored Tractor (AT) ballistic laminates whose effective lifetime was being shortened by some type of materials aging. These laminates, produced by PPG to a Sandia design, consist of several layers of glass with polyvinylbutyral interlayers, and an inboard polycarbonate (PC) ply laminated to the inmost glass ply with a polyurethane interlayer.

The literature cites many studies in which PC is shown to degrade rapidly upon exposure to UV light (including solar exposure) [1-7]. In fact, we were suprised to learn that light-induced loss of ballistic protection by PC-based laminates is even documented in the popular literature [8]. For this reason, we felt it very likely that the loss of ballistic protection with time in service was due to the photodegradation of the PC ply. In order to test this idea, we developed (with Ken Ward, now separated from Sandia) an infrared (IR) spectrophotometric technique to measure differences in the outboard surface of the PC ply. We showed that there were definite, and reproducible, differences in these surfaces when new, compared to those returned from service. Further, we showed that these differences could be reproduced by accelerated exposure to a simulated solar spectrum. These results gave us a high level of confidence that the failed laminates were experiencing significant solar-induced aging.

These findings are consistent with our optical absorption measurements on laminates which had the PC layer removed. These spectra showed the fraction of light filtered from reaching the PC layer, as a function of wavelength. As summarized in Table I, these spectra showed that virtually no light with wavelengths shorter than 330 nm would reach the PC layer. However, substantial levels of 350 nm light would be passed by the outboard plies (20 %). The literature clearly shows that shorter wavelength light is much more efficient in degrading PC than is longer wavelength light. However, at least one paper demonstrates degradation of PC with light in the 350 nm range [7]. PC left to solar exposure with no protection will age at a rate giving it a useful life of less than 6 months [1] (depending on latitude and PC grade), even though solar radiation consists almost entirely of light with wavelengths greater than 300 nm. We believe the longer effective service life of our ballistic laminates is due to the partial filtering of the solar spectrum.

PC photodegrades only within the surface region of the outboard side, because it absorbs so strongly that essentially all the incoming light is taken up in that region. The resulting photo-induced surface degradation leads to surface microcracks which eventually cause loss of impact strength. Therefore, it is difficult, if not impossible to provide effective photoprotection to PC via absorbing additives in the PC ply itself. Rather, a protective UV screen must be incorporated into some layer on the outboard side of the PC. As mentioned above, the glass plies and adhesive interlayers act to partially filter incoming solar flux. However, more complete UV screening was required for service life extension. We recommended to PPG, the laminate vendor, that UV absorbers of the benzotriazole class (sold by Ciba-Geigy under the Tinuvin trade name) be investigated as additives to the adhesive layers. As it turns out, PPG already had extensive experience with adhesive layers which contained what we later determined to be benzotriazole absorbers. PPG was comfortable, based on prior experience, substituting these screened interlayers for the adhesives used previously. At Sandia's request, PPG produced samples of laminates incorporating this design change.

The original laminate design used a polyvinylbutyral (PVB) as an adhesive interlayer between the glass plies. A polyurethane was used between the glass layer closest to the inboard side and the PC. The interlayers substituted for these adhesives in the reconfigured design were designated by PPG as B14 (between the glass) and KPUR 301 (between the glass and PC). In order to evaluate the level of UV screening imparted by these new interlayers, optical absorption spectra were taken. However, the absorption levels found were too large to measure directly. Therefore, we took spectra using thin samples (0.03 in. for KPUR300 and 0.012 in. for B14) and scaled the absorption to application thickness. These results are shown in Fig. 1. In this figure, absorption due to the KPUR 301, the B14, and the sum of the two are plotted, as well as the corresponding spectrum for the previous (unscreened) configuration. As is clear from the figure, a laminate configured with these new interlayers gives virtually complete protection of the PC from UV light with wavelengths of 370 nm or shorter.

Initial testing showed that these new laminates met ballistics specifications. At that point, we began accelerated photoaging to increase our confidence that the new design would decrease the rate of photo-induced aging, as anticipated. What follows is a description of those results.

Materials and Method

The naturally exposed ballistic laminate samples were acquired from windshields that had been removed from tractors after a documented service duration. Accelerated photo-aging of unaged ballistic laminate samples was accomplished using the Sandia Photovoltaic Test Evaluation Lab (PTEL) Accelerator for Ultraviolet Aging originally assembled by Sandia Departments 6219 and 1812. All samples (both aged and unaged) were cut from the original windshields into 1 inch by 1 inch squares.

The mostly Oriel equipment comprising the Accelerator is housed in a well-ventilated, steel instrument cabinet-bench, with removable doors and space underneath for the water chiller/recirculators. The samples are held in place via vacuum chuck on a water cooled, thermocouple-monitored stage. The stage is mounted on one end of a precision equipment rail. At the opposite end of the rail is placed an equipment box housing the 1000 Watt Xenon arc lamp backed by a high efficiency ellipsoidal reflector; the original and reflected beam are directed through a collimating lens down the rail toward the sample. Since the lamp intensity will vary during a run and decreases as the lamp ages, a photo feedback controller samples the intensity and adjusts the power output to keep the intensity within a pre-set interval. There are interlocks that will shut down the system if the lamp burns out, if the temperature at the sample stage or in the lamp housing is too high, or if either of the two water chillers is not recirculating. The system will also shutdown if there is a power surge disrupting the ability of the power supply to sustain the arc or if a power outage occurs. There is an elapsed timer that stops whenever the system shuts down, providing a precise way to calculate actual exposure hours. Filters are mounted on the equipment rail between the lamp housing and the sample stage: a water cooled water bath absorbs the infrared wavelengths ($>700\text{nm}$). A Pyrex filter and colored glass filter ensure a sharp cut-on at 300 nm, mimicking the terrestrial solar spectrum.

The sample is placed in the collimated beam so that the entire 1x1 inch sample is irradiated. The distance between the sample stage and lamp housing is determined by the acceleration desired. The light intensity is measured by placing a calibrated spectroradiometer (Licor 1800) in the beam so that the radiometer lens is at the same location on the rail as the sample on the sample stage. The beam intensity, or spectral irradiance E_λ , was measured in discrete wavelengths between 300-400 nm. The total irradiance between 300-400 nm is the integral of the E_λ

curve from 300 - 400 nm. This area is divided by the one-sun integrated global solar spectral irradiance for 300-400 nm, 46.17, to give the number of suns the sample was exposed to. The number of suns times the total elapsed time, in hours, divided by 2920 solar hours per year (365 days x 8 hours/day) equals the total calendar years of artificial exposure.

Initial attempts at accelerated aging of the screened samples employed a 38 sun beam, which proved to be too intense to collect appropriate data. Specifically, in the new configuration (screened) sample, the UV absorbers in the first interlayer, just inside the outboard glass ply, absorbed virtually all the incoming light and became very hot. As a result, the first interlayer turned brown and blistered. The glass was unaffected. The remainder of the sample was unaffected due to a combination of a cooling effect of the sample stage and the bulk of the UV light being absorbed at that first interlayer.

A second new configuration sample was exposed to the same total number of solar years at an average of 21.1 suns for 8.1 weeks (lower intensity for longer time) by moving the sample stage farther away from the lamp housing and lowering the lamp output. This change lowered the heat load enough that no discoloration or blistering was observed.

Both virgin and irradiated samples were disassembled for spectroscopic analysis by the following method. First, the 1 in. by 1 in. ballistic laminates were firmly secured in a vise. Vise-grips were used to twist the PC layer from the outer layers. If the relatively thick adhesive layer happened to remain with the PC layer, it was peeled off with pliers.

The outboard PC surface was examined spectroscopically. Fig. 2 shows the reflectance accessory that was placed in the sample chamber of a Nicolet 800 FTIR instrument for this purpose. Since one can readily vary the incident and exit angles, data were collected for several angles between 80° and 10° from normal. However, only 10° data are presented since these reflectance spectra, after the Kramers-Kronig transformation, are the most similar to true absorption spectra. Typically about 500 scans were averaged to produce a spectrum. The instrument resolution was set to 4 cm⁻¹ and a Mercury-Cadmium-Telluride detector was used. The data in Fig. 7, only, were obtained in an earlier study in the same laboratory using a Nicolet 7000 instrument and a Spectra-Tech IR Plan microscope in a reflectance mode. The incident angle is not so well controlled in the later apparatus and is a complex average of angles from about 0° to 45°.

Fig. 3b shows the reflectance spectrum of the PC surface after it is put on an absorbance-like scale by the usual calculation, $-\log S/R$, where S is the sample scan and R is the reference scan. The reference scan is acquired by placing a mirror in place of the sample in Fig. 2. Fig. 3 also shows the result of the Kramers-Kronig transform which produces a spectrum essentially equivalent to a true absorption spectrum. In fact, Fig. 3b is hardly distinguishable from a true absorption spectrum (not shown). The transformation was accomplished with the routine distributed with Lab-Calc by Galactic Industries. A normal incidence is assumed, hence the superior results noted above when near normal incidence was used.

The method described produces a spectrum which is essentially equivalent to the absorption spectrum of a thin layer at the surface of the sample, where the thickness is on the order of the wavelength of the light. Since the damage due to UV exposure of polycarbonate is surely a surface effect (*vide supra*), it would be useless to study the spectral changes in the bulk of the PC layer. Note that the use of the term *absorption spectrum* in this work indicates a spectrum acquired by the above methods and not true absorption spectra acquired in the traditional manner.

Exposures of the bare PC were accomplished with a 50 watt mercury lamp (Oriel) which was coarsely collimated with a fused silica condenser lens. The one inch square sample was placed in the center of the beam at a distance of one foot from the end of the lens housing. Careful quantitation of the exposure dose was not deemed necessary since the purpose of this phase of the work was simply to discover the type of spectroscopic changes to be expected upon exposure to UV light.

Results and Discussion

Photo-degradation of bare PC

Fig. 4 shows the evolution of the absorption spectrum of bare PC after exposure to a mercury lamp for the indicated time. The absorption bands drop in intensity and shift to lower energy with pronounced broadening. The spectra of Fig. 4 are not normalized but retain their original relative intensity information. In Fig. 5 the frequency scale in the neighborhood of the carbonyl stretching band is expanded. Fig. 5 contains the same spectra as in Fig. 4, with the spectra rescaled to a peak height of unity. The spectra for 0 and 4 minutes exposure are indistinguishable on this scale. The broadening and shifting of the peak to lower energy are very apparent for the longer exposures. These spectral shifts indicate a photo-induced weakening of at least some of the C=O bonds from chemical or morphological changes. Fig. 6 is a further frequency scale expansion of the PC spectra. Only the sample after 0 and 4 minute exposure are shown. The difference spectrum (unexposed - exposed) clearly shows that the change is principally a peak shift. This case of low exposure of bare PC is of interest because it shows a small change essentially equivalent to that seen below for much longer exposures of the PC in ballistic laminates.

Natural exposure of ballistic laminates

Figs. 7 and 8 show spectra from two different windshields that were in service for a number of years. The unexposed samples were from windshields stored in light-tight containers and manufactured at about the same time. As before, the difference spectra (unexposed - exposed) are displayed. The spectra presented in the Figs. 7 and 8 (also, Figs. 9 and 10) are the average (after baseline subtraction and normalization to unity) of a number of independent acquisitions of the spectrum of each sample. The peak positions and standard deviations (about 10 repetitions) are reported in Table II. The shift seen in Fig. 6 is reproduced in both Figs. 7 and 8. The fact that we see the same type of change for the naturally exposed PC as the rapidly exposed bare PC is a strong indication that the change in the former is principally due to exposure to light.

Artificial exposure of ballistic laminates (including effect of UV blocker)

Fig. 9 is a repeat of the experiments of Figs. 7 and 8, where the exposure was accelerated with a solar simulator. It is different from the experiment of Fig. 6 in

that the PC was part of a ballistic laminate when exposed, and the exposure spectrum and dose were carefully controlled. After a simulated 10 years of exposure we see a similar change as in Figs. 7 and 8. This verifies that the solar simulator is a valid method for acquiring data that is intended to represent natural exposures.

The primary focus of this work is to demonstrate the effectiveness of the UV blocker incorporated in what is known as the new configuration of the ballistic laminates. Fig. 10 presents data for the new configuration obtained in the same manner as Fig. 9 for the old configuration. The peak shift which is firmly associated with photo-degradation in Figs. 6 to 9 is absent in this sample. The two negative lobes in the difference spectrum are associated with a peak broadening, the origin of which is unknown.

Conclusions

We have shown that UV photodegradation of the PC surface produces a characteristic shift in the carbonyl absorption band at about 1775 cm^{-1} . This shift is consistently observed in PC samples that have been artificially exposed directly (Figs. 4 to 6), and both naturally (Figs. 7 and 8) and artificially (Fig. 9) exposed through thick glass. When a UV blocker is incorporated into the laminate, no peak shift is observed in the carbonyl band after 10 years equivalent exposure (Fig. 10). This is strong evidence that the expected protection against UV photodegradation is a reality and that the lifetime of ballistic laminates, when limited by this type of degradation, can be extended.

We should point out that, while Table II shows a consistent shift in the carbonyl band upon UV exposure, the unshifted peak position is sample-dependant. As a result, the degree of peak shift resulting from exposure can only be determined if a good control is available. This means that UV exposure cannot be inferred from data on old samples with many years in service, unless a very similar, but unexposed, sample is available for comparison.

Our simulation of 10 years service life has uncertainty because of differences in how each AT is deployed. However, we believe our estimate of 10 year equivalent exposure is conservative, for the following reasons: 1) ATs in service always spend at least part of the time inside, protected from solar exposure (e.g., during maintenance); 2) ATs in service spend part of the time facing away from the sun (e.g., when driving north or when parked facing north); and 3) our simulated exposures were always at normal incidence to the laminate, whereas the vertically mounted windshields will spend most of the time off-normal to the sun, resulting in a lower effective flux. For these reasons, we believe our 10 year simulations resulted in greater light exposure than will ever be seen by AT windshields in service over a 10 year span.

The photo-aging of these laminates may be accelerated by alternating light and dark cycles, that result in thermomechanical stresses, which we did not simulate. However, our spectroscopic results indicate no photo-induced changes at the PC surface, consistent with the extremely high level of UV screening provided by the new laminate configuration. At least some photoflux must reach the PC layer in order for any photo-aging to occur; therefore, we believe the absence of on/off cycles in our simulation does not represent a flaw in the methodology. Additionally,

we should note that thermally-induced stresses in the glass layers will not be transmitted to the PC, due to presence of the intervening elastomeric layer.

We believe, based on the results described above, that photo-induced aging will not occur in laminates made in the new UV-screened configuration over a 10 year service life. However, we recommend the following actions be undertaken: 1) repeat the above accelerated tests to whatever maximum service life these windshields may see (e.g., 13 years); 2) institute an ongoing program of ballistics testing (and possibly spectroscopic evaluation) using windshields removed from service at incremental intervals; 3) consider follow-on studies of other potential aging mechanisms (we anticipate that moisture-induced degradation will be the next most important aging mode); and 4) institute simple procedures to minimize windshield exposure to the elements (e.g., park ATs facing north and cover the windshields when not in use).

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Table I

Transmittance at selected wavelengths for light absorption by ballistic stackup with PC layer removed. Data shown are for original design, without supplemental UV screening. UV screened data are shown in Fig. 1.

Wavelength (nm)	Absorbance	% Transmittance
330	2.85	0.14
335	1.94	1.2
340	1.35	4.5
345	0.96	11
350	0.7	20

Table II

Detailed peak position data in units of cm^{-1} . Peak positions are determined by the algorithm distributed with Grams 3.0 by Galactic Industries. There were about 10 independent repetitions of the spectral acquisition in each case except for the bare PC for which there were no repetitions.

<u>Light Source</u>	<u>Sample</u>	<u>Unexposed</u>	<u>Std.Dev.</u>	<u>Exposed</u>	<u>Std.Dev.</u>	<u>Delta</u>
			<u>Unexposed</u>		<u>Exposed</u>	<u>Unexposed-Exposed</u>
Hg Lamp	Bare, Fig. 6	1773.86	NA	1773.37	NA	0.49
Natural	Old Config. #1, Fig. 7	1780.97	0.12	1780.21	0.28	0.76
Natural	Old Config. #2, Fig. 8	1775.17	0.12	1774.95	0.07	0.22
Solar Simulator	Old Config., Fig. 9	1774.52	0.23	1773.84	0.04	0.68
Solar Simulator	New Config., Fig. 10	1773.84	0.12	1773.88	0.42	-0.04

Fig. 1. Plot of wavelength (nm) vs. absorbance of previous (unscreened) laminate configuration and new (screened) laminate configuration. The new laminate configuration absorbance at each wavelength was obtained by adding up the KPUR300 and B14 absorbances at application thickness for that wavelength.

Fig. 2. A schematic drawing of the "seagull" reflectance accessory. The mirrors can be adjusted in concert to give different reflection angles. An angle of 10° off of normal was used.

Fig. 3. The reflectance spectrum of PC, where the usual calculation ($-\log S/R$) is made to convert the sample and reference scans to absorbance. The reference scan is taken with a mirror in place of the sample in Fig. 1. b. The reflectance spectrum is converted to an absorption spectrum by the fcc transform.

Fig. 4. A series of absorption spectra of PC, showing the effect of exposure to a 50 watt mercury lamp.

Fig. 5. The same series of PC absorption spectra as in Fig. 4. The frequency scale has been expanded around the carbonyl band, and all peak heights have been normalized to unity. The absorption spectra for 0 and 4 minutes are indistinguishable on this scale.

Fig. 6. A scale expansion of two PC absorption spectra from Fig. 5. The absorption bands for 0 and 4 minutes of exposure are shown, along with their difference (unexposed -exposed). The 4 minute exposure produces a change in unprotected PC that is similar to much longer exposures of PC in ballistic laminates. A baseline, drawn from 1820 to 1745 cm^{-1} , is subtracted from the spectra and the peak heights are normalized to unity.

Fig. 7. The carbonyl absorption bands for the PC layer of unexposed and naturally exposed ballistic laminate. The difference (unexposed-exposed) is also shown. A baseline, drawn from 1820 to 1745 cm^{-1} , is subtracted from the spectra and the peak heights are normalized to unity. Exposure is a few years of installation in a transport vehicle. These spectra, only, were obtained with an IR microscope in reflectance mode as explained in the Materials and Methods section.

Fig. 8. A second example of the carbonyl absorption bands for the PC layer of unexposed and naturally exposed ballistic laminate. The difference (unexposed-exposed) is also shown. A baseline, drawn from 1820 to 1745 cm^{-1} , is subtracted

from the spectra and the peak heights are normalized to unity. Exposure is a few years of installation in a transport vehicle.

Fig. 9. The carbonyl absorption bands for the PC layer of unexposed and artificially exposed ballistic laminate. The difference (unexposed-exposed) is also shown. A baseline, drawn from 1820 to 1745 cm^{-1} , is subtracted from the spectra and the peak heights are normalized to unity. Exposure is a few weeks in a solar simulator and equivalent to about 10 years in a transport vehicle.

Fig. 10. The carbonyl absorption bands for the PC layer of unexposed and naturally exposed ballistic laminate (new configuration). The difference (unexposed-exposed) is also shown. A baseline, drawn from 1820 to 1745 cm^{-1} , is subtracted from the spectra and the peak heights are normalized to unity. Exposure is a few weeks in a solar simulator and equivalent to about 10 years in a transport vehicle. Although the spectra do not precisely overlay (see text), there is no peak shift, indicating that the effect of exposure seen in Figs. 6 to 9 is absent.

Fig. 1.

Fig. 2.

Fig. 3.

Fig. 4.

Fig. 5

Fig. 6.

Fig. 7.

Fig. 8.

Fig. 9.

Fig. 10.

Distribution

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