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ACCELERATED WEATHERING TEST FOR GLASS-FIBER REINFORCED
POLYESTER (GRP) SHEETS

by R.S. Yamasaki and A. Blaga

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SOMMAIRE

Une étude détaillée des méthodes d'essai et des critères d'évaluation des nouveaux matériaux employés pour les toitures est décrite: les feuilles de polymères préfabriqués et d'élastomères, des membranes d'asphalte modifié préfabriquées et des membranes liquides appliquées. Les résultats résumés se rapportent à un certain nombre de matériaux choisis en raison de leur représentativité des trois groupes en vue de démontrer les restrictions et les problèmes inhérents aux divers essais.

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Accelerated Weathering Test for Glass-Fiber Reinforced Polyester (GRP) Sheets

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ABSTRACT: With the development of GRP's of improved durability to weathering it has become necessary to devise accelerated weathering tests that permit assessment of their performance and durability within a reasonable time. The new artificial weathering procedure subjects GRP materials to a cycle of 2 h xenon arc radiation (twice the intensity used in the previously reported cycle of 4 h radiation and 4 h cold water spray) and 0.67 h cold water spray. Exposure of fire-retardant, non-gel-coated, and conventional GRP sheets induced surface degradation similar to that produced by both the 4-4 cycle and outdoor exposure in a considerably shorter time. The acceleration rate was 2.0 to 3.5 (compared with the 4-4 cycle) and 5 to 30 (compared with outdoors).

KEY WORDS: accelerated weathering test, building materials, durability, surface degradation, fiber pop-out, surface microcracking, glass-fiber reinforced polyester, fire-retardant GRP, non-gel-coated GRP, conventional GRP

An artificial weathering procedure with a cycle of 4 h of xenon arc radiation followed by 4 h of cold water spray in a Weather-Ometer² (4-4 cycle) has been useful in studies of deterioration mechanisms during weathering of various glass-reinforced polyesters (GRP) [1-4].³ It produces a sequence of surface damage similar to that occurring outdoors. In addition, the 4-4 cycle accelerated such surface degradation, reducing the test period to about a year. To reduce the test period further and to permit, within a reasonable time, the study of weathering behavior of GRP's of improved durability, an artificial weathering procedure to accelerate degradative effects of the 4-4 cycle has been developed.

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²Trademark of Atlas Electric Devices Co.

³The italic numbers in brackets refer to the list of references appended to this paper.

Development of the Accelerated Weathering Cycle

Exposure of conventional (produced by hand lay-up process) [1,2] and other generic types [3,4] of GRP to the 4-4 cycle or outdoor conditions results in two main types of surface deterioration: fiber pop-out, which refers to the process leading to the exposure of the glass fibers, and surface microcracking of the resin. The proposed cycle consists of 2 h xenon arc radiation at twice the average ultraviolet (UV) intensity (340 nm) of the 4 h period of the 4-4 cycle so as to maintain the same total UV irradiance per cycle. This is followed by 0.67 h of cold water spray rather than 4 h as in the 4-4 cycle to shorten the duration of each cycle even more. It was found that the amount of moisture sorbed by a specimen of conventional GRP (made by a continuous process) was similar in the short cycle (0.12 percent water sorbed) to that in the long cycle (0.15 percent). The number and rate of thermal shocks applied to a GRP panel when cold water (9°C) strikes the heated panel (55°C), as well as the extreme temperatures to which it is subjected were the same in each cycle. Thus, each 2-0.67 cycle should produce a weathering effect equivalent to each 4-4 cycle in one third the time—an acceleration factor of three for the same duration of exposure.

Experimental

Materials

Three types of commercial GRP, fire-retardant, non-gel-coated, and conventional, were selected because they display a wide range of durability when exposed to the 4-4 cycle. Detailed descriptions of the materials are presented in Table 1.

Apparatus

GRP panels (165 by 57 by 1.5 mm) were subjected to artificial weathering in a xenon arc Weather-Ometer (Atlas Model 600-WR) using a 6000-W water-cooled xenon arc burner with borosilicate inner and outer filters to provide radiation with UV and visible bands closely simulating those of sunlight [5]. The Weather-Ometer was equipped with a light monitor and control device (Atlas Model A3F 60-600) to control and record the intensity of the UV (340 nm) at $0.46 \text{ (W/m}^2\text{)}/\text{nm}$. This is still lower than the $0.55 \text{ (W/m}^2\text{)}/\text{nm}$ at 340 nm observed outdoors at Chicago [5]. The original specimen rack in the weathering machine was modified so that GRP panels could be mounted along the horizontal, central axes of the rack where the radiation intensity corresponds to the intensity reading on the monitoring device. This procedure minimizes the difference in radiation intensity from the middle to the ends of a panel because the horizontal central axes of the

TABLE 1—Description of GRP sheet material.

Characteristics ^a	Composition ^b
Fire-retardant, made by continuous process	Sheet composed of 75% resin and 25% glass reinforcement. ^c Resin consists of 70 parts thermosetting, tetrachlorophthalic acid-modified, light-stabilized, unsaturated polyester crosslinked with 30 parts styrene.
Non-gel-coated, made by hand lay-up technique	Sheet composed of 75% resin and 25% glass reinforcement. ^c The resin consists of 60 parts thermosetting, UV stabilized, general purpose polyester, cross-linked with 40 parts of styrene using MEK and cobalt naphthenate without heating.
Conventional, made by continuous process	Sheet composed of 75% acrylic-modified resin and 25% glass reinforcement. ^c The resin consists of 60 parts thermosetting, UV stabilized, general purpose polyester, cross-linked with 40 parts of a 3:1 mixture of styrene and methyl methacrylate at 110 to 140°C.

^aFlat, smooth finish surface, colorless, and translucent sheets (1.5 mm thick).

^bAll compositions expressed by weight.

^cSilane-treated glass-fiber (E-glass) in the form of chopped strand mat.

burner, where the radiation intensity is greatest, coincide with the middle of each panel. During arc-off the test face of GRP panels was subjected to cold (9°C) deionized water spray from an F-80 nozzle at a flow rate of 4 litres/h.

Examination of Surface Degradation of GRP's by Scanning Electron Microscope

Surface degradation was assessed with a Stereoscan⁴ scanning electron microscope (SEM) operated at 20 kV and a tilt angle of 45 deg. GRP specimens cut from sheets were prepared for SEM examination by ultrasonic cleaning prior to coating them with carbon and gold to render them conductive.

Procedure

Fire-retardant, non-gel-coated, and conventional GRP specimens were exposed, to 2-0.67 and 4-4 cycles in the weathering machine and outdoors, as described in Table 2. The temperature-humidity conditions in both 2-0.67 and 4-4 cycles are given in Fig. 1.

At appropriate intervals 15 by 15-mm specimens were cut with a portable grinding tool fitted with a 20 by 1-mm grinding wheel and surveyed with an optical stereomicroscope prior to SEM examination. The nature and rate of surface degradation induced by exposure were monitored throughout the exposure period.

⁴Trademark of Cambridge Instrument Co., Inc.

TABLE 2—Exposure methods for GRP sheets.

Method No.	Exposure Conditions	
	General	Details
1	Cyclic variation of radiation, temperature and moisture (xenon arc Weather-Ometer) 2-0.67 cycle	2 h xenon arc radiation, intensity 0.46 (W/m^2)/nm at 340 nm, 55°C panel temp, 50% RH, waterspray off, 0.67 h cold water (9°C) spray, 12°C panel temp, 100% RH, radiation off 9 cycles/day
2	Cyclic variation of radiation, temperature and moisture (xenon arc Weather-Ometer) 4-4 cycle	4 h xenon arc radiation, average intensity 50% of that of 2-0.67 cycle, 55°C panel temp, 50% RH, waterspray off, 4 h cold water (9°C) spray, 12°C panel temp, 100% RH, radiation off, 3 cycles/day
3	Outdoor exposure at Ottawa (humid continental climate)	panels exposed horizontally on a test rack with no backing (ASTM D 1435) ^a

^aASTM Recommended Practice for Outdoor Weathering of Plastics (D 1435 - 75).

FIG. 1—Temperature and humidity conditions during exposure of GRP to 2-0.67 and 4-4 cycles in the xenon arc Weather-Ometer.

Results and Discussion

Fire-Retardant GRP

Exposure of fire-retardant GRP to the 2-0.67 cycle resulted in surface deterioration with characteristics similar to those occurring from exposure to both the 4-4 cycle and outdoor weathering at Ottawa [3]. In both of these,

deterioration was characterized by five distinct types of surface damage. Thus, specimens were examined carefully for the same features following the two accelerated cycles plus outdoor exposure. The five stages are illustrated in Figs. 2 to 6.

The first surface change to occur was fiber ridging, in which the resin layer formed ripples or round ridges above glass filaments near the surface. Figure 2 shows that fiber ridging occurred in all three exposures. The next damage

FIG. 2—Fiber ridging of fire-retardant GRP produced by artificial and outdoor weathering.

FIG. 3—Cracking of resin adjacent to fibers of fire-retardant GRP.

(a) 2-0.67 cycle, 39 days exposure
 (b) 4-4 cycle, 135 days exposure
 (c) Outdoors, 184 days exposure

FIG. 4—Fiber pop-out of fire-retardant GRP.

- (a) 2-0.67 cycle, 39 days exposure
- (b) 4-4 cycle, 135 days exposure
- (c) Outdoors, 1095 days exposure

FIG. 5—Isolated surface microcracking of fire retardant GRP.

(a) 2-0.67 cycle, 78 days exposure
 (b) 4-4 cycle, 267 days exposure
 (c) Outdoors, 1460 days exposure

FIG. 6—Network of surface microcracking of fire-retardant GRP.

feature was characterized by the resin layer in the ridges rupturing in a brittle mode, as evidenced by jagged fracture surfaces (Fig. 3). In the third stage, the glass fibers became essentially free of the resin matrix, producing the so-called fiber pop-out. Figure 4 illustrates that fiber pop-out was common to all three tests. Isolated surface microcracks in the resin were features of the fourth type of surface damage. It may be seen in Fig. 5 that cracks are oriented randomly, V-shaped in cross section, and comparable in dimensions in specimens from all three exposures. Finally, the fifth stage was characterized by a network of microcracks [2], as shown in Fig. 6. Thus, degradation of fire-retardant GRP was similar for the three exposures: all five types of damage occurred in the same sequence and with similar appearance at each stage.

The rates at which surface degradation proceeded in the three exposures were compared using as a basis the duration of exposure leading to inception of the five types of damage (Table 3). The 2-0.67 cycle produced surface degradation leading to and including fiber pop-out about 2 to 2.5 times faster than did the 4-4 cycle; formation of surface microcracking, isolated and network, took place 3.5 times faster. The acceleration factors of 2.5 for fiber pop-out and 3.5 for isolated surface microcracks are in good agreement with the predicted value of 3. In comparison with outdoor exposure, the 2-0.67 cycle accelerated initiation of fiber pop-out and isolated surface microcracks by factors of 5 and 30, respectively. Consequently, exposure of fire-retardant GRP in an accelerated weathering machine to the new cycle reduced the testing time from about 5 years of outdoor exposure to 1.5 months.

Non-Gel-Coated GRP

Exposure of non-gel-coated GRP to the 2-0.67 cycle led to surface deterioration characteristic of the five stages occurring in fire-retardant GRP. This was also true for exterior exposure and the 4-4 cycle [4], but in all three deterioration took place more slowly than with fire-retardant GRP. SE micrographs showed essentially the same fiber-related defects as occurred in Figs. 2 to 4 for fire-retardant GRP, but only after much longer exposure to each test. Surface microcracks (Fig. 7) in both accelerated cycles resemble each other in that individual cracks are similar in shape and length and are shorter than those induced outdoors, perhaps indicating more severe and rapid shrinkage during artificial weathering. The network in the 2-0.67 cycle is not so advanced at 176 days as that in the 4-4 cycle at 418 days.

Table 4 shows that the 2-0.67 cycle induced surface degradation of non-gel-coated GRP about 2 to 4 times faster than did the 4-4 cycle. Acceleration factors for fiber exposure (2.3) and isolated cracks (2.2) are close to the predicted values. With respect to outdoor exposure, the 2-0.67 cycle induced

TABLE 3—Rate of surface degradation of fire-retardant GRP produced by artificial and outdoor weathering.

Nature of Surface Degradation	Period of Exposure to First Occurrence of Degradation, days		Acceleration Factor	
	2-0.67 cycle	4-4 cycle	Outdoor	4-4/2-0.67 Outdoor/2-0.67
Fiber ridging	0.4	1	<60 ^a	2
Cracking of resin adjacent to fibers	2	4	<60 ^a	2
Fiber pop-out	34	90	170 ^a	2.5
Isolated surface microcracking	36	125	1070 ^b	3.5
Network of surface microcracking	39	135	1460 ^b	3.5

^aExposure initiated 10 March 1975 and panel specimens examined microscopically at 2-month intervals.^bExposure initiated 22 Feb. 1973 and panel specimens examined microscopically at 6-month intervals.

- (a) 2-0.67 cycle, 176 days exposure
- (b) 4-4 cycle, 418 days exposure
- (c) Outdoors, 1460 days exposure

FIG. 7.—Network of surface microcracks of non-gel-coated GRP.

TABLE 4—Rate of surface degradation of non-gel-coated GRP produced by artificial and outdoor weathering.

Nature of Surface Degradation	Period of Exposure to First Occurrence of Degradation, days			Acceleration Factor	
	2-0.67 cycle	4-4 cycle	Outdoor	4-4/2-0.67	Outdoor/2-0.67
Fiber ridging	0.4	1.5	<60 ^a	4	...
Cracking of resin adjacent to fibers	8	17	360 ^b	2	45
Fiber pop-out	75	175	940 ^b	2.3	13
Isolated surface microcracking	145	325	1070 ^b	2.2	7.5
Network of surface microcracking	175	400	1220 ^b	2.3	7.0

^aExposure initiated 10 March 1975 and panel specimens examined microscopically at 2-month intervals.^bExposure initiated 22 Feb. 1973 and panel specimens examined microscopically at 6-month intervals.

these failures 13 and 7.5 times faster, respectively, thus reducing the testing period for non-gel-coated GRP from 4 years to 7 months.

Conventional GRP

Exposure of conventional GRP to both accelerated cycles resulted in surface degradation similar to that of both the other GRP's. Five years of outdoor exposure, however, caused only the first three types of surface damage.

Table 5 shows that the 2-0.67 cycle induced fiber pop-out and surface microcracking 2.5 and 2.0 times faster, respectively, than did the 4-4 cycle. These factors are somewhat lower than the predicted value of 3, but the new cycle is still significantly faster for assessing durability of GRP. With respect to exterior weathering, the acceleration factor leading to fiber pop-out was 5 for the 2-0.67 cycle.

Conclusions

1. An exposure cycle of 2 h xenon arc radiation—0.67 h cold water spray in an artificial weathering machine has been designed to accelerate by a factor of three a previously used cycle of 4 h radiation—4 h cold water spray.

2. The 2-0.67 cycle produced, on both fire-retardant and non-gel-coated GRP's, surface deterioration similar to the five characteristic stages induced by exposure to the 4-4 cycle and to outdoor weathering at Ottawa.

3. Both accelerated cycles produced microcracking and a network of cracks that, so far, have not occurred outdoors on conventional GRP.

4. The acceleration factor of the 2-0.67 cycle for the initiation of fiber pop-out ranges, for all three GRP's, from 5 to 13 with respect to outdoor weathering and 2.3 to 2.5 in relation to the 4-4 cycle.

5. With respect to outdoor weathering for initiation of surface microcracks, the acceleration factor of the 2-0.67 cycle is 30 for fiber-retardant GRP and 7.5 for non-gel-coated GRP, showing much faster degradation of the former. No acceleration factor of this type of degradation can be calculated for conventional GRP because it has not yet failed outdoors.

6. The acceleration in deterioration of GRP resulting from the new cycles does not necessarily apply to other types of organic materials. The cycle was designed specifically to produce the types of failure that occur with GRP.

Acknowledgment

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TABLE 5—Rate of surface degradation of conventional GRP produced by artificial and outdoor weathering.

Nature of Surface Degradation	Period of Exposure to First Occurrence of Degradation, days			Acceleration Factor	
	2-0.67 cycle	4-4 cycle	Outdoor	4-4/2-0.67	Outdoor/2-0.67
Fiber ridging	0.4	1	<60 ^a	2	...
Cracking of resin adjacent to fibers	10	6	340 ^b	0.5	3
Fiber pop-out	115	285	580 ^b	2.5	5
Isolated surface microcracking	200	410	c	2.0	...
Network of surface microcracking	210	430	c	2.0	...

^aExposure initiated 10 March 1975 and panel specimens examined microscopically at 2-month intervals.

^bExposure initiated 22 Feb. 1973 and panel specimens examined microscopically at 6-month intervals.

^cSurface microcracking has not occurred to date after 1825 days of exposure.

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