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Natural Weathering of Mineral Stabilized Asphalt Coatings On Organic Felt

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Contents

Natural Weathering of Mineral Stabilized Asphalt Coatings on Organic Felt

Sidney H. Greenfeld

Sixteen years of outdoor weathering of laboratory-prepared smooth-surface and mineral-surfaced, felt-base roofing specimens has provided information on the effects of mineral additives on the durability of coating-grade roofing asphalts. Six finely divided mineral additives (blue black slate, clay, dolomite, fly ash, mica, and silica) were evaluated at concentrations up to 60 percent in California, Mid Continent and Venezuela asphalts. The mineral-surfaced specimens are all performing satisfactorily, and show only minor degrees of degradation. Of the smooth-surfaced specimens, the Mid-Continent asphalt performed the best and the California asphalt the poorest. The mica and blue black slate increased the durabilities of all three asphalts at all concentrations and two coating thicknesses. Fly ash, clay, dolomite, and silica were beneficial in some combinations, but had little effect in others. In general, these early results from outdoor exposure tend to corroborate the results obtained on these coatings exposed in weatherometers.

Key words: Additive; asphalt; durability; felt; stabilizer; weathering.

1. Introduction

The most certain way to determine how a product will weather in a given geographical area is to expose the product and observe it over the years. For materials or systems of low life expectancy, this procedure has been proven extremely satisfactory. For building materials, where the life expectancy is of the order of decades, the observed frequently outlives the observers. Those who design the tests, know their background and appreciate the significance of results, pass out of the picture and are replaced by others who find current work of greater interest. In many instances, the current production is no longer strictly comparable to that being observed.

Therefore, it has long been the practice to design rapid methods for evaluating materials and simultaneously exposing the same materials to the weather. The results of the laboratory evaluation become available in a relatively short period of time; those of the simulated-service tests (outdoor exposures) get lost in the shuffle and the validity of the rapid tests is never proven.

This study was designed in 1947 by the Research Committee of the Asphalt Roofing Manufacturers Association [1] to include a simulated-service study and several degrees of accelerated evaluation of the system: coating asphalt and mineral additives. The laboratory phase of the work was completed in 1954 and a series of reports and publications issued [1, 2, 3] [2]. The verification of the conclusions drawn in these articles will be found in the results obtained for the outdoor tests. This paper is in the nature of a progress report of the first 15–17 years of these outdoor tests.

1.1. Scope of Study

The mineral additive—asphalt systems included in this study consisted of three asphalts and six mineral additives at three concentrations on one grade of shingle felt. The coatings were applied in two film thicknesses, and were exposed unsurfaced and surfaced with roofing granules.

[1] Formerly the Asphalt Roofing Industry Bureau.
[2] Figures in brackets indicate the literature references at the end of this paper.

2. Experimental Details

2.1. Types of Specimens

Three types of specimens were included in this study. The basic specimen was a smooth-surface coated felt. These were exposed with coating thicknesses of 15 and 25 mils,[3] were back-coated with the same coating blend, and were dusted with mica retained on a No. 100 sieve.

[3] 1 Mil = 0.025 mm (approx).

The first type of specimen was made on 9- × 12-in[4] felts and trimmed to 7 × 11 in prior to exposure to remove any edge effects.

The second type of specimen also had a felt base. In addition to being front and back coated, it was surfaced with granules. These, too, were trimmed to 7 × 11 in.

[4] 1 in = 2.54 cm.

The third type of specimen was used for accelerated weathering tests. It consisted of the coating on $2\frac{3}{4}$- × 6-in aluminum panels. The results of these tests were reported earlier [3].

2.2. Equipment for Preparation of Specimens

a. Smooth-Surface, Felt-Base Specimens

Crimped (solder free) gallon cans.
Asphalt melting bath.
Six-ounce ladle.
Laboratory hydraulic press with 12- × 14-in cadmiun-plated platens.
Tempered hardboard, 12 × 14 in.
Kraft paper, 12 × 14 in.
Cellophane, 12 × 14 in.
Dextrin-coated paper, 12 × 14 in.

b. Granule-Surfaced Specimens

All of the granule-surfaced specimens were made on the apparatus shown in figure 1. Its principal parts are:

A. Hot plate for keeping the base warm during the pouring of the coating.
B. Variable-voltage-controlled, temperature-regulated doctor bar for spreading the coating and regulating its thickness.
C. Granule hopper and fluted granule spreader.
D. S-rolls for initial embedment of the granules and removal of excess.
E. Embedding rolls.
F. Belt drive for pulling canvas belt through the machine.

c. Back-Coater

The felt-base specimens were all back-coated with the apparatus shown schematically in figure 2.

d. Aluminum-Base Panels

The equipment used in preparing the aluminum base specimens is described in ASTM D1669, Standard Method for Preparation of Test Panels for Accelerated and Outdoor Weathering of Bituminous Coatings [1] and in reference [3].

2.3. Materials

a. Mineral Matter

The six minerals used in this investigation were blue black slate, Florida clay, Niagara dolomite, low-carbon fly ash, Tennessee mica, and Lake Erie silica. The properties of these materials are listed in table 1.

b. Asphalts

The descriptions and properties of the three asphalts used in this investigation were reported previously [3] and are tabulated in table 2.

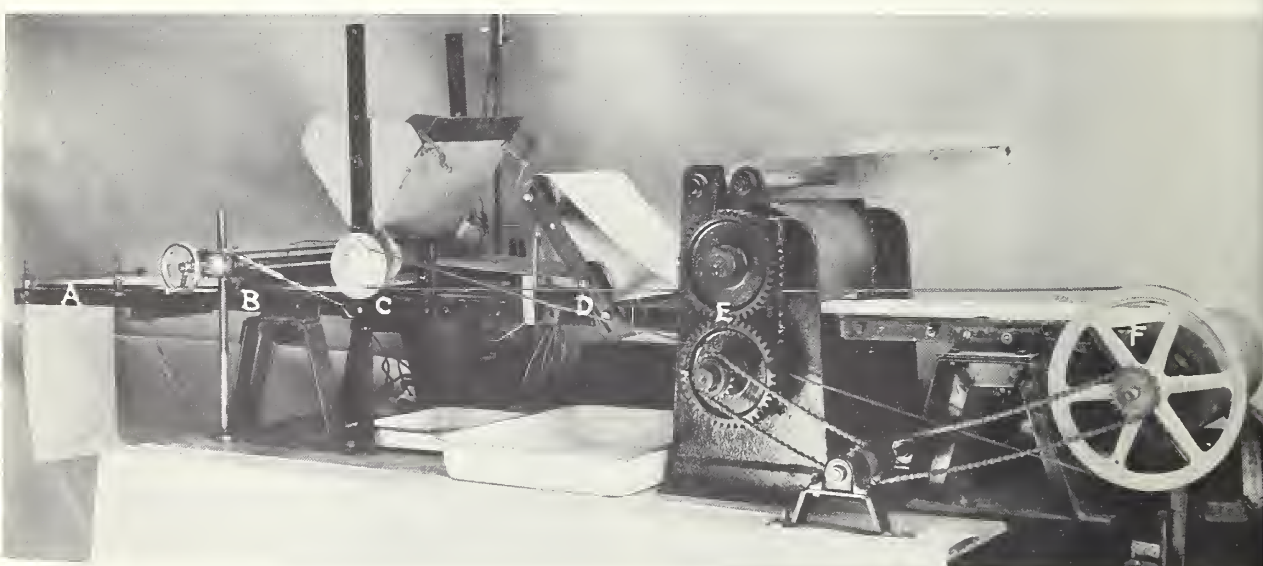

Figure 1. *Apparatus for preparing granule-surfaced specimens.*

A. Heated bed for keeping felts warm while asphalt coating is poured on them.
B. Doctor bar for leveling and defining coating thickness.
C. Granule hopper and distributor.
D. Granule placement S-rolls.
E. Granule pressing, or embedding, rolls.

TABLE 1. *Characteristics of mineral additives*

Stabilizer:	Blue Black Slate	Florida Clay [1]	Niagara Dolomite	Low Carbon Fly Ash	Tennessee Mica	Lake Erie Silica
Source:	# 50-Quarry Delta, Pa.	Mine Edgar, Fla.	Quarry Joliet, Ill.	Philadelphia Electric Co.	# 160-Pit Mine Johnson City, Tenn.	Dredged Lake Erie
Specific gravity [2] ... m²/g	2.94	2.64	2.87	2.62	3.01	2.68
Surface area [3]	1.0	27.2	2.0	2.0	2.7	2.5
Oil absorption [4] ... lb/100 lb	29.5	63.9	19.4	30.0	97.2	19.5
Water absorption [4] ... lb/100 lb	32.7	36.4	18.5	33.8	61.5	20.2
Loss on ignition at 1000 °F ... %	2.1	11.8	1.8	4.9	0.9	0.7
Loss on ignition at 1800 °F ... %	5.4	13.3	43.7	7.3	4.4	2.5
Moisture ... %	0.2	2.7	0.1	0.4	0.2	0.2
Solubility ... %	0.00	0.04	0.00	5.90	0.46	0.00
Free alkali [5] ... %	0.00	0.0	[6] 0.0	[6] 0.0	0.0	0.0
Chemical analysis [7] ... %						
SiO_2	56	47	6	40	50	98+
R_2O_3 (Al_2O_3 + Fe_2O_3)	32	38	1	48	35	
CaO + MgO			49	2.5	1	
K_2O + Na_2O	4				10	
SO_4					0.5	
Carbon	2			7.6		

Particle size, % finer than: Mils	μM	U.S. Sieve No.	Blue Black Slate	Florida Clay	Niagara Dolomite	Low Carbon Fly Ash	Tennessee Mica	Lake Erie Silica
9.84	250	60	99.8	100.0	99.9	99.6	100.0	99.9
6.97	177	80	99.3	99.9	99.9	98.9	96.0	98.2
5.86	149	100	97.9	99.8	99.6	97.5	91.0	80.8
4.92	125	120	96.2	99.8	99.3	96.7	85.6	53.7
3.47	88	170	91.3	99.6	96.6	94.2	65.9	15.3
2.91	74	200	86.9	99.3	93.4	92.3	56.8	8.1
2.44	62	230	83.6	99.1	89.9	90.6	50.3	5.4
1.73	44	325	76.7	98.9	81.0	84.8	37.8	3.4
1.57	40	Sed.[8]	68	97	73	73	65	9
0.79	20	Sed.[8]	54	12	39	60	22	6
0.39	10	Sed.[8]	8	2	26	43	4	4
0.16	4	Sed.[8]	2	1	13	15	2	1
0.08	2	Sed.[8]	1	—	7	2	—	—

Mixture with asphalt (40% minerals in asphalt III)	Blue Black Slate	Florida Clay	Niagara Dolomite	Low Carbon Fly Ash	Tennessee Mica	Lake Erie Silica
Ease of mixing	Good	Poor	Fair	Fair to Good	Good	Good
Softening point increase ... °F (°C)	15 (8.3)	28 (15.6)	13 (7.2)	20 (11.1)	40 (22.2)	5 (2.8)

[1] Plasticity index = 34. Plastic limit = 34. ASTM method D424–39.
[2] Isopropyl alcohol displacement.
[3] Low temperature nitrogen adsorption—B.E.T. method.
[4] ASTM D281–31, using a mineral oil and water instead of the specified oil.
[5] Lerch, W., and Bogue, R. H., Ind. Eng. Chem. 2, 296–300, 1930.
[6] Turns phenolphthalein pink in aqueous solution.
[7] Supplier's analyses.
[8] Sedimentation in isopropyl alcohol.

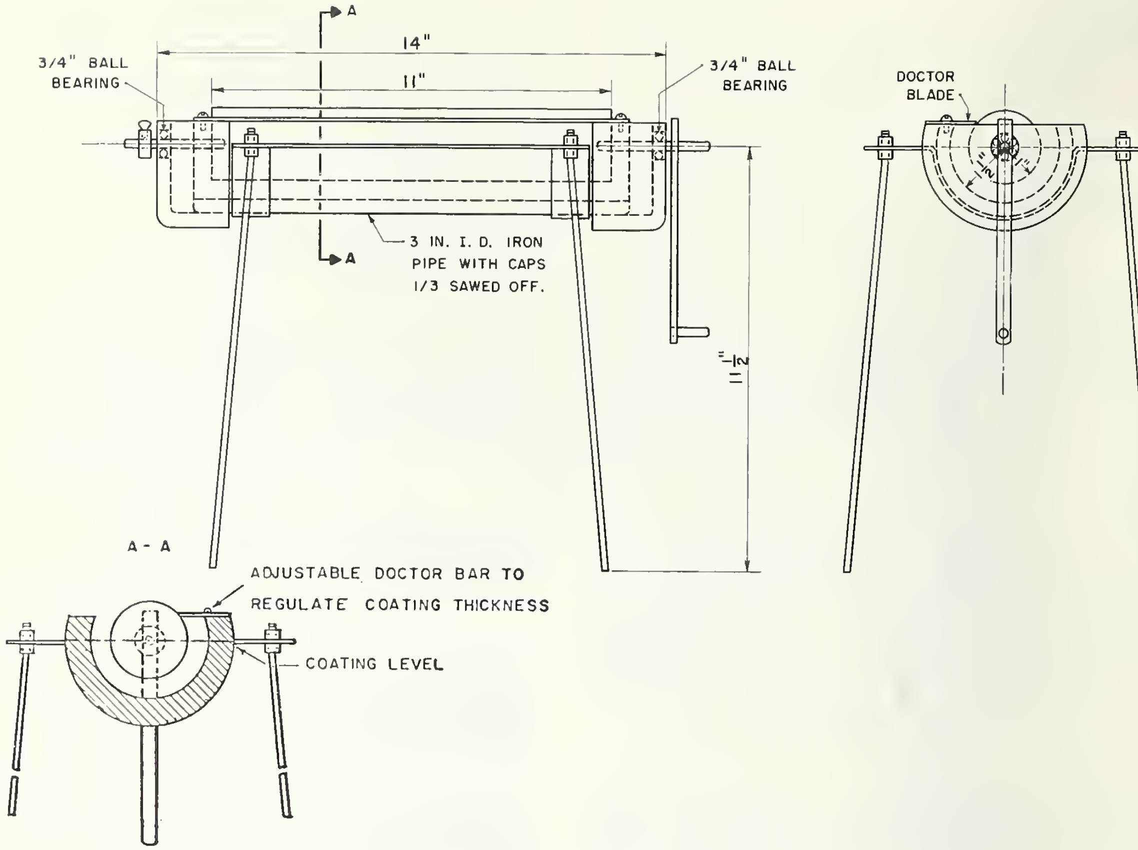

FIGURE 2. *Apparatus for coating the backs of the specimens.*

Each specimen was placed face up on the top of the 11 -incylinder and the crank turned to apply a thin coating on its back.

TABLE 2. *Characteristics of unstabilized asphalt coatings used as controls*

Asphalt	California	Mid-continent	Venezuela
Test			
Softening point [1] °F (°C)	223 (106.0)	224 (106.5)	227 (107.8)
Penetration at 32 °F (0 °C) [2] 0.1 mm	10	11	11
Penetration at 77 °F (25 °C) 0.1 mm	17	17	14
Penetration at 115 °F (46.1 °C) 0.1 mm	30	26	21
Penetration index [2]	4.7	4.7	4.5
Susceptibility [2]	1.16	0.87	0.73
Loss on heating,[3] %	0.22	0.03	0.10
Penetration at 77 °F (25 °C) after heating, 0.1 mm	17	17	14
Specific gravity at 77 °F (25 °C)	1.015	0.999	1.018
Viscosity, cP at 400 °F [4] (204 °C)	280	420	375
Viscosity, cP at 450 °F (232 °C)	100	140	130
Viscosity, cP at 500 °F (260 °C)	25	53	28
Water absorption, at 28 days [5] g/m ²	7.2	4.6	3.7
Shatter [5] in (cm) (ave)	6.3 (16.0)	8.3 (21.1)	2.7 (6.9)

[1] ASTM method D36–26.

[2] ASTM method D5–52. P. I. from Nomograph, and $S = \dfrac{\text{Pen. } 115\ °\text{F } (46.1\ °\text{C}) - \text{Pen. } 32\ °\text{F } (0\ °\text{C})}{\text{Pen. } 77\ °\text{F } (25\ °\text{C})}$. (6)

[3] ASTM method D6–39T.

[4] Brookfield viscometer; 1 cP = $0.001 \frac{\text{N.s}}{\text{m}^2}$.

[5] Specimens, 3-in diam × 3/16-in thick, submerged 1/4-in in distilled water at 77 °F (25 °C).

[6] A 1/2-lb weight was dropped on a 3-in diam specimen, 3/16-in thick, in a mixed ice and water bath.

c. Saturated Felts

The dry felt was prepared for this project in rolls 12 ± 1/2 in wide by 300 ft long and shipped by one of the sponsoring companies to another of the sponsors for saturation by personnel on the project. The dry and saturated felts had the characteristics listed in table 3.

TABLE 3. *Characteristics of felt*

Dry felt			
Furnish, %:	Rags	20	
	Paper	35	
	Asplund	40	
	Scrap felt	5	
Gauge, lb/480 ft^2 [2]			53 ± 2
Kerosene capacity [1]		g ker/g felt:	1.90 ± 0.01
Caliper, mils:			57 ± 3
Tensile, lb/in			31
Ash, %:			6.8

Saturated felt	Saturation	
	Percent of felt weight	Percent of kerosene capacity
California asphalt	177	93
Mid-Continent asphalt	183	96
Venezuela asphalt	187	98

[1] Burette method ARIB No. 2.125, April 1954.
[2] 1 lb = 0.45 kg (approx).

d. Aluminum Panels

The panels, made from S–2 aluminum alloy, were 6 in long, 2¾ in wide, and 0.065 in thick.

e. Granules

The granules were of the ceramic type, with the characteristics listed in table 4. These characteristics were supplied by the granule manufacturer and no attempt was made to verify them.

2.4. Specimen Preparation

a. Smooth-Surface, Felt-Base Specimens

The asphalts were melted in a double-boiler, consisting of a one-gallon can, wrapped in aluminum foil to prevent contamination, sitting in a two-gallon bucket half full of asphalt. The temperatures of both the asphalt under investigation and the asphalt in the bath were watched constantly to prevent any overheating. When the temperature of the test asphalt was in the range of 400 to 450 °F (204 to 232 °C), any required mineral matter, preheated to 300 °F (149 °C), was stirred into it with a large chromium-plated ladle. The mixture was continually stirred at 450 °F (232 °C) until it no longer frothed. All compositions were made and are reported on a weight-percent basis.

The saturated felts, cut into 9- × 12-in sheets, were heated in an air oven at 300 °F (149 °C) to warm them and to eliminate moisture. They were ready for use when the saturant, which came to the surface as the felts were heated, was completely reabsorbed.

When the felts and coating were in working condition, a felt was removed from the oven and placed on a sheet of cellophane backed by a double thickness of 12- × 14-in Kraft paper and a 12- × 14-in piece of tempered hardboard. Approximately one and a half times the required quantity of coating was distributed over the felt and spacers of the required thickness (felt thickness plus 3 mils) were placed along two of the sides of the felt. A sheet of dextrin-coated paper, two sheets of Kraft paper and a piece of tempered hardboard were successively placed over the coating, and the assembly was put into the hydraulic press. The press was closed and the pressure raised to about 120 psig. After about 30 s, the panel was removed from between the sheets of Kraft paper and placed in a water bath at room temperature, where the cellophane and dextrin paper soon floated free. The panel was thoroughly washed and dried and its thickness measured.

When the required number of acceptable panels had been prepared, the hot coating was ladled from the melting bath to the back-coater, which had previously been heated to about 450 °F (232 °C). While one man rotated the roller slowly, another passed the back of the panel over the roller, picking up a thin coating. The back coating was dusted with mica while still warm. Upon cooling, each specimen was cut to 7 × 11 in and exposed the next day on wooden roof decks, which had been covered with smooth-surface roll roofing (facing due south and at an angle of 45°). An area of 4 × 7 in was exposed, leaving each specimen a 3-in head lap.

TABLE 4. *Characteristics of granules*

Base Rock: − 10 + 35 mesh Wausau, Wisconsin graystone (Argillite).

Chemical Analysis

Component	Percentage
SiO_2	70.0
Al_2O_3	17.4
Fe_2O_3	5.7
MgO	1.5
CaO	0.3
Na_2O	4.8
CO_2	0.6
H_2O (at 105 °C)	0.04

Pigments:	Chromium oxide and phthalocyanine green, bonded with sodium silicate.[1]
Granules:	
Acidity:	Extract from 25 g is equivalent to 0.1 ml of 0.1N acid.
Color fixation:	30 percent pigment loss.
Wettability:	80 percent.
Oil absorption:	6 lb/ton [2] (3 g/kg).

Particle Size (Tyler)

Sieve No.	Percent
Retained on 10	0.7
Retained on 14	33.9
Retained on 20	36.0
Retained on 28	21.6
Retained on 35	7.2
Passing 35	0.5

Granules were oiled with 5 lb per ton (2.5 g/kg) of lightweight paraffin oil, complying with the following specification:

Sp. Gr.	26–30 ° API
Viscosity at 100 °F (37.8 °C)	96–105 S.U.S.
Flash point (COC)	350 °F (177 °C) min.
Fire point (COC)	395 °F (202 °C) min.
Pour point	25 °F (− 3.9 °C) max.

[1] U.S. Patent No. 2,417,058.
[2] Basis of test not reported.

b. **Granule-Surfaced, Felt-Base Specimens**

The asphalt and felts were prepared as in 2.4a. The hot plate (A) on the laboratory roofing machine (fig. 1) was heated to about 250 °F (121 °C) and the doctor bar (B) to 450 °F (232 °C). The canvas belt was threaded through the machine and a sheet of 12-in wide Kraft paper three feet long was fastened to its end, about 4 in in front of the doctor bar. In order to be assured that the doctor bar was at the proper distance from the machine bed, a blank run was made in which no granules were used. When cool, the thickness of the coated panel was measured.

In a normal run the felt panel was fastened with masking tape to the Kraft paper on the hot plate. About one and a half times the required coating was distributed over the panel. The motor was started and the panel was pulled under the doctor bar (B) and granule hopper (C); then the motor was stopped for 2 min to permit the panel to cool and the granules to become firmly set. Then the panel was drawn through the S rolls (D) and embedding rolls (E). The panel was removed from the belt and passed backwards through the embedding rolls again. Because of the heavy gear on one side of the embedding rolls and because the rolls were not crowned, this procedure was necessary to make the embedment of the granules more uniform. The panels were backcoated as in 2.4a.

c. **Smooth-Surface, Aluminum-Base Specimens**

The smooth-surface, aluminum-base specimens were prepared as in ASTM D 1669, which is based on work reported in reference [3].

2.5. Specimen Exposure

a. **Felt-Base Specimens**

Within 24 hr after their preparation the felt-base specimens, both smooth and granule surfaced, were exposed outdoors on the roof of the Industrial Building at the former NBS Washington site, facing due south and at an angle of 45 deg. The 7- × 11-in specimens were mounted by nailing shingle fashion on protected decks with 4- × 7-in tabs exposed to the weather. The decks for the smooth-surface specimens were protected with 55-lb roll roofing, which also served as the under-layment for these specimens; the granule-surfaced specimens had a 90-lb mineral-surfaced roll roofing as underlayment and deck cover.

b. **Aluminum-Base Specimens**

The aluminum-base specimens were exposed in two weatherometers to cycle 51-9C of ASTM D 529, within 24 hr of the time they were prepared. A detailed account of the apparatus and procedure is given in reference [3].

2.6. Specimen Evaluation

a. **Felt-Base Specimens**

The 4- × 7-in exposed portions of the specimens were examined through a transparent grid with 98 squares [0.40 × 0.40 in] covering the central 3¼- × 6¼-in area. The areas around the edges [0.325 in wide] of the 4- × 7-in area were excluded from the examination to eliminate the edge effects. The numbers of squares with cracks and spalled coating were counted and recorded during each inspection. Inspections were made every three months during the first two years of exposure and semiannually thereafter. The semiannual inspections were made in March and September.

b. **Aluminum-Base Specimens**

The aluminum-base specimens were examined with a high voltage probe in accordance with ASTM D 1670. Spark photographs were taken of all specimens with three or more breaks in the coating. These photographs were examined through a transparent 60- rectangle grid covering the central 2- × 5-in portion of the specimen. Only failures by cracking were recorded for this type of specimens.

3. Progress of Deterioration

3.1. Smooth-Surface Specimens

When the smooth-surface specimens were first prepared, they had a matte finish corresponding to the texture of the dextrin-coated release paper used on them. After short periods of exposure, the surfaces became glossy. Continued exposure produced elevated ridges, followed by thinner connecting ridges. These ridges were the result of expansion of the surface as it became oxidized. As oxidation progressed, the surface became progressively more water soluble; material was dissolved by rain outdoors or water spray in the weatherometers, and the glossy, ridged surface assumed a matte appearance. With time, widely spaced cracks appeared. These cracks usually penetrated the felt-base specimens fairly rapidly through the coating to the felts. In the aluminum-base specimens, the cracks frequently remained on the surface for relatively long periods of time and failure was evidenced by pinholing in noncracked areas or at crack intersections.

In the felt-base specimens, widely spaced cracks became interlaced with narrower cracks until spalling of the small areas surrounded by cracks began. As coating spalled from the specimens, the substrates were exposed to the weather. In the aluminum-base specimens, exposure was usually discontinued prior to the spalling phase of weathering. In the felt-base specimens, the spalling permitted the saturated felt to be degraded rapidly by the weather. Photooxidation rapidly destroyed the saturant and permitted water to enter the felts and, through alternate wetting and drying, shrink and distort them. Finally, the weakened tabs were blown away.

3.2. Granule-Surfaced Specimens

The granule-surfaced specimens have shown only the early stages of deterioration during the 15 years they have been exposed to the weather. Changes observed early in the exposure period consisted of small losses of granules in most of the specimens and heavier losses of granules in the few specimens in which poor granule adhesion was measured prior to exposure. After about 12 years of exposure, cracks began to appear in the coatings. About a quarter of the specimens had cracks in 5 percent of their surface areas (five grid areas) after 12 and a half years. No spalling of the coatings has been observed in any of these specimens since their exposure in 1952. However, in the normal course of events, as weathering progresses, generally more granules will be lost, more cracking will occur and, finally, the coatings will spall off the saturated felts, just as in the smooth-surface, felt-base specimens. The granules protect the coating from solar radiation and greatly delay the later stages of deterioration.

3.3. Blistering

Early in the exposure of the smooth-surface, felt-base specimens some small blisters were observed. These were principally around the edges, but in some of the specimens made with Mid-Continent and Venezuela asphalt, blisters appeared well within the areas inspected. Only a very few specimens made with California asphalt had any blisters; none had more than three. While blistering contributes to poor appearance, it does not interfere with the essential function of roofing, which is to keep the structures weatherproof. Therefore, blistering is not normally considered as a criterion of failure. However, blisters can act as centers of stress concentration and lead to earlier cracking and spalling of the coating. Thus, the effect of blistering is manifested in the reported cracking and spalling. The only other comment that can be made concerning blistering is that the largest incidence of blistering was associated with the coatings made from the more durable asphalts.

The aluminum-base specimens and the granule-surfaced, felt-base specimens experienced essentially no blistering.

4. Failure Criteria

When a product exposed to the weather no longer performs the function for which it was designed, it has failed. However, it has become customary to replace roofing for reasons other than, and earlier than, complete failures. Quite frequently, shingles are replaced because they no longer have an acceptable appearance; on other occasions failure can be anticipated in the near, but indefinite, future. Consequently, certain standards of failure were arbitrarily selected by the sponsors of this program.

These failure criteria appear in table 5.

Table 5. *Failure criteria*

Specimen type	Criterion of failure
Smooth-surface, felt base	Spalls in 25 grid areas (25%).
Granule-surfaced, felt-base	50% Granule loss in 49 grid areas (50%) or spalls in 25 grid areas (25%).
Aluminum-base	Cracks in 30 grid areas (50%)

5. Outdoor Performance

5.1. Smooth-Surface, Felt-Base Specimens

The results of 200 months (16⅔ years) of exposure of the smooth-surface, felt-base specimens to the weather are summarized in tables 6 and 7. Information on crack development is presented along with the observations of spalling. The ratio of the time in which an asphalt coating with mineral matter in it reaches a given level of deterioration to the time in which the same asphalt alone reaches that same level of deterioration is also reported. This ratio is frequently more significant than the actual number of months of exposure, for this comparison of a coating with mineral additives to its base asphalt tends to compensate for variations in the weather, or climate. This technique was used in reference [3], where the results of the accelerated weathering on these coatings were discussed. (A few of the coatings 15 mils thick (table 6) and many more of those 25 mils thick (table 7) had not reached some of the levels of deterioration reported. This condition is indicated by dashes in both tables).

At every level of performance and in both thicknesses the Mid-Continent asphalt outperformed the Venezuela asphalt, which, in turn, outperformed the California asphalt. The mica-stabilized coatings out performed all the others; only one coating had deteriorated to any of the selected levels in tables 6 and 7 in 200 months. The 15-mil California coating with 35 percent mica exhibited its first spalled areas in 164 months.

Before discussing specific performances, however, a word of explanation is in order concerning the mechanism of cracking and why cracking, though ultimately contributing significantly to shingle

Table 6. *Smooth surface specimens* [1]

15 Mil (0.38 mm) Thickness

Coating description	25% Cracked		50% Cracked		Initial Spall		25% Spalled		50% Spalled	
	Months	Ratio	Months	Ratio	Months	Ratio	Months	Ratio	Months	Ratio
California Asphalt	15	1.0	15	1.0	35	1.0	49	1.0	55	1.0
+ 35% BBS	18	1.2	18	1.2	71	2.0	104	2.1	116	2.1
+ 50% BBS	38	2.5	40	2.7	101	2.9	139	2.8	145	2.6
+ 60% BBS	99	6.6	140	9.3	174	5.0	—	—	—	—
+ 35% Clay	19	1.3	19	1.3	42	1.2	60	1.2	66	1.2
+ 50% Clay	9	0.6	9	0.6	42	1.2	79	1.6	91	1.7
+ 35% Dolomite	18	1.2	18	1.2	37	1.1	55	1.1	61	1.1
+ 50% Dolomite	24	1.6	24	1.6	37	1.1	55	1.1	58	1.1
+ 60% Dolomite	24	1.6	24	1.6	40	1.1	61	1.2	69	1.3
+ 35% Fly Ash	9	0.6	9	0.6	49	1.4	65	1.3	77	1.4
+ 50% Fly Ash	9	0.6	9	0.6	52	1.5	85	1.7	93	1.7
+ 60% Fly Ash	28	1.9	28	1.9	100	2.9	133	2.7	139	2.5
+ 35% Mica	—	—	—	—	164	4.7	—	—	—	—
+ 35% Silica	23	1.5	23	1.5	48	1.4	63	1.3	79	1.4
+ 50% Silica	19	1.3	19	1.3	54	1.5	72	1.5	78	1.4
+ 60% Silica	48	3.2	48	3.2	72	2.1	90	1.8	102	1.9
Mid-Continent Asphalt	32	1.0	32	1.0	82	1.0	120	1.0	130	1.0
+ 35% BBS	94	2.9	109	3.4	118	1.4	—	—	—	—
+ 50% BBS	71	2.2	100	3.1	142	1.7	155	1.3	176	1.4
+ 60% BBS	[2] 201	[2] 6.3	—	—	—	—	—	—	—	—
+ 35% Clay	29	0.9	32	1.0	79	1.0	102	0.9	113	0.9
+ 50% Clay	26	0.8	29	0.9	72	0.9	98	0.8	107	0.8
+ 35% Dolomite	53	1.7	56	1.8	88	1.1	130	1.1	139	1.1
+ 50% Dolomite	56	1.8	59	1.8	144	1.8	152	1.3	159	1.2
+ 60% Dolomite	62	1.9	68	2.1	105	1.3	157	1.3	166	1.3
+ 35% Fly Ash	38	1.2	38	1.2	113	1.4	140	1.2	T	T
+ 50% Fly Ash	36	1.1	44	1.4	88	1.1	143	1.2	148	1.1
+ 60% Fly Ash	62	1.9	100	3.1	96	1.2	—	—	—	—
+ 35% Mica	—	—	—	—	—	—	—	—	—	—
+ 35% Silica	31	1.0	38	1.2	41	0.5	148	1.2	.54	1.2
+ 50% Silica	31	1.0	34	1.1	93	1.1	158	1.3	173	1.3
+ 60% Silica	34	1.1	37	1.2	80	1.0	161	1.3	181	1.4
Venezuela Asphalt	27	1.0	27	1.0	55	1.0	91	1.0	101	1.0
+ 35% BBS	36	1.3	36	1.3	101	1.8	156	1.7	166	1.6
+ 50% BBS	54	2.0	57	2.1	95	1.7	159	1.7	190	1.9
+ 60% BBS	60	2.2	73	2.7	133	2.4	—	—	—	—
+ 35% Clay	17	0.6	17	0.6	54	1.0	84	0.9	94	0.9
+ 50% Clay	17	0.6	17	0.6	60	1.1	96	1.1	113	1.1
+ 35% Dolomite	21	0.8	21	0.8	66	1.2	101	1.1	113	1.1
+ 50% Dolomite	23	0.9	23	0.9	57	1.0	100	1.1	115	1.1
+ 60% Dolomite	29	1.1	30	1.1	63	1.1	114	1.3	126	1.2
+ 35% Fly Ash	20	0.7	20	0.7	96	1.7	152	1.7	155	1.5
+ 50% Fly Ash	27	1.0	27	1.0	66	1.2	152	1.7	155	1.5
+ 60% Fly Ash [3]										
+ 35% Mica	—	—	—	—	—	—	—	—	—	—
+ 35% Silica	16	0.6	16	0.6	51	0.9	79	0.9	91	0.9
+ 50% Silica	16	0.6	16	0.6	60	1.1	91	1.0	97	1.0
+ 60% Silica	19	0.7	19	0.7	53	1.0	99	1.1	111	1.1

[1] Average of two specimens.
[2] One specimen only.
[3] Inspection sheets lost.
— Specimens have not progressed to this failure level after 200 months of exposure.
T Tab torn off.

failure, cannot be used as an early warning of impending failure. While early cracking of the coating on shingles may be startling to an uninformed observer, the data in tables 6 and 7 for smooth-surface specimens show the lack of correlation between early cracking and failure. Thus, cracking has not been used as one of the criteria of failure.

Cracking of coatings on shingles results from the large differences in thermal coefficient of linear expansion between the saturated felt and the shingle coatings. Data on these materials are difficult to obtain, but a few order-of-magnitude figures have been published. These are listed in table 8.

It is evident from the above data asphalt moves roughly 25 times as much as the saturated felt does in the cross direction and 50 times as much as the saturated felt in the machine direction as the temperature changes. At the higher end of the range of temperatures encountered by roofing during exposure, the asphalt can accommodate the differential movements by flow, but at the lower end of the temperature range flow is not rapid enough and partial accommodation results by the curling of the shingle tabs. When this movement can no longer cope with the stresses, cracking occurs. These cracks usually run at right angles to the greatest differential movement. In the exposure of the smooth-surface specimens, all early cracking first occurred in the cross direction. These occurred very early in the exposure period, in some instances during the first winter, but the specimens continued to perform well for many years. There was no relationship between any of the levels of cracking and spalling. Therefore, nothing more will be said about cracking, for spalling has been set as the criterion for failure.

Returning to the performances of specific systems, it can be seen that many of the specimens containing blue black slate have not deteriorated to the 25 percent spalled level after 17 years of exposure. For these systems, just as with the asphalts without

Table 7. *Smooth surface specimens* [1]

25 Mil (0.64 mm) Thickness

Outdoor Exposures

Coating description	25% Cracked		50% Cracked		Initial Spall		25% Spalled		50% Spalled	
	Months	Ratio	Months	Ratio	Months	Ratio	Months	Ratio	Months	Ratio
California Asphalt	10	1.0	10	1.0	46	1.0	80	1.0	95	1.0
+ 35% BBS	21	2.1	25	2.5	95	2.0	136	1.7	T	—
+ 50% BBS	38	3.8	58	5.8	116	2.5	T	—	T	—
+ 60% BBS	121	12.1			149	3.2	—	—	—	—
+ 35% Clay	11	1.1	11	1.1	63	1.4	100	1.2	115	1.2
+ 50% Clay	26	2.6	29	2.9	72	1.6	127	1.6	142	1.5
+ 35% Dolomite	18	1.8	18	1.8	58	1.3	79	1.0	101	1.1
+ 50% Dolomite	9	0.9	9	0.9	61	1.3	88	1.1	103	1.1
+ 60% Dolomite	23	2.3	26	2.6	61	1.3	88	1.1	101	1.1
+ 35% Fly Ash	8	0.8	8	0.8	61	1.3	91	1.1	109	1.1
+ 50% Fly Ash	14	1.4	14	1.4	58	1.3	94	1.2	118	1.2
+ 60% Fly Ash	27	2.7	31	3.1	97	2.1	130	1.6	142	1.5
+ 35% Mica	—	—	—	—	—	—	—	—	—	—
+ 35% Silica	9	0.9	9	0.9	63	1.4	87	1.1	100	1.1
+ 50% Silica	9	0.9	9	0.9	75	1.6	103	1.3	126	1.3
+ 60% Silica	8	0.8	8	0.8	145	3.2	145	1.8	145	1.5
Mid-Continent Asphalt	28	1.0	36	1.0	156	1.0	174	1.0	200	1.0
+ 35% BBS	121	4.3	135	3.7	157	1.0	—	—	—	—
+ 50% BBS	81	2.9	108	3.0	201	1.3	—	—	—	—
+ 60% BBS	—	—	—	—	—	—	—	—	—	—
+ 35% Clay	36	1.3	39		103	0.7	143	0.8	151	0.8
+ 50% Clay	34	1.2	39	1.1	109	0.7	166	1.0	176	0.9
+ 35% Dolomite	44	1.6	50	1.4	117	0.8	177	1.0	—	—
+ 50% Dolomite	72	2.5	83	2.3	107	0.7	—	—	—	—
+ 60% Dolomite	62	2.2	80	2.2	99	0.6	178	1.0	—	—
+ 35% Fly Ash	38	1.4	38	1.1	165	1.1	—	—	—	—
+ 50% Fly Ash	50	1.8	65	1.8	171	1.1	—	—	—	—
+ 60% Fly Ash	62	2.2	100	2.8	96	0.6	—	—	—	—
+ 35% Mica	—	—	—	—	—	—	—	—	—	—
+ 35% Silica	33	1.2	38	1.1	123	0.8	195	1.1	—	—
+ 50% Silica	32	1.1	38	1.1	105	0.7	—	—	—	—
+ 60% Silica	35	1.2	41	1.1	95	0.6	—	—	—	—
Venezuela Asphalt	25	1.0	33	1.0	101	1.0	159	1.0	167	1.0
+ 35% BBS	48	1.9	58	1.8	107	1.1	171	1.1	—	—
+ 50% BBS	79	3.2	94	2.8	151	1.5	—	—	—	—
+ 60% BBS	88	3.5	139	4.2	97	1.0	—	—	—	—
+ 35% Clay	21	0.8	21	0.6	[1] 86	0.9	[2] 144	0.9	[2] 149	0.9
+ 50% Clay	12	0.5	12	0.4	68	0.7	[2] 144	0.9	[2] 149	0.9
+ 35% Dolomite	21	0.8	21	0.6	57	0.6	159	1.0	166	1.0
+ 50% Dolomite	29	1.2	31	0.9	72	0.7	162	1.0	166	1.0
+ 60% Dolomite	37	1.5	39	1.2	65	0.6	149	0.9	159	1.0
+ 35% Fly Ash	28	1.1	28	0.8	108	1.1	171	1.1	187	1.1
+ 50% Fly Ash	23	0.9	29	0.9	107	1.1	—	—	—	—
+ 60% Fly Ash	32	1.3	32	1.0	107	1.1	202	1.3	—	—
+ 35% Mica	—	—	—	—	—	—	—	—	—	—
+ 35% Silica	16	0.6	16	0.5	84	0.8	152	1.0	159	1.0
+ 50% Silica	16	0.6	18	0.5	123	1.2	159	1.0	180	1.1
+ 60% Silica	16	0.6	16	0.5	78	0.8	—	—	—	—

[1] Average of two specimens.
[2] One specimen only.
— Specimens have not progressed to this failure level after 200 months of exposure.
T Tab torn off.

additives, the thicker films are performing better and the asphalts are tending to fall in the same order of durability. It is also apparent, with one exception, that coatings with higher concentrations of blue black slate are deteriorating more slowly than those with lower concentrations.

For materials with other than those with plate-like particle shapes, the response to mineral additives cannot be discussed in general terms [3]. The other additives used tended to have blocky to spherical shapes; their performances were varied in the three asphalts.

Fly ash was the next best performer in that it increased the durability of some coatings as much as 170 percent and in no instance were coatings containing fly ash less durable than the asphalt with which they were made. The higher concentrations again produced the best performers and the thicker films lasted longer than the corresponding thinner ones. Most of the Mid-Continent coatings with fly ash have not failed. The Venezuela coatings 25 mils thick with 50 and 60 percent fly ash are just beginning to fail, and the California-fly ash coatings have all failed in 1.1 to 1.5 times the durability of the

Table 8.

Material	Coefficient of linear expansion	Temperature Range	Source
	°C⁻¹	°C	
Asphalt saturant	6.1×10^{-4}	15–60	(7)
Asphalt coating	5.9×10^{-4}	15–60	(7)
Petroleum asphalt and fluxes	$6.3\text{–}6.8 \times 10^{-4}$	0–230	[1] (7)
Asphalt— various sources	$5.8\text{–}6.2 \times 10^{-4}$		(8)
Organic felt— machine direction	1.1×10^{-5}	(−34)–(−1)	[1] (9)
Organic felt— cross direction	2.4×10^{-5}	(−34)–(−1)	[1] (9)
Aluminum alloys	$2.4\text{–}2.6 \times 10^{-5}$		[1] (10)

[1] Converted to metric system.

California asphalt without additives. Thus, the pattern established by the unstabilized asphalts has been carried through to the coatings containing fly ash.

The effects of clay, dolomite, and silica on the durability of the smooth-surface specimens were more variable than those of blue black slate, fly ash, and mica. The silica was most beneficial in the California asphalt, for it increased its durability (25 percent coating spalled) as much as 80 percent. In all instances it increased the durability by at least 10 percent. Similarly, in the Mid-Continent asphalt it also increased the durability of the asphalt by at least 10 percent; the 25-mil coatings with 50 and 60 percent silica have not failed after 200 months, or a 15 percent greater time than the straight asphalt specimens. The specimens made from the Venezuela asphalt-silica system again were in between those with Mid-Continent and California asphalts, but the inprovement in durability by the silica was not as consistent as in the other two asphalt systems. Three of the coatings performed only as well as the asphalt alone, one not quite as well, one a little better, and the one with 60 percent silica 25 mils thick is still performing well after 200 months.

In only a few combinations containing clay or dolomite were improvements in durability of more than 20 percent observed. Fifty percent clay increased the durability of the California asphalt in both thicknesses by 60 percent. Fifty percent dolomite improved the thin films of both Mid-Continent and Venezuela asphalt by 30 percent. However, the other combinations performed within 20 percent of the durability of the straight asphalts.

Thus, in summary, the Mid-Continent asphalt was more durable than the Venezuela asphalt, which was more durable than the California asphalt. The mineral additives either improved the durability or had little effect. Mica and blue black slate always improved the durability of the three asphalts. Fly ash, silica, dolomite, and clay improved the durability to a lesser extent and only in specific combinations of composition and coatings thickness.

Figure 3 is a photograph of some of the specimens made with Mid-Continent asphalt after 16½ years of exposure. The test specimens are the five in each column under the identification tags, as identified in figure 3.

The variation of performance with composition and coating thickness is evident in this photograph.

5.2. Granule-Surfaced, Felt-Base Specimens

Prior to exposure, samples of all of the granule-surfaced specimens were subjected to granule adhesion tests. The procedure used was ARIB Index No. 1.119 [10], using the electrically operated equipment. As recommended in the procedure, the average weight loss for 10 2- × 10-in specimens when abraded dry must be in the range of 0.2 to 0.7 g. The only composition that produced specimens that did not fall within this range was 60 percent blue black slate in Venezuela asphalt. A second set of specimens was made, but it also did not pass the test. Both sets of specimens were exposed and both experienced large losses of granules early during their exposure. The California specimens containing clay passed the test marginally. They, also, lost some granules early during exposure. However, none of these specimens has failed by the established criteria.

Figures 4 and 5 are photographs of California and Venezuela specimens after 15 years of exposure. The unstabilized asphalt coatings show noticeable losses of granules and cracking of the coatings. Cracking of the coatings is also evident in the California coatings containing clay. However, all of the granule-surfaced specimens are still performing well after 15 years and should give many more years of protection from the weather.

6. Correlation With Exposures in Accelerated Weathering Machine

Because most asphalt-prepared roofing is exposed as granule-surfaced material, the ultimate correlation must be between the granule-surfaced specimens exposed outdoors and the specimens exposed in the accelerated weathering test. Because none of the granule-surfaced specimens has failed by any of the established criteria for failure, a discussion of correlation would be meaningless. However, many of the smooth-surface, felt-base specimens have failed. While no quantitative correlation can be made until more specimens fail, certain parallels can be seen in figure 6 between the performances of these specimens and their corresponding aluminum-base specimens exposed in the weatherometer. The dots represent the average durability of four specimens on aluminum panels exposed in the weatherometers and the bars represent the average durability of two felt-base specimens exposed outdoors. Based on those specimens exposed outdoors that have failed, it is quite apparent that many of the findings in the weatherometer test [3] can be correlated with the exposures outdoors.

The California asphalt (Asphalt I in reference [3]) produced the least durable specimens. The Venezuela asphalt (Asphalt III in ref. [3] produced the next more durable specimens, and the Mid-Continent asphalt (Asphalt II in ref. [4])the most durable specimens. However, for some systems there was little or no difference in the durabilities of corresponding coatings made from the latter two asphalts.

For all three asphalts, mica (shown in tables 6 and 7, but not in fig. 6) made the greatest improvement in performance. Blue-black slate was also beneficial to all three asphalts. And dolomite, just as in the weatherometer tests, did not appreciably affect the durability of the three asphalts. On the other hand, clay, fly ash, and silica all are performing

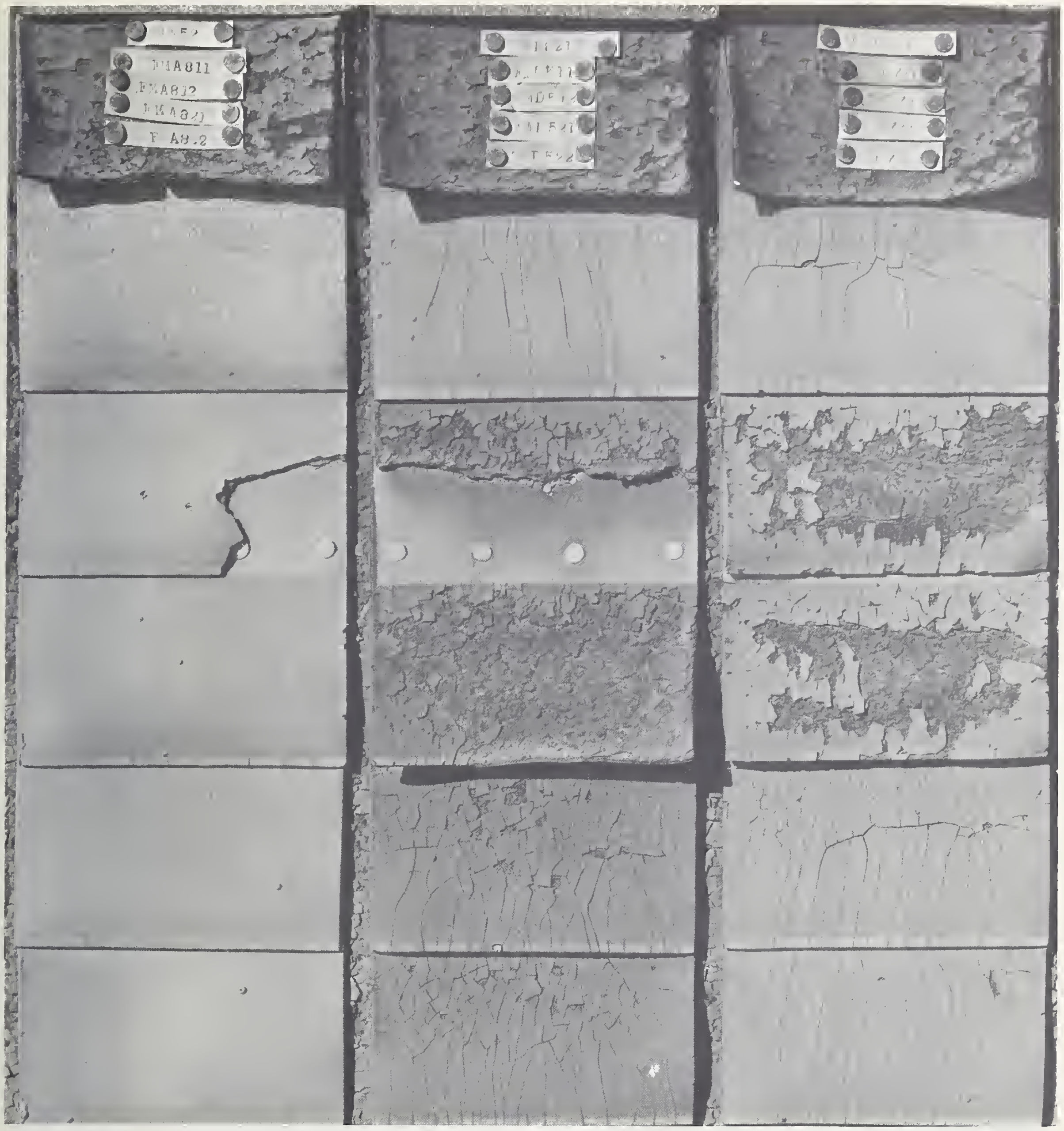

Figure 3. *Smooth-surface, felt-base specimens after 16½ years of exposure* [1]

Column 1		Column 2		Column 3	
Coating composition	Thickness	Coating composition	Thickness	Coating composition	Thickness
	mils		*mils*		*mils*
35% Mica	25	60% Fly Ash	25	60% Fly Ash	25
60% Blue Black Slate [2]	15	35% Fly Ash [2]	15	50% Fly Ash	15
60% Blue Black Slate	15	35% Fly Ash	15	50% Fly Ash	15
60% Blue Black Slate	25	35% Fly Ash	25	50% Fly Ash	25
60% Blue Black Slate	25	35% Fly Ash	25	50% Fly Ash	25

[1] Mid-Continent Asphalt.
[2] Tabs partially torn off.

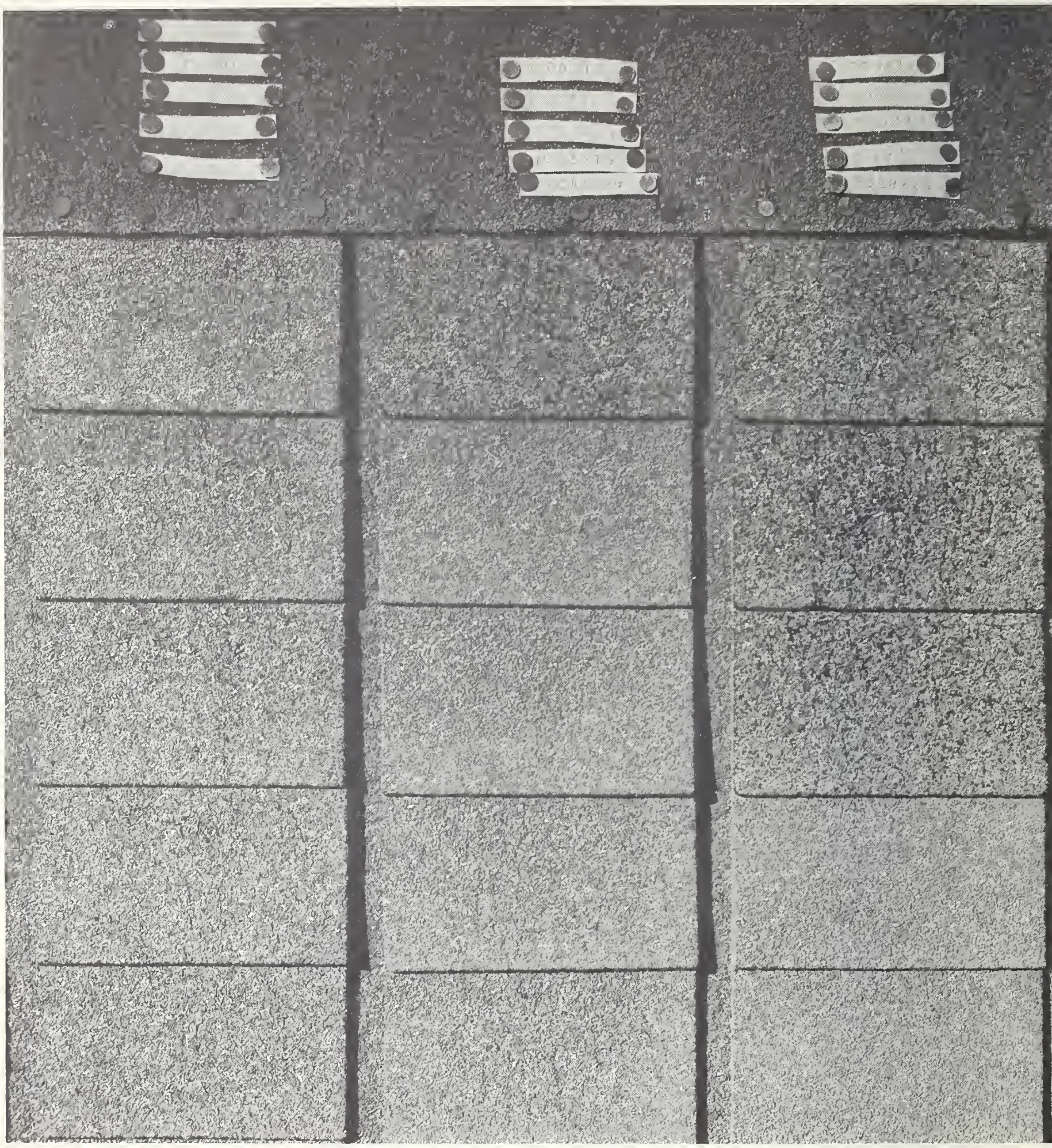

FIGURE 4. *Granule-surfaced, felt-base Venezuela specimens after 15 years of exposure*

(Initial granule coverage approximately 25 lb/100 sq ft (1.22 kg/m ^{2}))

Column 1		Column 2		Column 3	
Composition	Coating thickness	Composition	Coating thickness	Composition	Coating thickness
	mils		*mils*		*mils*
60% Blue Black Slate	25	50% Blue Black Slate	25	35% Florida Clay	25
60% Blue Black Slate	25	50% Blue Black Slate	25	35% Florida Clay	25
35% Blue Black Slate	25	35% Blue Black Slate	25	60% Blue Black Slate	25
35% Blue Black Slate	25	35% Blue Black Slate	25	60% Blue Black Slate	25

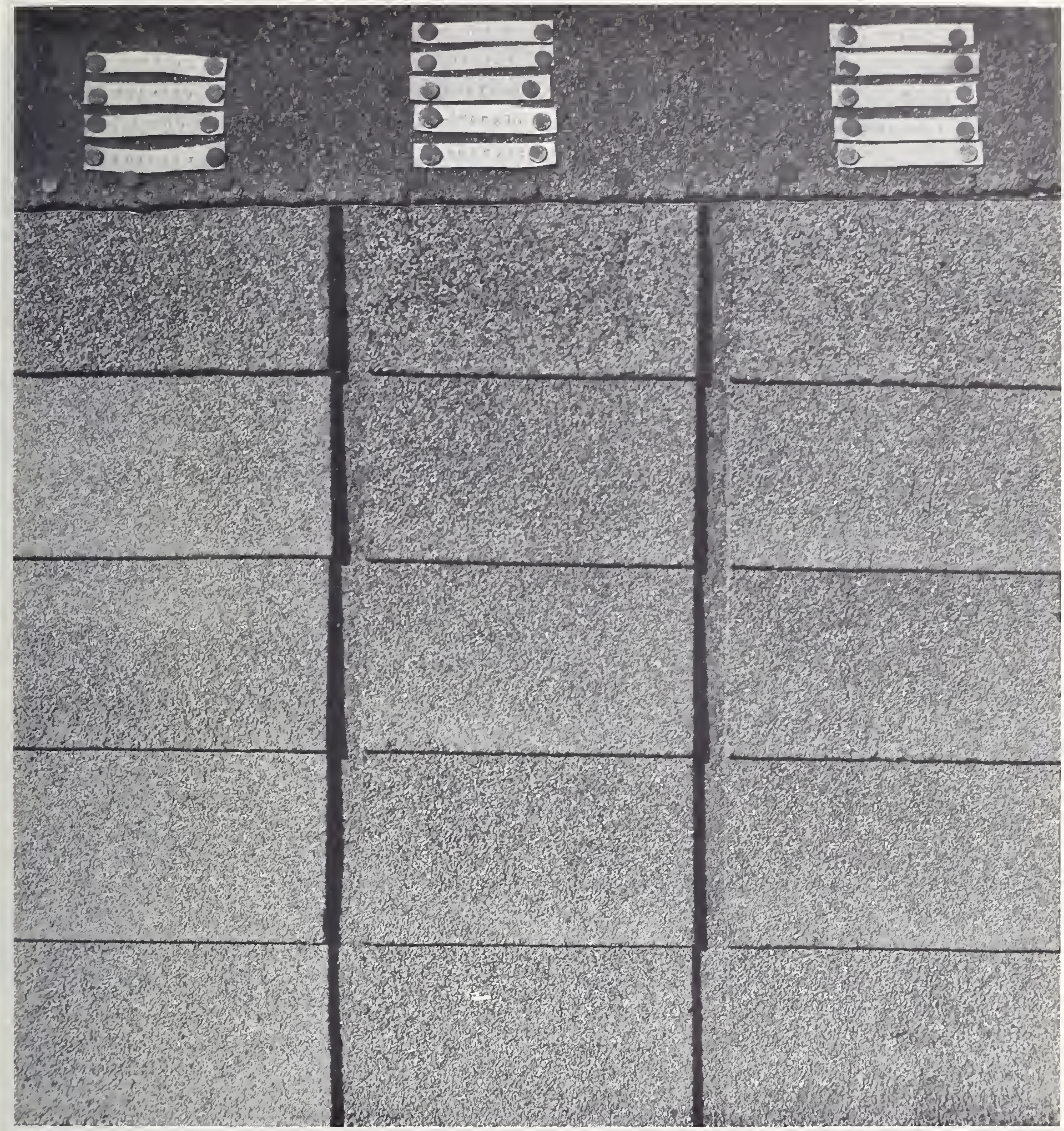

Figure 5. *Granule-surfaced, felt-base California specimens after 15 years of exposure*

(Initial granule coverage approximately 25 lb/100 sq ft (1.22 kg/m ^{2}))

Column 1		Column 2		Column 3	
Composition	Coating thickness	Composition	Coating thickness	Composition	Coating thickness
	mils		*mils*		*mils*
60% Dolomite	25	100% Asphalt	25	100% Asphalt	45
50% Dolomite	25	50% Blue Black Slate	25	100% Asphalt	45
50% Dolomite	25	50% Blue Black Slate	25	100% Asphalt	25
35% Dolomite	25	35% Blue Black Slate	25	60% Blue Black Slate	25
35% Dolomite	25	35% Blue Black Slate	25	60% Blue Black Slate	25

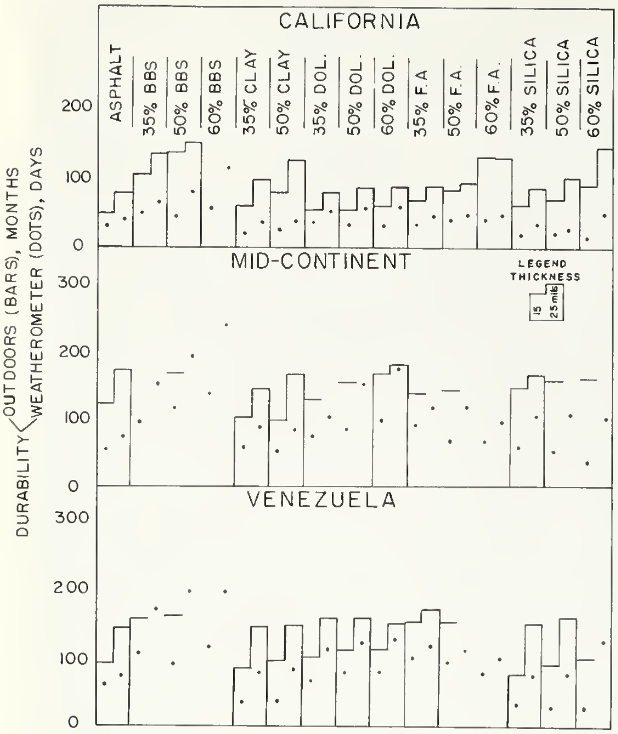

Figure 6. *Durability of smooth-surface specimens.*

The outdoor exposures are represented by bars and the weatherometer exposures by dots. Where there are no tops on the bars, the specimens have not failed after 200 months of exposure.

better than anticipated from the results of the weatherometer tests. Whereas, in a few of these systems in the weatherometers the coatings with additives were 20 percent less durable than their corresponding straight asphalt coatings, none exposed outdoors was less durable than its corresponding asphalt coating. In the extreme cases, such as 60 percent silica in the 15 mil coatings of all three asphalts, the specimens exposed outdoors were more durable than the corresponding straight asphalt specimens and the specimens in the weatherometers were roughly half as durable. Thus, where the results of the weatherometer tests have not corresponded with those obtained with the exposure outdoors, they have been on the conservative side.

The 25-mil thick coatings were more durable than the 15-mil coatings of the same mixes, but in many instances not 40 percent more durable. Both of these conditions were also apparent in the results of the weatherometer tests.

7. Conclusions

After 16 and a half years of exposure of smooth-surface and granule-surfaced roofing in Washington, D.C., the following conclusions can be drawn:

(1) All of the granule-surfaced specimens are performing satisfactorily. A few, which failed or were borderline in the granule-loss test, have lost noticeable quantities of granules during exposure.

(2) Of the smooth-surface specimens, those with 35 percent mica and 35, 50, and 60 percent blue black slate in all three asphalts have consistently outperformed the corresponding asphalt specimens without additives. The systems containing fly ash, dolomite, clay, and silica gave variable performances; each combination must be considered separately.

(3) The thicker coatings are outperforming the thinner coatings.

(4) There were differences in durability among the specimens made with the three asphalts, with the Mid-Continent asphalt being the most durable and the California the least. When mineral additives were mixed with the asphalts, corresponding differences in durability were observed.

(5) Based on the smooth-surface specimens, the results in the weatherometer tests in most instances predicted the relative durabilities of the coating. Where the weatherometers were different, the difference was in the conservative direction.

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[1] Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D.C. 20234.
[2] Located at Boulder, Colorado 80302.
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