NATIONAL BUREAU OF STANDARDS REPORT

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EFFECTS OF THERMAL SHOCK ON THE DURABILITY OF ASPHALT COATINGS UNDER ACCELERATED TEST

by

Sidney H. Greenfeld

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SYNOPSIS

Three asphalts, representative of the major sources of coating asphalt used in the manufacture of prepared roofings in the United States, were exposed, without stabilizer and with 30% and 60% of two mineral stabilizers, to accelerated durability tests. Four panels of each coating were subjected daily to 21 hours of exposure to the radiation from an enclosed, low-intensity carbon arc, with the introduction of a chilled water spray (40 F) for three minutes every 20 minutes (17-3 cycle). Two of the four panels with each coating were also exposed to air at -5 F for two hours daily. The use of the 40 F water produced results equivalent to those obtained when both the 40 F water and exposure to air at -5 F were employed.

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INTRODUCTION

Because of the continual change in the sources of asphalts and the relatively long life of roofings made from them, the need for an accelerated test for determining the probable durability of asphalt has long been recognized. Exposure of films of asphalt to the radiation of a carbon arc and intermittent water spray has been generally accepted as a useful way of determining the durability of a new asphalt relative to that of an asphalt of known performance, if the two are exposed at the same time in the same machine. Beyond the use of radiation and water, there has been little agreement on what other factors should be included in an accelerated durability test or on the amount and intensity of any of the exposure factors.

In 1939, the American Society for Testing Materials (1) adopted as a tentative standard a recommended procedure for conducting accelerated durability tests. Three 24-hour exposure cycles were included, to be used at the discretion of the operator, because "...weather varies considerably from place to place." In all three cycles, there was included a period of 1 3/4 hours of exposure to air at -10 F. In 1943, Weetman (2) conducted a systematic study of many of the variables involved in "accelerated weathering"

using a cycle very similar to the ASTM "A" cycle. Among other things, he found that in the range of -10 to +10 F, the refrigeration temperature did not affect the results.

Since that time, the commercially available models of Weather-Ometers have been changed in design to make them more automatic, and many laboratories have adopted other cycles, which required less attention to the machines. The use of refrigeration as an adjunct to the radiation and water exposure of asphalts has been discussed frequently without resulting in any definite statement of its merits.

However, it was generally agreed that in planning an extensive program, in which a large number of asphalt coating specimens would be subjected to accelerated durability tests, some form of thermal shock should be included, and that it would be desirable to introduce the shock in a manner less cumbersome than the procedure of transferring specimens to and from a freezer. It was decided to investigate the applications of this shock directly in the accelerated durability machines by using an automatically-controlled cold-water spray (40 F) as part of the exposure cycle. To establish the effectiveness of this procedure, duplicate panels subjected to this cycle, in addition, would be exposed to air at -5 F for two hours daily for comparison purposes.

EQUIPMENT AND MATERIALS

An accelerated durability machine was built at the National Bureau of Standards employing a low-intensity, enclosed, alternating-current arc (3) as a source of radiation. It was similar to commercial machines (3) but in addition to the regular voltmeter, ammeter, time and cycle clocks, it had a kilowatt hour meter and a time-delay relay, which would turn off the main power supply if the arc should fail for more than 30 seconds. A blower provided a continuous circulation of air across the exposed panels.

A commercial demineralizer was used to reduce the salt content of the spray water to less than two ppm. This water was chilled to a temperature of 40 F (±2 F) before being sprayed on the panels at a nozzle pressure of 25 psig (±5 psi).

MATERIALS

Three asphalts, representative of those used in the manufacture of prepared roofing in the United States, were used in this investigation. Each asphalt was commercially processed into five products of different softening points as shown in Table I.

TABLE I.

PROPERTIES OF ASPHALTS

Asphalt	PRODUCT 1			PRODUCT 2		PRODUCT 3		PRODUCT 4		PRODUCT 5		
	S.P.[a] deg Fahr	Pen.[b]	Sp. Gr.[c]	S.P.[a] deg Fahr	Pen.[b]	S.P.[a] deg Fahr	Pen.[b]	S.P.[a] deg Fahr	Pen.[b]	S.P.[a] deg Fahr	Pen.[b]	Sp. Gr.[c]
Source I	187	31.7	1.013	197	25.3	211	21.7	213	20.0	223	18.8	1.017
Source II	185	29.0	0.995	196	25.0	211	21.8	220	19.0	231[d]	17.2	1.003
Source III	185	25.3	1.015	189	24.5	207	20.7	218	18.0	224	16.8	1.021

[a]Softening Point determined by R & B Method, ASTM D36-26.
[b]Penetration determined by ASTM Method D5-49 at 77 F, 100 g, 5 sec.
[c]Specific Gravity was determined only on Products 1 and 5.
[d]This product was not used because of its high softening point.

Two commercial, non-metallic, mineral stabilizers, having the properties and attributes shown in Table II, were used in this investigation.

Table II. Characteristics of Stabilizers

Stabilizer	Stabilizer A	Stabilizer C
Moisture, % by weight	0.2	0.1
Loss on Ignition, % by weight:		
1000 F	2.1	1.7
1800 F	5.4	43.6
Water Soluble, % by weight	0.0	0.0
Free Alkali, % by weight	0.0	0.0
Sieve Analysis, % by weight passing U. S. Sieves:		
#60	99.8	99.9
#80	99.3	99.9
#100	97.9	99.6
#120	96.2	99.3
#170	91.3	96.6
#200	86.9	93.4
#270	83.6	89.9
#325	76.7	81.0
Particle Shape	Flat Plate	Blocky
Density, g/cc	2.94	2.87
Surface Area,[a/] m^2/g	1.0	2.0
Oil Absorption g/100g. (mineral oil)	29.5	19.5

a/ Determined by Nitrogen Adsorption.

Asphalt products (adjacent in Table I) from each source were blended with the proper concentrations of stabilizers to produce coatings (with the exception of 60% Stabilizer A) with softening points in the range of 221-226 F. Although those containing 60% Stabilizer A were made with 40% of Product 1, their softening points were as high as 237 F. For identification purposes, each

coating is designated by the asphalt source followed by the concentration and letter of the stabilizer; i.e., II60 C is 60% of Stabilizer C in Source II asphalt.

PREPARATION AND EXPOSURE OF PANELS

The panels to be exposed were made by spreading the asphalt coating on a 2 3/4- by 6- by 0.064-in. aluminum sheet with the aid of a hydraulic press as described in reference (4). All coating thicknesses were between 0.023 and 0.027 in., with a maximum variation of ±0.001 in. on any individual panel. Panels were calipered immediately after they were prepared and were exposed within 24 hours.

Six panels were made from each of 15 different coatings: three coatings were made without stabilizers, six with 30% of two stabilizers, and six with 60% of two stabilizers. Two of these panels, designated "1" and "2", were exposed to the four steps of cycle A:

(1) alternating 17 minutes of radiation and three minutes of radiation and cold-water (40 F) spray for 21 hours.

(2) inspection for 1/2 hour.

(3) exposure to air at -5 F for two hours.

(4) inspection for 1/2 hour.

Two of these panels, designated "3" and "4", were exposed to the two steps of Cycle B:

(1) alternating 17 minutes of radiation and three minutes of radiation and cold-water (40 F) spray for 21 hours.

(2) exposure to air at 77 F for three hours.

Cycle A differs from Cycle B only in the two-hour exposure to air at -5 F.

To compare the results of machine exposure with actual weathering, two panels were exposed out-of-doors on the roof of the Industrial Building, National Bureau of Standards, at an angle of 45° facing south. (At the end of 2 1/2 years, no failures had occurred.)

Those panels exposed in the accelerated durability machines were held by stainless steel clips, two to an aluminum support, one above the other. On the odd-numbered days, the supports were inverted; and on the even-numbered days, the panels were inverted in their supports. In this way, the vertical variation in intensity of light and heat was taken into consideration. Once a week, all of the panels were inspected by means of a high-voltage probe (5) to detect any openings in the coatings. These inspections were made on dry panels following a water-spray period. In Cycle A, inspections were made both preceding and following

the refrigeration period. Those panels showing a partial failure were spark-photographed according to the procedure of Hunter, et al. (6) When any panel developed failures on a minimum of 50% of its surface, as determined by means of a 60-square grid, (i.e., failure in 30 of the 60 areas on the grid) it was removed from the machine as a final failure.

RESULTS

After failure each panel was spark-photographed after it was removed from the machine and was then stored in a dark place at room temperature for future reference. The exposure of those panels containing 60% Stabilizer A and asphalts from Sources II and III was discontinued after 300 cycles, even though final failure had not occurred.

Table III is a summary of the durability of all of the panels, arranged according to increasing stabilizer concentration. Figure 1 is a bar graph of the data in Table III presenting a comparison of the refrigerated and non-refrigerated coatings.[2/]

In all instances, all four panels of each kind of coating, two refrigerated and two not refrigerated, showed the same type of failure pattern. Figures 2, 3, and 4 are

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[2/] For this discussion the term "refrigerated" will refer to the panels that were exposed to air at -5 F.

TABLE III.

LENGTH OF EXPOSURE TO FINAL FAILURE

Description	Refrigerated Panels[a] (Cycle A)			Non-Refrigerated Panels[b] (Cycle B)		
	1	2	Average	3	4	Average
	Days[c]	Days[c]	Days[c]	Days[c]	Days[c]	Days[c]
I	68	68	68	74	74	74
II	79	79	79	86	79	82
III	110	103	106	89	89	89
I 30 A	82	82	82	82	82	82
II 30 A	87	87	87	106	93	100
III 30 A	164	157	160	150	129	140
I 30 C	72	72	72	72	72	72
II 30 C	96	82	89	89	96	92
III 30 C	148	148	148	155	155	155
I 60 A	-	219	219	219	213	216
II 60 A	300[d]	300[d]	300[d]	300[d]	300[d]	300[d]
III 60 A	300[d]	300[d]	300[d]	300[d]	300[d]	300[d]
I 60 C	72	66	69	72	72	72
II 60 C	115	115	115	122	122	122
III 60 C	154	147	150	140	147	144

[a] Inspected after the exposure at -5 F.
[b] Inspected after the spray period.
[c] Multiply by 21 to get hours of radiation, by 2 to get hours of exposure at -5 F (for numbers 1 and 2), and by 3.15 to get hours of spray.
[d] Discontinued before final failure.

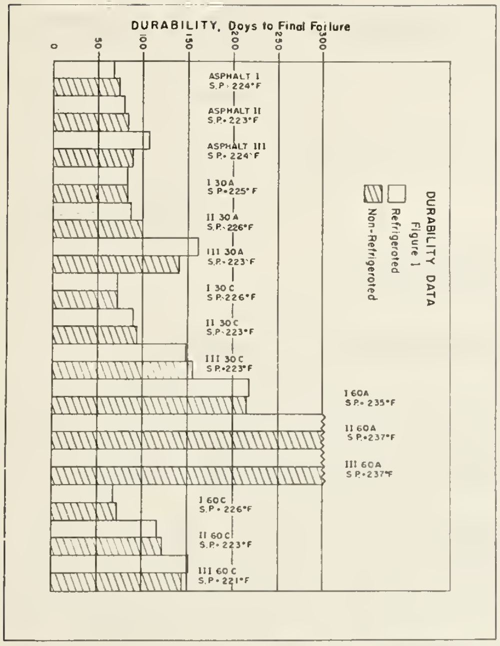

FIG. 1. A COMPARISON OF THE DURABILITY OF REFRIGERATED AND NON-REFRIGERATED COATINGS AT FINAL FAILURE.

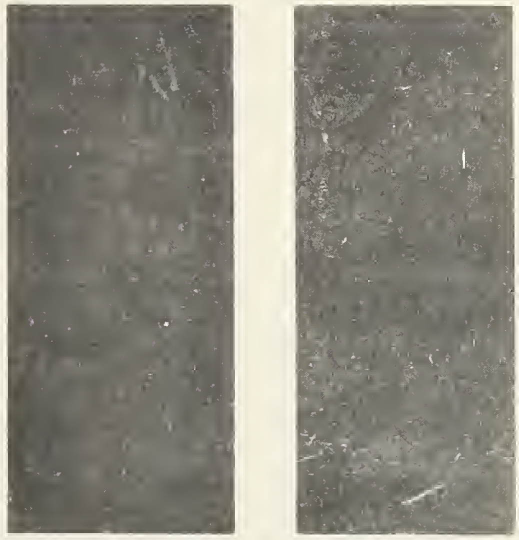

FIG. 2. THE MOST FREQUENTLY OCCURRING TYPE OF FAILURE IS REPRESENTED BY UNSTABILIZED SOURCE II ASPHALT SHOWN IN THIS PHOTOGRAPH. THE PANEL ON THE RIGHT WAS EXPOSED TO AIR AT – 5°F.

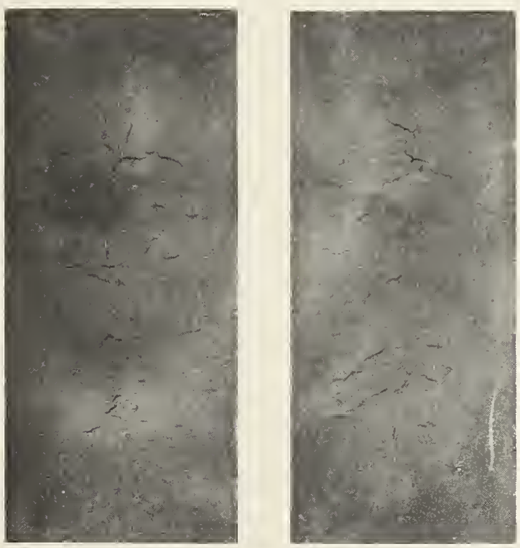

FIG. 3. COATING I–60 A. THE PANEL ON THE RIGHT WAS EXPOSED TO AIR AT – 5°F.

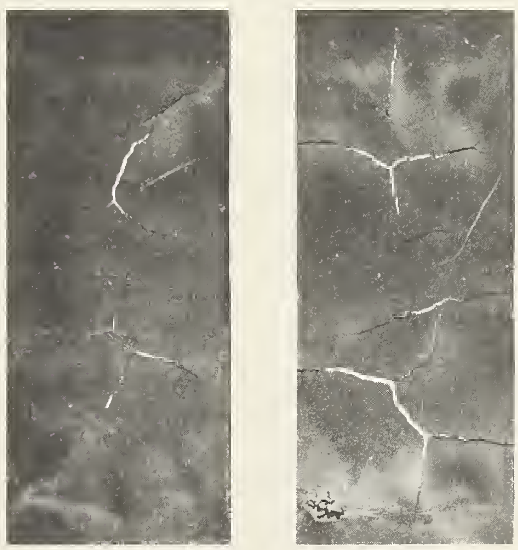

FIG. 4. COATING II–60 A. THE PANEL ON THE RIGHT WAS EXPOSED TO AIR AT – 5°F.

photographs of typical sets of panels showing the similarity of the crack patterns in each set. All of the panels failed with one of these three types of patterns.

DISCUSSION OF RESULTS

Because inspections for failures were made weekly, differences in durability of seven days or less are not significant. From Table III it can be seen that in all but three pairs of panels similarly exposed, failures were revealed either concurrently or during consecutive inspections. Of the three pairs that did not follow this pattern, one was refrigerated, Panels II 30 C; and two were not, Panels II 30 A, and Panels III 30 A. Thus, refrigeration added very little to the degree of reproducibility of results.

Figure 1 shows that in only three instances were there more than seven cycles, or one inspection period, difference between the durabilities of the refrigerated and corresponding non-refrigerated panels. In one case, Panels II 30 A, the refrigerated panels failed 13 cycles earlier than the non-refrigerated ones; but in two cases, Panels III and Panels III 30 A, the refrigerated panels actually lasted longer than those not refrigerated by 17 and 20 cycles, respectively! Thus, it must be concluded that refrigeration did not hasten the failure

of the coating asphalts under these conditions. (It should be noted that two of these three cases involve asphalt stabilizer mixtures in which the duplicate panels failed more than seven cycles apart.)

The photographs in Figures 2, 3, and 4 show all of the crack patterns that were present in the failed coatings. In every instance all four panels of each coating, the two that were refrigerated and the two that were not, had similar crack patterns. Refrigeration at -5 F did not modify the type of failure or alter the durability of the coatings insofar as these characteristics were revealed by the spark-photographs or the appearance of the crack patterns.

The 17-3 cycle (17 minutes of radiation followed by three minutes of radiation and water spray) was designed to duplicate in the new machines, in which the drum rotates at one r.p.m., the conditions of exposure that prevailed in the older Weather-Ometers, in which the drum rotated once every twenty minutes. The chilled water (40 F) was added to increase the thermal shock to which the panels were periodically subjected in order to accelerate the failure and possibly to eliminate the necessity for the use of refrigeration. The data show that for this cycle, exposure

to air at -5 F does not alter the durability of the coatings. Further, the frequent periodic cold-water shocks fatigued the coating asphalts rapidly, and very little difference was found between the number of failures after the refrigeration period and after the spray periods, examinations having been made at both times. Thus, if chilled spray water is used, the wide divergence in durability introduced by varying the time of inspection that has been reported by other investigators is no longer apparent (6).

CONCLUSIONS

Exposure to air at -5 F has no significant effect on the durability or failure pattern of both stabilized and unstabilized asphalts under accelerated test if the test includes frequent cyclic thermal shocks.

ACKNOWLEDGMENT

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(1) "Tentative Recommended Practice for Accelerated Weathering Test of Bituminous Materials" (D529-39T), A.S.T.M. Standards 42, II, 1335, (1942).

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(4) Greenfeld, S. H., "A Method for Preparing Uniform Films of Bituminous Materials", A.S.T.M. Bulletin.

(5) Boenau, A. H., and Baum, L. H., "The Design and Application of a Spark-Gap Instrument for Detecting Crack Failures of Asphalt Coatings During Weathering Tests", A.S.T.M. Symposium on Accelerated Durability Testing of Bituminous Materials, 1949.

(6) Hunter, J. B., Gzemski, F. C., and Laskaris, L., "A New Method for Evaluating Failure of Bituminous Materials Due to Weathering", A.S.T.M. Symposium on Accelerated Durability Testing of Bituminous Materials, 1949.

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