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NATIONAL BUREAU OF STANDARDS REPORT

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A METHOD OF PREPARING UNIFORM

FILMS OF BITUMINOUS MATERIALS

by

Sidney H. Greenfeld

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A METHOD OF PREPARING UNIFORM FILMS OF BITUMINOUS MATERIALS

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Sidney H. Greenfeld[1/]

SYNOPSIS

A hydraulic press was adapted to the preparation of uniform films of both stabilized and unstabilized bitumens for exposure out-of-doors and to accelerated durability tests. Films in the range of 0.005 to 0.050 in. were made consistently within a maximum variation of ±0.001 in. in each film with fewer than 10 per cent rejections. Coatings up to 0.100 in. thick can also be made.

INTRODUCTION

One of the problems encountered in the laboratory testing of bituminous materials is the reduction of these materials to uniform films, for many of their properties are functions of film thickness. The problem has been approached from various points of view with varying degrees of success.

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Probably the earliest methods of preparing films of bitumen on rigid supports involved the pouring of the material on the support and heating both, while the support was maintained in a horizontal position, until a more-or-less uniform film was formed. Early investigators recognized the shortcomings of this method, and in 1930, Dr. O.G. Strieter (1) described the earliest form of the apparatus in current use. He mounted a heated, hollow, brass cylinder horizontally in such a manner that its height above a wooden bed could be adjusted. A rigid panel covered with molten asphalt was passed beneath this heated cylinder a number of times until the desired film thickness was attained.

An apparatus similar to the above, but in a refined form, became available commercially[2/] (2) and was adopted by many laboratories throughout the United States. A more advanced apparatus in which the entire bed was moved under a heated, stationary doctor bar by means of a geared drive was developed by Kirschbraun and was also reported by Strieter (1); however, this apparatus has not been very widely accepted. Because of the surface configurations and irregularities introduced by these doctor-bar methods, many investigators gave their panels various heat treatments to produce nearly uniform surfaces.

[2/] Panel Trimmer, Atlas Electric Devices Mfg. Co., Chicago, Ill.

If the operators of the doctor bar are very skillful and extremely careful, it is possible to obtain a fairly high percentage of acceptable panels when bitumens unmixed with other materials, such as stabilizers, are used. While most investigators find it difficult to maintain a tolerance of ±0.003 in. (in the 0.025-in. range), some claim to be capable of producing a ±0.001-in. tolerance. However, very often streaks are left in the coating and the asphalt near the surface is changed markedly due to its prolonged exposure to the elevated temperature of the doctor bar. The necessary cleaning and readjusting of the bar after each pass make the task of preparing suitable panels unduly long and onerous.

A new difficulty was encountered using the doctor-bar method when attempts were made to make coatings of bitumens containing mineral stabilizer. It was observed that even during the first pass of the coating under the doctor bar, the appearance of the surface changed materially. The prolonged period at elevated temperatures required for the numerous passes permitted the stabilizer to settle from the surface, leaving it deficient in stabilizer and, consequently, causing the remainder of the coating to be of a higher concentration of stabilizer than desired.

A method that could eliminate these objections was sought for preparing uniform films of bituminous materials over a wide range of thicknesses. In the search for this method, it was observed[3/] that a hydraulic press could be adapted if certain precautions were taken to prevent the bitumen from adhering to the press platens and to retard the cooling of the bitumen until it had spread to the desired thickness. This paper describes the precautions taken to obtain consistently the required thickness and uniformity of coating.

EQUIPMENT AND MATERIALS

The bitumen being tested was melted in the apparatus shown in Figure 1. The exposure specimens were made on a hydraulic press, which is shown in Figure 2, together with auxiliary equipment: feeler-gauge spacers, variac-controlled hot plate, thickness gauge, and aluminum panel fastened to a sheet of Kraft paper with masking tape.

The materials used directly for making exposure panels were aluminum-base panels, 2 3/4 by 6 by 0.040-0.070 in., Kraft and dextrin-coated paper, 6 by 8 by 0.005 in., and gray cardboard, 6 by 8 by 0.020-0.030 in. (all

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3/ The Ruberoid Company has used a press with amalgamated platens for a number of years.

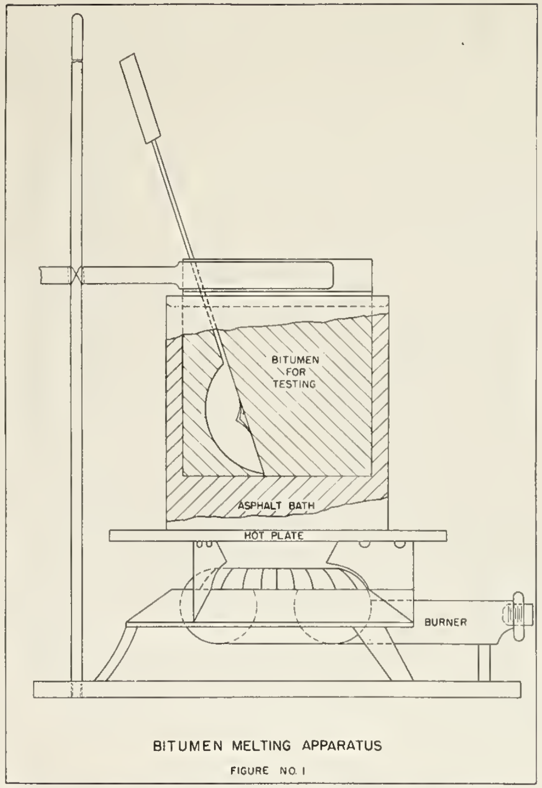

FIG. 1. BITUMEN MELTING BATH – THE BITUMEN UNDER TEST IS MELTED IN THIS TYPE OF ASPHALT BATH TO PREVENT ITS BEING DEGRADED BY LOCAL OVERHEATING.

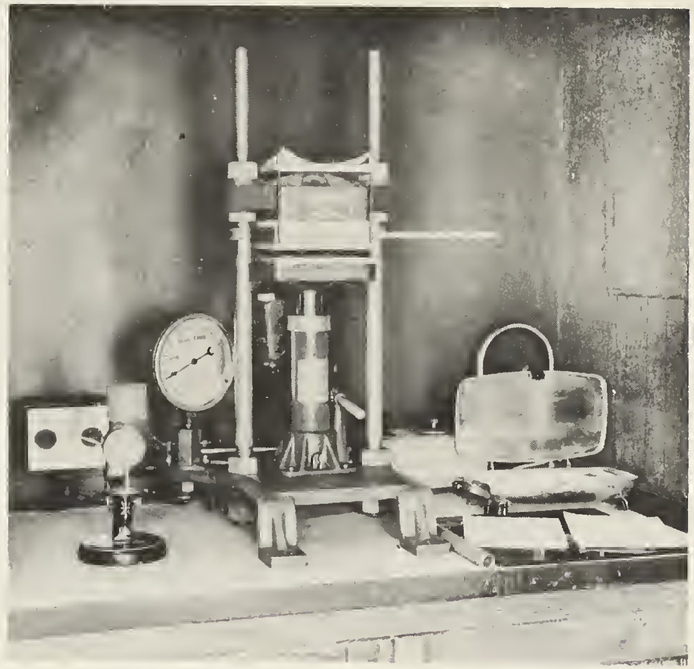

FIG. 2. PANEL MAKING EQUIPMENT – THE HYDRAULIC PRESS AND AUXILIARY EQUIPMENT ARE SHOWN IN OPERATING POSITIONS PRIOR TO THE MAKING OF AN EXPOSURE PANEL.

calipering ±0.0005 in.). When the upper platen of the press was heated, the cardboard was omitted and aluminum panels as thin as 0.032 in. could be used.

In addition to the above equipment, a tank of running water, two wide-mouth glass containers of carbon tetrachloride, metal-marking letters, a vise, a file, and thermometers were used in the procedure.

PREPARATION OF MATERIALS

In order to obtain uniformity of results, it was necessary to clean the aluminum panels well. A number of aluminum panels of the same thickness were calipered, deburred, and placed in the first jar of carbon tetrachloride. After about 10 minutes, the panels were transferred individually to the second jar of solvent. Each panel was removed individually from the second jar and, after it had air-dried, its surface was rubbed with a clean, lint-free cloth to remove any dust or loosely adhering films of insoluble materials. Other methods of cleaning the panel surface may be satisfactory, but this was a minimum treatment. Each panel was fastened to a sheet of Kraft paper with masking tape, covering one-quarter inch on three sides and one-half inch at one of the ends (for the panel number). For the thinner films the tape was not put along the sides of the panels. A panel was put in the hot grill with a thermometer in contact with its surface.

The bitumen was melted in the melting bath at 400-425 F, maintained at that temperature, and continually stirred until the sample had a smooth, bubble-free appearance.

A set of spacers was placed on each side of the lower platen; these were equal to the combined thickness of the aluminum panel and the desired coating thickness plus about 0.003 in. to allow for thermal expansion.

PROCEDURE

The panel was removed from the grill when it reached a temperature of 300 F and was placed on a flat, thermally-insulated surface. About 1 1/4 times the quantity of bitumen needed was poured on the panel. Any bubbles present were broken by tapping the panel against a solid object or by contacting with a graphite rod, i.e., a long-pointed pencil.

The bitumen was covered with a sheet of dextrin-coated paper and a sheet of cardboard. When the upper platen of the press was heated to within about ±20 F of the softening point of the coating, the upper cardboard was omitted. The timing of the procedure was not nearly so critical when the

platen was heated.[4/] The assembly was placed in the press in such a manner that the spacers were under the paper and cardboard, but not the masking tape.

The press was closed with a thrust of approximately 3500 lb. This force was taken up by the spacers and was only large enough to insure the spreading of the bitumen to the thickness defined by the spacers before it solidified. The entire procedure took 5 to 10 seconds. After about 30 seconds, the assembly was removed from the press and the panel was immersed in the water bath. After the dextrin-coated paper floated free, the panel was removed from the water, washed thoroughly, stripped of its masking tape, and dried. The panel was then calipered. The timing of the above procedure was critical when the upper platen was not heated.

The most important single variable which was subject to control was the bitumen temperature. Panels were most easily made when the bitumen temperature was such that its viscosity was in the range of 100-500 centipoises. However,

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[4/] Heating the upper platen was suggested in a private communication from Mr. G.W. Clarvoe, Johns-Manville, Manville, N. J.

by adjusting the spacers and the insulation above and below the panel, films of controlled thickness were made from melts with viscosities as high as 10,000 centipoises.

For identification purposes a number was pressed into the bare aluminum at the top of the panel by means of a bench vise.

Panels as large as 9 by 12 in. were also made on saturated felt with a large hydraulic press. The method was modified slightly, as follows:

> Felts were heated for about two hours at 300 F. The air and moisture had to be driven from the felt, and the saturant re-absorbed. A sheet of felt was placed on a sheet of cellophane (instead of Kraft paper) and, after the asphalt was poured, a sheet of dextrin paper and two sheets of Kraft paper were placed on it. This assembly was sandwiched between two Masonite boards which were placed between the platens of a large press.

The remainder of the procedure was the same as for aluminum-based panels.

Unsupported films can be made by the press procedure, the only change being the substitution of a sheet of dextrin paper for the aluminum panel. The soaking period, of

course, removes both the upper and lower papers, leaving a free film.

RESULTS

Once suitable conditions were established for any particular bitumen, it was possible to obtain, consistently and rapidly, panels with a maximum variation of ±0.001 in. with fewer than 10 percent rejections. Although this procedure was developed for making coatings of asphalt in the range of 0.010- to 0.050-in. thick, some have been made with thicknesses as low as 0.005 and as high as 0.100 in. Typical results are shown in Table 1.

The results in Table 1 have been picked at random in the 0.013 to 0.043 in. range from data on hundreds of panels made by these two procedures. A variation in individual panels up to two mils can be seen readily, as can the possible differences among consecutive panels. Despite the simplicity of operation inherent in both procedures, the necessity for timing and coordination of the several steps in each procedure remains. Those coatings designated by (b) and (c) show what happened when deviations from the normal timing occurred. Tolerances as high as ±0.003 in. appeared to be an acceptable operating range when felt-based

TABLE 1.

REPRESENTATIVE VARIATIONS OBTAINED BY THE PRESS METHOD OF PREPARING ASPHALT FILMS ON ALUMINUM

The coating thickness for each panel is the average, to the nearest mil, of eight measurements; the variations are the maximum deviations from this average.

COATING COMPOSITION	PLATEN AT AMBIENT TEMPERATURE			PLATEN HEATED[a]		
Proportion of Stabilizer by Weight	CARDBOARD INSULATION ABOVE PANEL			NO INSULATION		
	Coating Thickness			Coating Thickness		
	Thickness	Maximum Variation		Thickness	Maximum Variation	
percent	mils	mils +	mils −	mils	mils +	mils −
0	25	0	1	25	0.5	0
	25	1	1	25	1	0
	23 (b)	1	1	25	0	1
	25	1	1	25	0.5	1
35	13	1	0	5	1	0.5
	14	1	0.5	5	1	0
	13	0.5	0.5	5	1	0.5
	16 (c)	1	1	-	-	-
50	43	0	1	13	1	0.5
	43	0	1	13	1	0.5
	43	0.5	0	13	1	0.5
	43	0.5	0	13	1	0.5
60	13	1	1	25	0.5	1
	14	1	0	25	1	1
	13	1	0.5	25	1	1
	13	0.5	0.5	25	1	0

(a) A few panels have been made with coating thicknesses of 0.100 in. by this procedure with some difficulty.
(b) Press closed too rapidly.
(c) Press closed too slowly.

panels were made, because of the considerably greater variation in the thickness of the saturated felt, as compared with the aluminum panels.

The methods described proved successful on a wide variety of materials including coal tars and many types of asphalt, unstabilized and stabilized with a variety of mineral stabilizers in concentrations up to sixty per cent by weight.

This panel-making technique was developed on the Mineral Stabilizer Project, sponsored by the Asphalt Roofing Industry Bureau at the National Bureau of Standards. The author expresses his thanks and appreciation to Mr. Wm. H. Appleton for his assistance in the laboratory and to members of the Bureau staff for advice and suggestions.

REFERENCES

(1) O. G. Strieter, B. S. Journal of Research 5, 247-253, 1930, (RP197).

(2) Abraham, H., Asphalts and Allied Substances, 5th Ed., 1484 (1945), and "Proposed Method for Accelerated Weathering Tests of Bituminous Materials", Proc. A.S.T.M., 33, Part I, 383, 1933.

THE NATIONAL BUREAU OF STANDARDS

Functions and Activities

The functions of the National Bureau of Standards are set forth in the Act of Congress, March 3, 1901, as amended by Congress in Public Law 619, 1950. These include the development and maintenance of the national standards of measurement and the provision of means and methods for making measurements consistent with these standards; the determination of physical constants and properties of materials; the development of methods and instruments for testing materials, devices, and structures; advisory services to Government Agencies on scientific and technical problems; invention and development of devices to serve special needs of the Government; and the development of standard practices, codes, and specifications. The work includes basic and applied research, development, engineering, instrumentation, testing, evaluation, calibration services and various consultation and information services. A major portion of the Bureau's work is performed for other Government Agencies, particularly the Department of Defense and the Atomic Energy Commission. The scope of activities is suggested by the listing of divisions and sections on the inside of the front cover.

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The results of the Bureau's work take the form of either actual equipment and devices or published papers and reports. Reports are issued to the sponsoring agency of a particular project or program. Published papers appear either in the Bureau's own series of publications or in the journals of professional and scientific societies. The Bureau itself publishes three monthly periodicals, available from the Government Printing Office: The Journal of Research, which presents complete papers reporting technical investigations; the Technical News Bulletin, which presents summary and preliminary reports on work in progress; and Basic Radio Propagation Predictions, which provides data for determining the best frequencies to use for radio communications throughout the world. There are also five series of nonperiodical publications: The Applied Mathematics Series, Circulars, Handbooks, Building Materials and Structures Reports, and Miscellaneous Publications.

Information on the Bureau's publications can be found in NBS Circular 460, Publications of the National Bureau of Standards ($1.00). Information on calibration services and fees can be found in NBS Circular 483, Testing by the National Bureau of Standards (25 cents). Both are available from the Government Printing Office. Inquiries regarding the Bureau's reports and publications should be addressed to the Office of Scientific Publications, National Bureau of Standards, Washington 25, D. C.

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