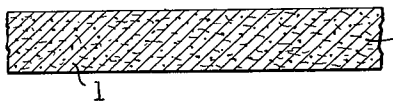
Feb. 1, 1955 W. F. FAIR, JR 2,701,219

BITUMINOUS MASTICS PRODUCTS

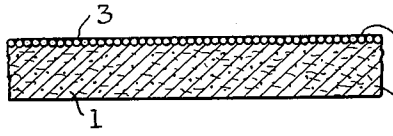
Filed April 24, 1951

Fig.1



IMPROVED BITUMINOUS MASTIC CONTAINING FILLER AND FIBRE.

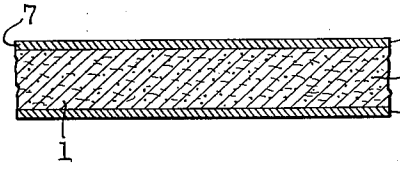
Fig.2



IMPRESSED GRANULES OR MINERAL DUST.

IMPROVED BITUMINOUS MASTIC.

Fig.3

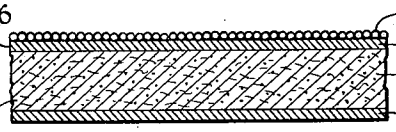


COAT.

IMPROVED BITUMINOUS MASTIC.

COAT.

Fig.4



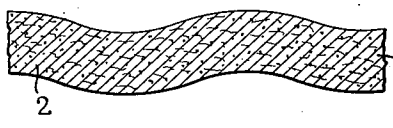
GRANULES OR MINERAL DUST.

DIP-COAT.

IMPROVED BITUMINOUS MASTIC.

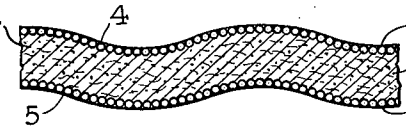
DIP-COAT.

Fig.5



IMPROVED BITUMINOUS MASTIC CONTAINING FILLER AND FIBER.

Fig.6

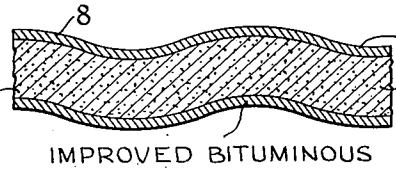


IMPRESSED GRANULES OR MINERAL DUST.

IMPROVED BITUMINOUS MASTIC.

IMPRESSED GRANULES OR MINERAL DUST.

Fig.7



IMPROVED BITUMINOUS DIP-COATING.

MASTIC.

IMPROVED BITUMINOUS DIP-COATING.

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BITUMINOUS MASTICS PRODUCTS

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Application April 24, 1951, Serial No. 222,639

7 Claims. (Cl. 117—168)

The present invention relates to improvements in bituminous mastic sheet material and methods of preparing such material.

Bituminous mastics have been widely used for various purposes as cements, as coatings for surfaces, as flooring, and on walls. They have generally been applied by brushing, or trowelling and rolling, or in the form of solid blocks or cakes. Little success has been realized commercially in the past in providing a coherent, flexible, self-sustaining, prefabricated, non-reenforced type of bituminous mastic sheet that can be sold and handled as such, as distinct from saturated felt sheets or sheets containing reenforcing webs or inserts. Conventional bituminous mastic was considered objectionable for prefabricated sheeting for various reasons and mainly because it become too brittle in cold weather or was subject to fusion and flow in hot weather.

The present invention overcomes the objections raised with respect to previously known bituminous mastic sheets by providing a prefabricated bituminous mastic sheet that is pliable at low atmospheric temperatures and that shows no tendency to flow at high atmospheric temperatures, and that retains its flexible, solid-sheet form. The sheet is for wide commercial application in view of its high insulation value, fire resistance, resistance to weathering, inertness to the action of water and ultraviolet light, resistance to flow, and its non-cracking and non-shattering characteristics. By treatments described below, these desirable properties are also effectively imparted to bituminous mastic sheets or to conventional tar, pitch and asphalt mastics, rendering them more highly useful in structures such as roofing including shingles, rolls, sidings, panels, insulation, subflooring, expansion joints, and other articles.

In the preparation of the improved mastic sheet, the materials employed include coal-digestion pitch, a finely divided filler, and discrete fiber particles. Coal-digestion pitch comprises coal dispersed by digestion in a heat-liquefiable bituminous medium. The medium contains heavy hydrocarbon oil; or heavy hydrocarbon oil containing a preponderating proportion of aromatic hydrocarbons or constituents is added during or preferably after the digestion and dispersion process, or during or preferably after thermal treatment of coal by such process.

In the accompanying drawings, Figs. 1 to 7 are diagrammatic illustrations, in vertical section, of fragments of sheets made in accordance with the present invention.

A sheet, in flat form **1** (Figs. 1 to 4) or in corrugated form **2** (Figs. 5 and 6), is prepared from a mixture, more particularly described hereinbelow, which is shaped by rolling, extrusion, or by compression in a mold. The improved bituminous mastic, because of its composition, lends itself well to shaping into sheets by any suitable mechanical means. To provide for additional surface protection, granules or ducts of solid, inert, oil and water-insoluble, heat-resistant mineral, or metallic, or artificial pigment material, are distributed on the surface of a sheet. Colored granules or dusts are employed for producing various color schemes. The granules or dust particles are anchored as at **3** (Fig. 2) on one side of a sheet, or as at **4** and **5** (Fig. 6) on both sides of a sheet by impressing them into the surface, or they are bonded to the surface by means of a bituminous cement or a dip-coat of coal-digestion pitch, as described below, and as shown at **6** (Fig. 4).

Suitable coatings applied by brushing or spraying, or preferably a dip-coat, as shown at **7** (Fig. 3), are provided for various purposes, if desired.

In preparing the coal-digestion pitch, coal, such as bituminous coals or coking coals including either high- or low-volatile bituminous coal, and certain commercial non-coking bituminous coals, is heated while admixed with tar or pitch. The temperature of the mixture while stirring the latter, is gradually increased over an extended period of time to substantially 300° C. or preferably to a temperature in the approximate range of 300° C. to 310° C. The temperature employed is generally not lower than about 270° C. nor higher than about 350° C. The coal is uniformly distributed in the course of the thermal digestion and mixing process and, in effect, constituents of the coal at the temperature employed appear to undergo a dissolution in the tar or pitch. Depending on the temperature and the time of digestion, it has in certain instances been impossible to determine whether a chemical change has occurred in the coal, and in other instances such change has been more or less definitely detectable. For purposes of the present invention, control tests comprising softening point and penetration determinations are employed to provide a sheet with pitch having the designated improved characteristics resulting from a digestion and blending process.

To obtain a pitch of highly desirable characteristics for the purposes of the present invention, either before but preferably after dispersion of coal in the tar and/or pitch in the digestion process, there is added the above-mentioned heavy hydrocarbon oil. The heavy hydrocarbon oil is a high-boiling distillate obtained by distilling tar and separating the distillate recoverable above approximately 300° C. Only a minor proportion (about 15 per cent or less) of the oil boils below 300° C. The boiling points may be within the approximate range of 250° C. to 450° C.

Heavy hydrocarbon oils that have been employed are such oils as heavy water gas tar heavy oil, light water gas tar heavy oil; and coal tar heavy oils from tars known as low temperature coal tar and high temperature coal tar.

Heavy water gas tar heavy oils, used advantageously in the mastic, are substantially aromatic in character, and are obtained by distillation from heavy water gas tar which in turn is obtained from water-gas generator plants in which Bunker-C or similar grades of residual petroleum fuel oil are used for carburetting. Heavy water gas tar is also known as residuum tar and is thus termed to distinguish it from what was formerly known as water gas tar or is known today specifically as light water gas tar. Light water gas tar heavy oil is obtained from light water gas tar which is produced in the carburetter of a water gas plant when petroleum distillates are used as carburetting agents.

Depending upon the proportions of coal, tar and/or pitch, and/or heavy oil, in the coal-digestion pitches, or on the proportion of such oil added to these pitches the resulting pitch products have been prepared with ring and ball softening points in the approximate range of 35° C. to 150° C., all exhibiting improved rheological properties, as well as considerably better temperature susceptibilities (that is, less change in viscosity with temperature change) and greater resistance to flow than the commonly used bitumens, when in layers or on surfaces in vertical position as well as in inclined position.

The following, by way of illustration, are examples of coal-digestion pitch that is employed in the present invention:

A. About 187 parts by weight of heavy water gas tar and about 47 parts by weight of powdered bituminous coal are heated together in a still preferably while stirring. The temperature is gradually raised to a temperature of about 305° C. over a period of approximately five and one-half hours. This temperature is maintained for about four hours during which time there results a distillate of about 5 to 6% based on the tar. At the end of this time heating is discontinued and the mixture permitted to cool. After about forty minutes and while the temperature is slowly dropping, about 54 parts by weight of heavy

water gas tar heavy oil are stirred into the heat-treated coal and tar mass. The resulting product is discharged from the still at around 225° C. The softening point of this product is about 102° C., and the penetration at 32° F. is 14; at 77° F. is 20.5; and at 115° F. is 40.

B. A mixture prepared from approximately 174 parts by weight of coke oven tar and approximately 58 parts by weight of pulverized bituminous coal is heated to about 300° C. over a period of seven to eight hours. The heating is discontinued and about 72 parts by weight of heavy water gas tar heavy oil are added whereupon the heating is resumed at about 300° C. for an additional one to two hours. Then another 15 parts by weight of heavy water gas tar heavy oil are added whereafter the heating is discontinued and after thorough mixing the product is permitted to cool. The softening point of the product is about 89° C. and the penetration at 32° F. is 24; at 77° F. is 38; and at 115° F. is 67.

In either Examples A or B, the heavy water gas tar heavy oil may be substituted by light water gas tar heavy oil provided high melting coke oven pitch is included in the composition; though the heavy water gas tar heavy oil alone is preferred.

C. A still is charged with 310 parts by weight of crude light water gas tar and 40 parts of light water gas tar heavy oil. To this mixture 40 parts of molten high melting coke oven pitch having a melting point of 145° C. (cube in air), and 72 parts of pulverized coal are added. This mixture is heated to 300° C. in approximately one hour and maintained at about 300° C. for approximately three hours with agitation, 23 parts of distillate being removed. The residual pitch has a softening point of 88° C (ring and ball) and penetrations at 32° F. (200 grams, 60 seconds) of 20, at 77° F. (100 grams, 5 seconds) of 32 and at 115° F. (50 grams, 5 seconds) of 61.

D. A mixture of 39.5% by weight of residuum tar, 37.4% by weight of coal-tar heavy oil, and 23.1% by weight of pulverized bituminous coal is subjected to a digestion temperature of about 315° C. to obtain a coal-digestion pitch with an R. and B. softening point of 109° C., a penetration at 32° F. of 12, a penetration at 77° F. of 22, and a penetration at 115° F. of 50.

In some cases, one to two parts by weight of sulfur may be included in the coal-digestion mixture, or air may be introduced for varying periods. The air and sulfur serve as dehydrogenating agents by which with subsequent addition of heavy oil, the characteristics of the pitch may alternatively be adjusted.

The filters employed in the mastic may be sand, clay, slate dust and the like or mixtures thereof. The fibers may be mineral, vegetable or animal fibers or mixtures thereof including asbestos, cotton (such as that reclaimed from tires), hair and so forth. As previously indicated, it is preferable, particularly if the mastic is not subsequently dip-coated, to precoat the fibers. For this purpose there are employed tars such as coal tars including coke oven tar, horizontal retort tar, and vertical retort tar; heavy water gas tar and light water gas tar; oil-gas tar, Pintsch gas tar; wood tar; pitches of such tars; oils from such tars; asphalt; mixtures of these bituminous substances; or coal-digestion pitches either molten or dispersed in volatile solvent. If desired, any water repellent material may be used to precoat the fibers but it should preferably be miscible or compatible with the bituminous material in the mastic. For instance, a non-aromatic petroleum distillate should not be used to precoat the fibers, if a tar-derived composition is to be used in the mastic.

The precoating of the fibers is preferably accomplished in a Banbury mixer, though this is also done by dip-coating and then centrifuging to remove the excess coating material. In a Banbury mixer, about 15 parts by weight of coal-digestion pitch, such as exemplified above, satisfactorily coats about 85 parts by weight of fiber.

A rubber roll mill, pug mill, or a Banbury mixer may be used to prepare the mastic. The fillers and bituminous material used in the mastic are first mixed together and then the fibers, either precoated or untreated are added and mixed with the first two ingredients. When using a rubber roll mill, the bituminous material is melted on the heated rolls and then the filler and fibers are successively added and mixed. When using a pug mill, the melted bituminous material and filler are mixed in the mill, and the fibers are mixed with the resulting mixture in a pug mill, a rubber mill or in a Banbury mixer.

The proportions of constituents employed in the mastic may vary widely. Too much of the bituminous material of a low softening point should not be used if the mastic is to be pliable and yet retain its pressure-molded form. To avoid brittleness, the coal-digestion pitch should not be too high-melting or be present in too small proportions. In other words, pliability is increased while retaining the non-flowing properties of the mastic by increasing the amount of bituminous materials or fibers, or by using a bituminous material of lower softening point. Less pliable products are obtained by increasing the amount of filler, decreasing the amount of bituminous material, or by using bituminous material of higher softening point. For some purposes, up to twenty-five percent by weight of heavy oil, above mentioned, is advantageously added to the coal-digestion pitch prepared as described, for blending therewith.

More specifically, and by way of illustration, mastic sheets are provided which are obtained by use of bituminous constituents with softening points in the range of about 75° C. to about 150° C., though coal-digestion pitch with a softening point as low as 52° C. has been used to advantage. A pliable mastic sheet that is non-brittle at low atmospheric temperatures and non-flowing at highest atmospheric temperatures has been provided by use of coal-digestion pitch of above 90° C. softening point, and penetrations of not less than 10 at 32° F. and not more than 70 at 115° F. The stiffer mastic sheets have been provided which have been obtained by use of bituminous constituent with softening point of at least 75° C. and penetrations of 0 at 32° F., 0 to 5 at 77° F. and 15 to 35 at 115° F. More pliable mastic sheets than the latter have been obtained by the use of coal-digestion pitches with softening points of as low as 52° C. and up to about 75° C. to 80° C. and with penetrations of above 10 at 32° F., above substantially 60 at 77° F., and of substantially 230 to 240 and up to 300 at 115° F., all such sheets showing no tendency to flow at 140° F.

Specifically, and for purposes of illustration, as to the proportions of the constituents of the mastic, the bituminous constituent varies from about 25 percent by weight to about 60 percent by weight (generally about 30 to about 50); the fibers are present to the extent of at least about 8 or 10 percent by weight; and the filler up to about 65 percent by weight, but generally of the order of 40 to 55 percent.

Examples of mastics employed in various sheet products including those illustrated in the accompanying drawings are as follows:

Example 1.—140 parts by weight of coal-digestion pitch, such as that prepared in Example A above, are melted in a rubber mill. 90 parts by weight of dry clay are mixed with the molten coal digestion pitch, and finally 40 parts by weight of fibers of the type recovered from rubber automobile tires are incorporated. This forms a pliable mastic suitable for extruding or molding shingles.

Example 2.—A pliable mastic composition suitable for roll roofing or for strip shingles is made by mixing 52 parts by weight of coal-digestion pitch having a softening point of about 75° C. (ring and ball), with 40 parts by weight of dry clay, finely divided slate dust, or other filler, on a rubber mill, or in a pug mill, and then incorporating 8 parts by weight of fiber, previously coated with tar, pitch or heavy oil, on a rubber mill, or in a Banbury mixer.

Example 3.—A relatively stiff sheet mastic product suitable for use in pre-formed expansion joints, or insulation board, is prepared with the materials set forth in Example 2 but in proportions of 50 parts of coal-digestion pitch, 36 parts of filler and 14 parts of fiber.

Example 4.—Sheets of semi-rigid character for use in sidings or panels, for various purposes and formed by milling at moderate temperatures, are prepared from mastic compounded from 325 parts by weight of coal-digestion pitch of about 75° C. softening point (ring and ball), 575 parts by weight of slate dust, and 100 parts by weight of fiber precoated with 15% by weight (based on the weight of fiber) of coal-digestion pitch, coal tar pitch or other pitch, tar, or high-boiling tar distillate.

Example 5.—Pliable sheets are prepared from coal-digestion pitch, prepared in accordance with Example D above, by melting the pitch and mixing the molten pitch with clay and reclaimed tire fibers, in the proportion of 140 parts by weight of the pitch, 90 parts by weight of clay, and 40 parts by weight of fibers.

Example 6.—One part by weight of coal tar heavy oil from coal tar obtained in high temperature carbonization of coal, and with a boiling point range above substantially 300° C. is mixed with 3 parts by weight of coal-digestion pitch prepared as in Example D. To 52% by weight of this mixture, are added 40% by weight of slate dust and 8% by weight of reclaimed tire fibers, and mixing is completed on a rubber mill. A sheet is prepared from this mix which is strong and pliable.

Example 7.—To three parts by weight of coal-digestion pitch prepared as in Example D, is added one part by weight of coal tar heavy oil from coal tar obtained in high temperature carbonization of coal, and topped to substantially 300° C. To 50% by weight of this mix of pitch and oil, are added 36% by weight of slate dust and 14% by weight of reclaimed tire fibers. This mastic is rolled out into strong, pliable sheets.

Example 8.—57.5% by weight of slate dust, and 10.0% by weight of reclaimed tire fibers precoated with 1% coal-tar heavy oil are mixed on a rubber mill with 32.5% by weight of a mixture of three parts by weight of coal-digestion pitch prepared as in Example D, and one part by weight of coal tar heavy oil from coal tar obtained in high temperature carbonization of coal, and topped to substantially 300° C. The mix is rolled out into strong, pliable sheets.

By way of further examples, which may be designated as Examples 9, 10 and 11, in accordance with the above Examples 6, 7 and 8 respectively, strong pliable sheets are prepared from each of the mixes of constituents in the proportions stated in each of the latter examples but substituting for the coal-digestion pitch and heavy oil mixture, a mixture of 85 parts by weight of said coal-digestion pitch and 15 parts by weight of coal tar heavy oil from coal tar obtained in low temperature carbonization of coal, and topped to substantially 300° C.

Sheets are prepared in the aforegoing examples, which exhibit no tendency to flow at temperatures up to 140° F. and above, and therefore above highest recorded summer atmospheric temperatures, even in vertical position. Granules applied to a sheet as heretofore indicated, remain in their position as originally placed, because of absence of plastic flow in the material of the sheet surface.

For certain purposes, the mastic in sheet material is alternatively prepared from asphalt of about 100° C. (or above) softening point, or from coal tar pitch or other pitches of high softening points (120° to 140° C.) mixed with 30% to 40% heavy oil such as heavy water gas tar heavy oil or other oil of high aromaticity and with a boiling point above about 300° C. These bituminous substances are mixed with the desired proportions of filler and fiber (preferably precoated). A mastic sheet prepared from the resulting compounded material is preferably surface-treated as described below, with coal-digestion pitch, particularly of the type described in Examples A and D.

If a bituminous cement or dip-coat are employed to hold the granules on the mastic, as shown in Fig. 4, it is preferred that the bituminous base thereof be a coal-digestion pitch. Alternatively, pigmented bituminous paints or metal paints such as aluminum or bronze paints containing a bituminous vehicle may be applied for heat and light reflective purposes, as in Fig. 3.

The article to be dip-coated is submerged in the molten pitch having a consistency appropriate for forming a relatively thin coating on withdrawal. If the pitch has a higher melting point than the melting point of the bitumen in the mastic the time for submergence is less than that for coating with pitch of a lower melting point. The time may be long enough to permit some penetration which, if desired may also be obtained by applying pressure. The withdrawal of an article, particularly sheets or panels of large dimensions is preferably at a decelerating rate to obtain a uniform coating or a coating of uniform thickness throughout the area of a sheet. The total time of submergence of the lowermost portions of a sheet should not be such as to result in any substantially different change in physical or structural condition of the lowermost portion from that of the uppermost portion, as further indicated hereinbelow.

In preparing the above-mentioned bituminous cement in a preferred manner for cold application, a low-boiling solvent, such as coal tar naphtha, is added slowly at room temperature to molten coal-digestion pitch with agitation. The agitation is continued until all the solvent has been introduced, and the temperature of the final mixture has dropped to a substantial extent below the boiling point of the solvent.

Solvent naphtha in the above procedure may in whole or in part be replaced by other low-boiling coal tar solvents; fractions of heavy water gas tar and of light water gas tar distillates; and petroleum distillates of high aromaticity or containing a preponderating proportion of aromatics. The boiling point ranges of these solvents may be from about 100° C., or 135° C., or from 150° C. to about 200° C., depending upon the drying properties desired. The boiling points may also range higher than 200° C., as shown in the following table, depending upon the use of the cement:

Distillation of typical solvents

Sample Source	(1) Coal Tar	(2) Coal Tar	(3) Heavy W. G. Tar	(4) Light W. G. Tar
	° C.	° C.	° C.	° C.
First Drop	105	145	140	148
5%	127	160	153	175
10%	138	165	163	187
20%	148	169	173	201
30%	157	171	182	209
40%	164	174	191	218
50%	168	176	203	225
60%	173	177	207	228
70%	175	179	216	233
80%	179	182	225	241
90%	184	188	242	258
95%	195	198	258	280

The relative rate of evaporation of solvent and consequent setting-up time of the adhesive at atmospheric temperatures may be changed as desired by proper choice of an aromatic solvent or solvent fraction.

If relatively quick-drying adhesives are desired, the lower boiling solvents of any of the sources mentioned may be selected for use, but if slower setting cements are required the higher boiling fractions are selected for incorporation in the cement. In the above table, solvents (1) and (2) provide a relatively quick-drying cement; (3) provides a slightly slower drying cement; and (4) a considerably slower drying cement.

Coal tar solvent naphtha, when employed in the cement, is prepared from coal tar distillate from which most of the tar acids, and in some instances the tar bases, have been removed. Material boiling below 100° C. is preferably removed to prevent too rapid setting of the cement upon application, and to minimize fire hazards arising from too low boiling distillates if present. Coal tar solvents and solvents of high aromaticity are particularly compatible with coal-digestion pitches and prevent undesirable sludging and separation of different ingredients into layers.

The proportion of these solvents to be used in the cement depends upon the desired consistency of the product. A relatively viscous product, suitable preferably for warm weather or warm climate use, and better for daubing, rather than brush application, is made from a mix of approximately 80% by weight of coal-digestion pitch (softening point about 75° C. to about 125° C.), and 20% by weight of a selected solvent. A more fluid product, suitable for easy application in warm weather or warm climates is made with about 75% by weight of such coal-digestion pitch and 25% by weight of a selected solvent. A product fluid enough (specific Engler viscosity, 50 cc. at 50° C., of approximately 18) for convenient application at low to moderate temperatures is made with about 70% by weight of such coal-digestion pitch and 30% by weight of a selected solvent.

A very fluid cement for brush or spray application is made with 60 parts by weight of coal-digestion pitch (softening point about 75° C. to about 125° C.) and about 40 parts by weight of a selected solvent (boiling range 100° C. to 200° C.). The specific Engler viscosity of such a cement is approximately 5 (50 cc. at 50° C.).

A fluid cement generally suitable for brush or spray application to pitch coated articles, is made by cutting back 60 parts to 55 parts by weight of molten coal-digestion pitch of relatively high melting point with 40 to 45 parts by weight of aromatic solvent (boiling range about 100° to 200° C.), thus producing an adhesive, quick drying bituminous cement, that is applied cold, and having

an Engler specific viscosity of approximately 8 to 15 (50 cc. at 25° C.).

Fillers, such as, slate dust or flour, finely divided talc or clay may be added to the cement, if desired, to the extent of 25 to 30%, or even up to 35% to provide a desired weather-resistant adhesive.

The coal-digestion pitch in the cement is a coal-digestion pitch of the types specifically described above. If the pitch in a mastic surface has the composition of the coal-digestion pitch described in Example A above, it is preferred to use this pitch in the cement or dip coating since better bonding is obtained than in cases where the compositions are different. The same is true in the case of the coal-digestion pitches described in the other examples. Advantageous results are obtained however by using a coal-digestion pitch in the cement or as dip-coatings **6** (Fig. 4) prepared with constituents of any one of the tars or pitches mentioned and by using for the preparation of sheets a mastic prepared with constituents of any one of the other tars or pitches not used in the coal-digestion pitch selected for said coatings, but the physical characteristics (softening point, penetration etc.) of the coal-digestion pitches employed should preferably be substantially similar. For instance, in such combinations an effective bond is obtained by employing a heavy water gas tar-heavy water gas tar heavy oil-coal-digestion pitch in the mastic and a coal tar-heavy water gas tar heavy oil-coal-digestion pitch as the dip-coating or in the cement, or vice versa; or a light water gas tar-light water gas tar heavy oil-coal-digestion pitch in the mastic and a heavy water gas tar-heavy water gas tar heavy oil-coal-digestion pitch as the dip-coating material or in the cement, or vice versa; and so forth.

Though a dip-coat treatment alone provides for superior weather-resistance of a mastic article, it may be omitted as illustrated in Figs. 1 and 2 particularly if the fibers in the mastic have been precoated as indicated above.

Mastic compositions prepared from bituminous materials of inferior weather-resistance or having less desirable rheological properties than improved mastics prepared with coal-digestion pitch, may be considerably improved by surface treatment by hot-dipping sheets and articles such as shingles, panels and others, in molten batches of the above-exemplified coal-digestion pitches, or by application of compositions such as the above cements prepared with selected solvents. Only a thin coating of the above-described coal-digestion pitch, as at **8** in Fig. 7, is needed on the conventional asphaltic or tar or pitch or other bituminous mastic material **9**, to provide structural materials having superior weather-resistant and rheological properties over wide temperature ranges. These properties are improved by increase of depth of penetration of the coating material toward the interior. The coal-digestion pitch of the type set forth in Example A is of particular value for this purpose. The coal-digestion pitches prepared as in Examples B, C, and D are also advantageously so applied, with or without blended heavy oil in the proportions set forth.

By way of further detail, the hot-dip surface treatment includes raising a submerged sheet while in substantially vertical position in the body of the liquid coal-digestion pitch. While so raising the sheet in the pitch the rate of movement of the sheet in the bath is preferably uniformly decelerated to provide the uniform application of the pitch. Since the sheet may normally be subject to change of shape while submerged, the total time of submergence must not be such as to cause deformation or a change in the physical make-up of the sheet by contact with the hot bath.

This surface treatment may be applied to mastic sheets of the present invention as well as to conventional bituminous mastic sheets, so as to obtain a mastic sheet having a uniformly high concentration of coal-digestion pitch of improved characteristics in at least the service-exposed surfaces. While this serves to prevent surface-cracking in the sheet on sudden bending or from mechanical shock, particularly in the conventional bituminous mastic sheet, the treatment is not entirely necessary for sheets made from mastics of the aforegoing examples since the coal-digestion pitch is already present throughout the body of such sheets as in Figs. 1 and 5, for instance.

The improved mastic has many advantages particularly in constructional use as siding and roofing. Strength in a finished molded product is obtained by application of moderate heat and pressure upon the mastic to be shaped and with a minimization of metal consumption. The sheet, as such, can be attached to surfaces and applied in a manner well understood in the various arts. In adapting the sheet to particular uses, it can be included in plural-ply sheets or attached to webs or layers of other material to suit requirements. In the shaping of articles made of the mastic, molding temperatures and pressures are employed, for instance, respectively of about 75° C. to about 150° C. depending upon the melting point of the binder, and of from about 200 lbs. per sq. in. to about 2000 lbs. per sq. in. depending upon the thickness of a sheet and amount of compression required. Sticking of the mastic to molding faces is minimized by applying simply a thin film of a lubricating oil or grease preferably of petroleum origin to the mold surfaces, or by applying a light covering of finely divided mineral aggregate such as ground talc or slate dust or stone dust or the like, or by simultaneous use of both of these treatments.

The improved mastic herein described serves not only as heat insulation, but also as sound and electrical insulation. Its unique water-repelling properties are clearly shown by comparison with conventional mastics. After immersing the improved mastic in water for ten days at room temperature and also at 50° C., a change in weight of only 1% is noted; whereas other commercially known bituminous mastics submitted to similar tests show increases in weight of 15% to 25%.

The present application is a continuation-in-part of application Serial No. 508,856, filed November 3, 1943, now abandoned.

What is claimed is:

1. An article of manufacture consisting of an improved coherent, self-sustaining, prefabricated, flexible, bituminous mastic sheet that is non-brittle and non-flowing at atmospheric temperatures and that is composed of about 25 percent to about 60 percent by weight of coal-digestion pitch, at least about 8 percent by weight of discrete fiber particles and up to about 65 percent by weight of inert, finely-divided, granular filler, the said coal-digestion pitch in at least the service-exposed surfaces of the said sheet containing coal thermally digested and dispersed in heat-liquefiable bitumen and having blended therewith as an essential ingredient heavy hydrocarbon oil having a preponderating proportion of aromatics, and the said coal-digestion pitch having in combination a softening point above 90° C., a penetration of 32° F. of not less than 10 with 200 grams for 60 seconds, and a penetration at 115° F. of not more than 70 with 50 grams for 5 seconds, for preventing surface-cracking in said sheet on sudden bending and from mechanical shock.

2. An article of manufacture consisting of an improved, coherent, self-sustaining, pre-fabricated, flexible, bituminous mastic sheet that is non-brittle and non-flowing at atmospheric temperatures and that is composed of about 25 percent to about 60 percent by weight of coal-digestion pitch, at least about 8 percent by weight of discrete fiber particles and up to about 65 percent by weight of inert, finely divided, granular filler, the said pitch containing in at least the service-exposed surfaces of the sheet thermally digested coal in heat-liquefiable bitumen and blended with heavy water gas tar heavy oil, and said coal-digestion pitch having in combination a softening point above 90° C., a penetration at 32° F. of not less than 10 with 200 grams for 60 seconds, and a penetration at 115° F. of not more than 70 with 50 grams for 5 seconds, for preventing surface-cracking in said sheet on sudden bending and from mechanical shock.

3. An article of manufacture consisting of an improved, coherent, self-sustaining, pre-fabricated, flexible, bituminous mastic sheet that is non-brittle and non-flowing at atmospheric temperatures and that is composed of about 25 percent to about 60 percent by weight of coal-digestion pitch, at least about 8 percent by weight of discrete fiber particles and up to about 65 percent by weight of inert, finely divided, granular filler, the said pitch containing in at least the service-exposed surfaces of the sheet thermally digested coal in heat-liquefiable bitumen and blended with light water gas tar heavy oil, and said coal-digestion pitch having in combination a softening point above 90° C., a penetration at 32° F. of not less than 10 with 200 grams for 60 seconds, and a penetration at 115° F. of not more than 70 with 50 grams

for 5 seconds, for preventing surface-cracking in said sheet on sudden bending and from mechanical shock.

4. An article of manufacture consisting of an improved, coherent, self-sustaining, pre-fabricated, flexible, bituminous mastic sheet that is non-brittle and non-flowing at atmospheric temperatures and that is composed of about 25 percent to about 60 percent by weight of coal-digestion pitch, at least about 8 percent by weight of discrete fiber particles and up to about 65 percent by weight of inert, finely divided, granular filler, the said pitch containing in at least the service-exposed surfaces of the sheet thermally digested coal in heat-liquefiable bitumen and blended with coal tar heavy oil, and said coal-digestion pitch having in combination a softening point above 90° C., a penetration at 32° F. of not less than 10 with 200 grams for 60 seconds, and a penetration at 115° F. of not more than 70 with 50 grams for 5 seconds, for preventing surface-cracking in said sheet on sudden bending and from mechanical shock.

5. An article of manufacture consisting of an improved, coherent, self-sustaining, pre-fabricated, flexible, bituminous mastic sheet that is non-brittle and non-flowing at atmospheric temperatures and that is composed of about 25 percent to about 60 percent by weight of coal-digestion pitch, at least about 8 percent by weight of discrete fiber particles and up to about 65 percent by weight of inert, finely divided, granular filler, the said pitch containing in at least the service-exposed surfaces of the sheet thermally digested coal in heat-liquefiable bitumen and blended with an oil selected from a group consisting of heavy water gas tar heavy oil, light water gas tar heavy oil, and coal tar heavy oil, and said coal-digestion pitch having in combination a softening point above 90° C., a penetration at 32° F. of not less than 10 with 200 grams for 60 seconds, and a penetration at 115° F. of not more than 70 with 50 grams for 5 seconds, for preventing surface-cracking in said sheet on sudden bending and from mechanical shock.

6. An article of manufacture consisting of an improved, coherent, self-sustaining, pre-fabricated, flexible, bituminous mastic sheet having at least in its service-exposed surfaces a uniformly high concentration of coal-digestion pitch non-flowing in said surfaces in vertical position at highest atmospheric temperatures and for preventing surface-cracking in said sheet on sudden bending or from mechanical shock.

7. An article of manufacture consisting of an improved, coherent, self-sustaining, pre-fabricated, flexible, bituminous mastic sheet that is non-flowing at temperatures up to 140° F. and that is composed of about 25 percent to about 60 percent by weight of coal-digestion pitch, at least about 8 percent by weight of discrete fiber particles and up to about 65 percent by weight of inert, finely-divided, granular filler, the said coal-digestion pitch containing coal thermally digested and dispersed in heat-liquefiable bitumen and having blended therewith heavy hydrocarbon oil having a preponderating proportion of aromatics.

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