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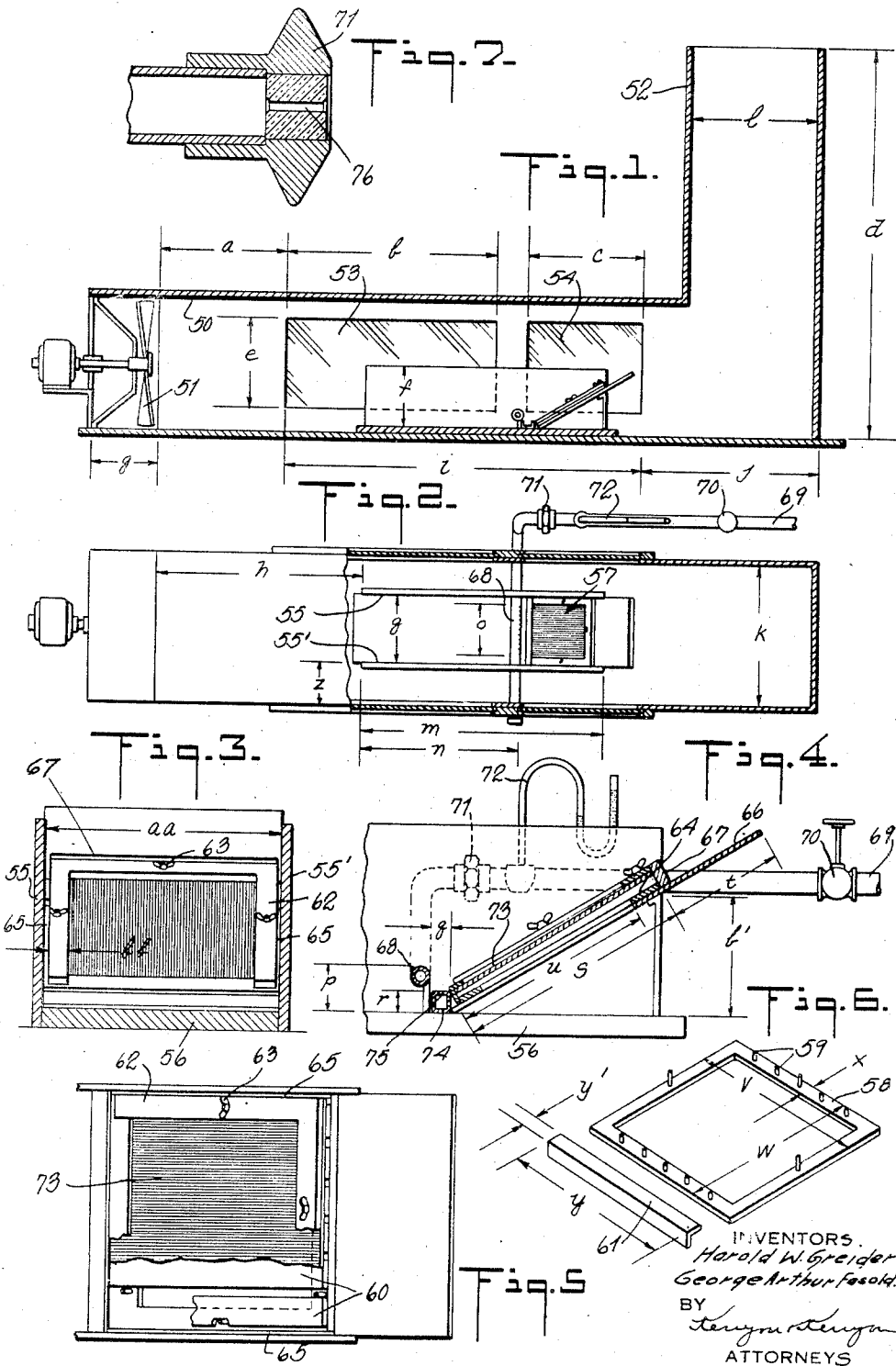
G. A. FASOLD ET AL

2,555,401

ROOFING AND THE MANUFACTURE THEREOF

Filed Dec. 28, 1944

4 Sheets-Sheet 1



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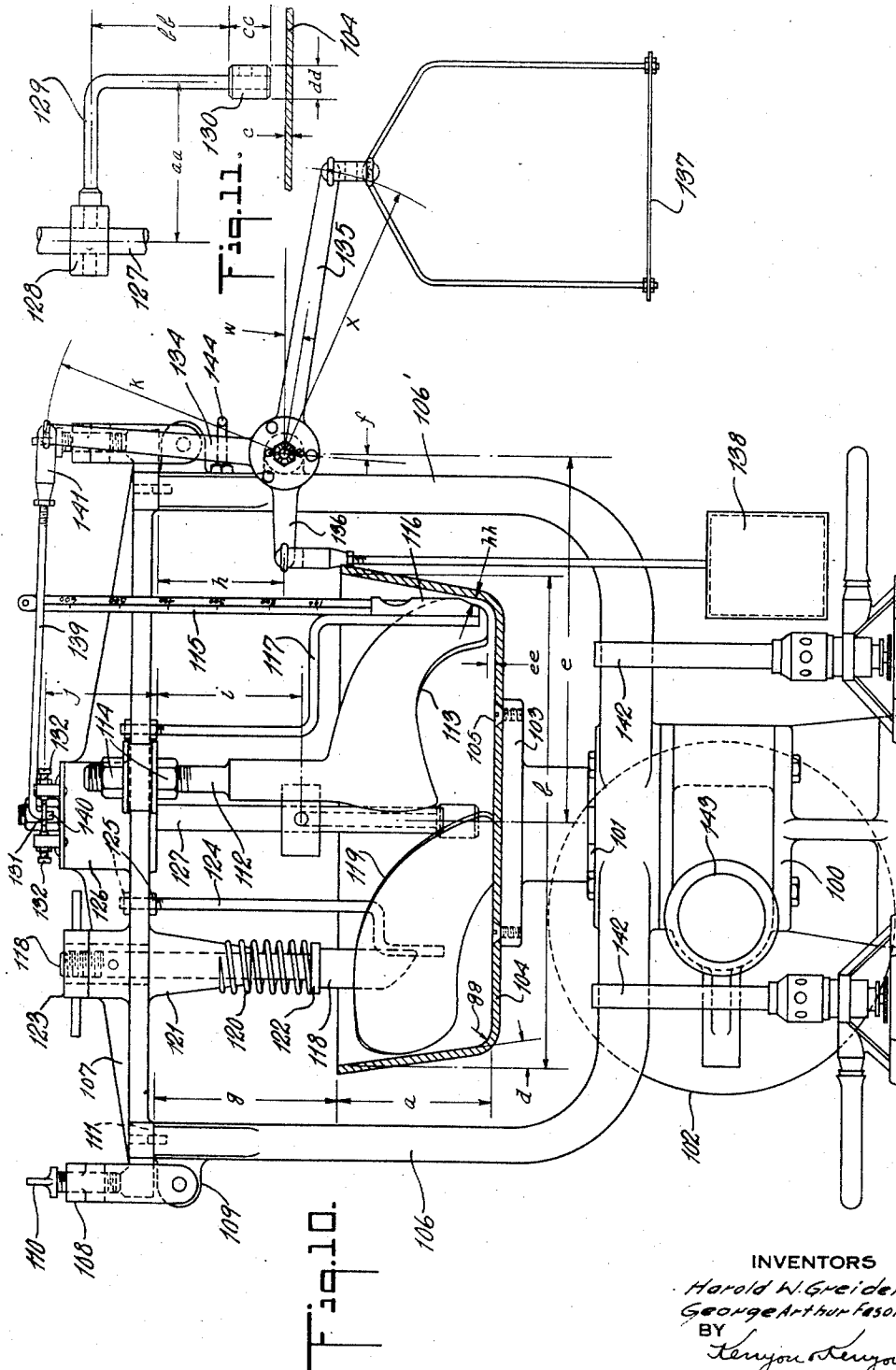
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ROOFING AND THE MANUFACTURE THEREOF

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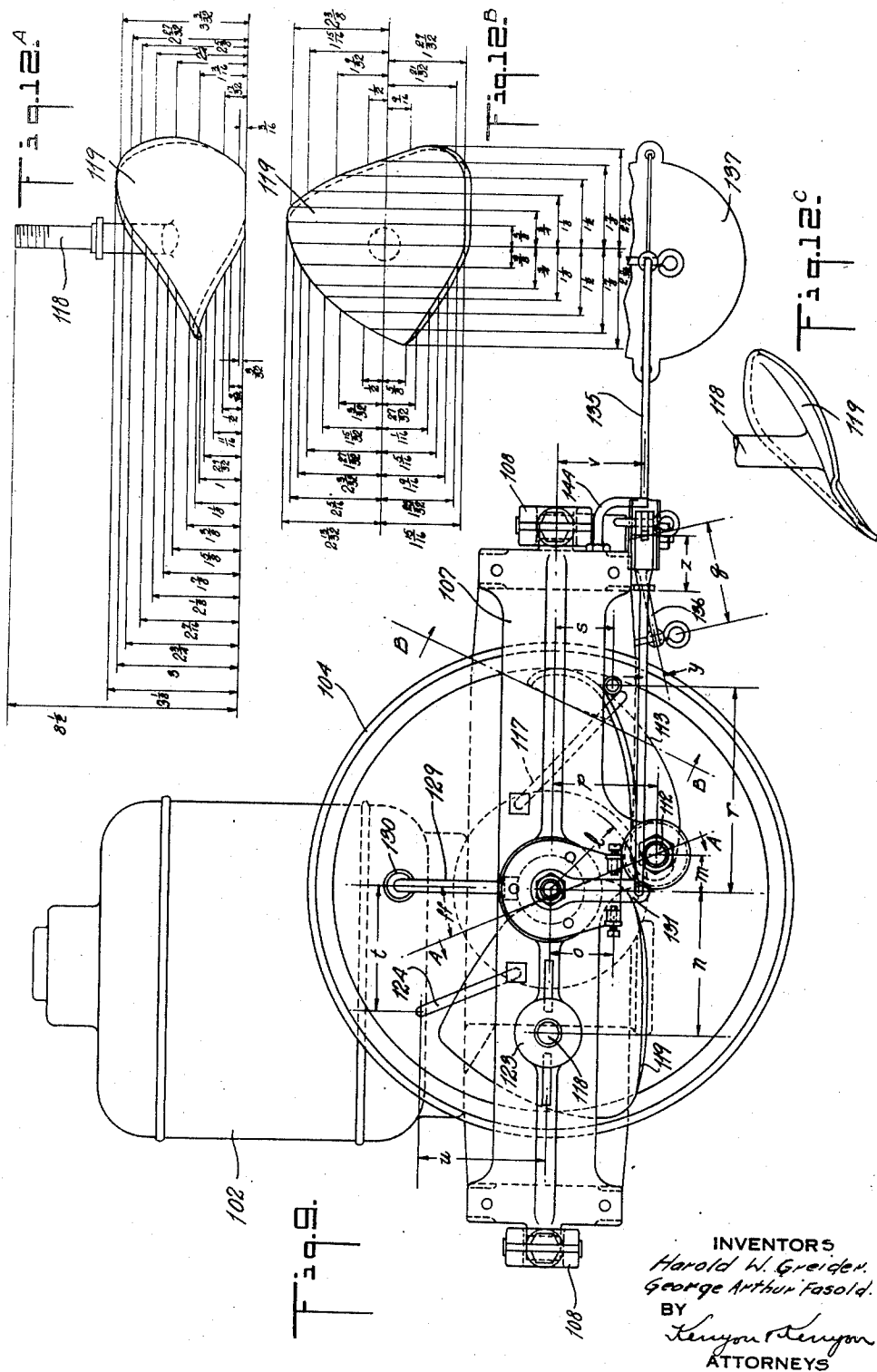
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ROOFING AND THE MANUFACTURE THEREOF

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4 Sheets-Sheet 4



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ROOFING AND THE MANUFACTURE
THEREOF

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This invention relates to roofing and the manufacture thereof. It relates particularly to roofing embodying bituminous material as a waterproofing.

The term "roofing" is used herein in the broad sense as referring to water and weather-resistant coverings such as shingles (individual or strip shingles), roofing tiles, cap sheets, sidings, roof deck coverings made from such preformed materials, built-up roofings and the like.

Heretofore bituminous prepared roofing has been very extensively manufactured comprising a fibrous base sheet impregnated with bituminous waterproofing material and a coating of weather-resistant bituminous composition on the weather exposed side of the base sheet. Usually there is applied to the bituminous coating on the weather exposed side of the roofing a suitable granular material such as slate granules, or a more finely-divided mineral surfacing such as mica or talc. A thin layer of bituminous waterproofing is usually applied to the back of the roofing and a finely-divided dusting material such as mica flakes, talc, silica dust or the like is made adherent to the backing coat as an anti-stick.

In addition to prepared roofing materials of the character aforesaid, built-up roofing has been fabricated by applying successive layers of "mopping" asphalt and fibrous sheet material to the surface of a roof-deck.

The fibrous sheet material that is employed in roofings of the character aforesaid is so-called "roofing felt." In the case of prepared roofings such as shingles, roll roofing, cap sheets and the like, the roofing felt is prepared by water laying organic fibers to form a felted sheet about .025 to about .075 inch in thickness, the fibers being from about $\frac{1}{16}$ to about $\frac{3}{16}$ inch in length. The fibers that are used are generally composed of a mixture of fibers, the bulk being vegetable fibers such as liberated wood fibers, fiberized rags, and the like although some animal fiber such as wool is generally present in small proportion. Such short fibers are relatively inexpensive as compared with the grades of fiber having longer fiber length. Moreover, in ordinary water suspension, longer fibers tend to form into objectionable clumps or clots. It is for these reasons that "roofing felt" has remained a standardized product that has not been modified substantially, at least as far as commercial production of roofing is concerned, for many years. In the construction of built-up roofing, asbestos felt is quite extensively used, although ordinary organic felt is also used to a large extent in the fabrication of built-up roofing. In the manufacture of asbestos felt, the

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asbestos fibers used are of the paper making grade of the order of length that has been mentioned above. Roofing felt made from organic fibers normally does not contain any binder, since the felt has sufficient strength to be passed through the roofing machine merely by virtue of the interfelting of the fibers and the partially hydrated condition of the fibers at the time of water laying. In the case of asbestos felt, a small amount of starch, e. g. about 5% to 10% on the weight of the asbestos fiber, is generally used to give the asbestos paper sufficient strength so that it may be handled.

It is an object of this invention to improve upon the fibrous sheet material used in the manufacture of roofing. It is a further object of this invention to improve upon the water-resistance and durability of roofings. It is a further object of this invention to improve upon the resistance of roofings to the defects of sliding and of blistering. It is a further object of preferred embodiments of this invention to improve upon the fire resistance of bituminous roofings.

It is a feature of this invention that instead of employing conventional "roofing felt" as the foundation or base sheet of a roofing product, which conventional "roofing felt" is composed of short fibers felted into a dense sheet, long fibers are used which in major proportion by weight are at least about one half inch in length and which are disposed in essentially individual fiber form in an open textured arrangement that is highly porous and highly permeable as compared with ordinary roofing felt.

It is a further feature of this invention to employ an open textured fibrous base sheet of the character aforesaid that is thinner and lighter in weight than the conventional roofing felt used in the manufacture of bituminous roofing so that the weather resistant bituminous coating constitutes the bulk of the roofing. At the same time it is a feature of this invention to employ a base sheet that is much more highly saturated relative to the total weight of fiber than has been prior practice.

It is a further feature of the invention to employ in the base sheet relatively long mineral fibers such as glass fibers although long organic fibers such as cotton may be employed.

It is a further feature of the invention to provide a fibrous base sheet that is impregnated with a bituminous waterproofing that is of a higher softening point than has been used according to prior practice and to provide a roofing that is more uniform throughout with regard to the bituminous material contained therein and that has greater stability and weather resistance.

It is a further feature of this invention that the bituminous coating layer is more uniformly keyed into and made integral with the foundation or base sheet, thereby rendering the roof more resistant to weathering, to sliding and to fire.

It is a further feature of this invention to impregnate an open textured base sheet with a bituminous impregnating material comprising filler and to introduce the filler into the body portion of the base sheet preferably so that more of the filler occurs adjacent the surface of the base sheet that carries the waterproofing coating layer than occurs in the mid portion of the base sheet.

It is a further feature of preferred embodiments of this invention that bituminous coating composition of special formulation having high flow resistance characteristics under flame temperature is employed in combination with the special long fiber open textured base sheet so as to improve the fire resistance properties of the roofing. It is a further feature of this invention that such special bituminous composition is introduced into the open textured base sheet as an impregnant therefor.

As mentioned above, the base sheet of the roofing is composed of long fibers, a major proportion of which are over $\frac{1}{2}$ inch in length, the individual fibers being disposed in an open textured relation as by very loose felting. A wide variety of fibers may be used including vegetable, animal or mineral fibers. Mineral fibers are especially desirable due to the fact that they are not affected by moisture and do not absorb moisture and likewise are noncombustible. One type of mineral fiber which is especially suitable, due to the fact that it can be produced in the form of long filaments at relatively low cost, is glass fiber.

Glass fiber can be produced by known methods such as by blowing molten glass into elongated filamentary form. Such fibers are best handled by dry felting as by deposition from air to form a fiber web or "felt" of any desired degree of openness or porosity. The felt, especially when disposed in a highly porous and open condition, has very little strength and for this reason it is desirable to apply some suitable binder which merely acts to bind the fibers together at their intersections without filling the pores in the felt. A suitable binder for this purpose is starch or a suitable resinous binder such as a phenol-aldehyde or a melamine type of resin. The felt preferably is of the minimum thickness that is consistent with affording sufficient strength to permit the felt to be passed through a roofing machine for impregnation with bituminous waterproofing and for application of the bituminous coating layer and mineral granules thereto. A tensile strength of the order of 10 pounds per linear inch of width is usually sufficient for the purpose and a tensile strength of the order of 20 pounds per linear inch of width is preferable. When glass fibers are employed, the thickness of the felt may be of the order 10 to 30 thousandths of an inch according to preferred practice of the invention.

The tensile strength values above mentioned are as determined by the method prescribed in A. S. T. M. Standard D 202-41 T using a Scott tensile testing machine, the test specimens having been conditioned at 45% relative humidity and 77° F. for four hours before testing. In the finished roofing, the base sheet or web, after having been impregnated with a bituminous im-

pregnating material, should provide substantial strength and dimensional stability, and it is desirable that the impregnated web should have a tensile strength of at least 10 pounds per linear inch of width and preferably at least 20 pounds per linear inch of width and that the impregnated web should exhibit not more than about 5% elongation at break when tested for tensile strength at 77° F.

As mentioned above, the long fibers are laid in an open textured relation. The openness or porosity of such a fiber web can be measured by determining the resistance to the passage of air therethrough under controlled conditions. We have used for making such determination, a Gurley Densometer, manufactured by W. & L. E. Gurley, Troy, New York. This densometer testing device is described in A. S. T. M. Standards, 1943 supplement, at page 220, under the designation D 726-43 T. The test consists in clamping a specimen of the material to be tested between two plates having registering openings therein of a given accurately dimensioned circular area, forcing a known volume of the air through the aperture under a given pressure and noting the time for the known volume of air to be forced through the sample. In the Gurley Densometer a cylinder is used which is vertically disposed and vertically movable by gravity and which is of known weight, the cylinder being sealed with a liquid seal and being calibrated on its side in cubic centimeters. The cylinder is raised prior to the determination, and, after the sample has been placed in position, the cylinder is released. A stop watch is started when the cylinder passes the zero calibration thereon during its descent by gravity and is stopped when the cylinder passes some other calibration such as the 100 cc., 200 cc. or 300 cc. calibration. For samples of such high density and low air permeability that the cylinder moves very slowly, a smaller volume calibration is generally employed, while if the sample is of relatively low density and high air permeability usually the 300 cc. volume calibration is employed. When the sample is of relatively low porosity, the aperture through which the air is forced is normally one square inch in area and the results of the test are expressed as the time in seconds for 100 cc. of air to be forced through the sample. When the sample is of lower density, use of a smaller diameter opening is desirable and openings of $\frac{1}{4}$ square inch and $\frac{1}{16}$ square inch are supplied by W. & L. E. Gurley as optional equipment. In making the tests referred to herein, the opening used was the $\frac{1}{16}$ inch opening due to the fact that the fibrous felts which are employed according to this invention are of very low density and in this respect are in an entirely different category as compared with ordinary roofing felts. In making the test, a standard cylinder weighing five ounces was employed. When references are made herein or in the claims to the "fiber web Densometer value" as expressed in seconds, this value is measured and determined by the above-described test for passage of 100 cubic centimeters of air using the $\frac{1}{16}$ square inch opening. A low "fiber web Densometer value" is indicative of a felt of low density, and of high porosity, while a high "fiber web Densometer value" is indicative of a felt of high density and of low porosity.

A glass felt of the character above described ordinarily has a fiber web Densometer value of only about .5 to 2.0 seconds. Such a material is in an entirely different category from ordinary roofing felt which has a fiber density ranging

from about 100 to about 400 seconds. The high density of ordinary roofing felt is due to the fact that the short fibers tend to become intimately associated and compacted during water laying. The thickness of ordinary roofing felt has very little effect on the fiber density value and the fiber web Densometer value for ordinary roofing felt which is about .050 inch in thickness is substantially the same as the fiber density value of roofing felt made from the same stock which is .030 inch in thickness.

The practice of this invention may be illustrated in connection with a preformed roofing material such as shingles (either single shingle units or strip shingles) wherein the bituminous coating layer for the felt is of the conventional type that has been used in the industry for many years. Glass fiber felt about .020 inch in thickness and having a fiber web Densometer value of about .5 second is used. The glass fiber may be initially bonded with a small amount of a resinous binder so as to give the felt sufficient strength so that it can be passed through the roofing machine.

The felt is first saturated with a bituminous saturant having a softening point of about 190° F. The saturation occurs very readily due to the extreme openness and porosity of the felt, and the felt will take up a very high proportion of the saturant. Thus the felt may become saturated to about 450% of the weight of the fiber. After the felt has been saturated, a layer of bituminous coating composition is deposited thereon to form a layer about .080 inch in thickness. The bituminous coating composition may consist of regular roofing coating asphalt having a softening point of about 205° F., which ordinarily contains a conventional filler such as about 20% to 35% by weight of the coating composition of limestone dust or slate flour. Conventional granules such as slate granules are partially embedded in the outer surface. A layer of back coating about .005 inch thick is applied to the back of the felt and an anti-stick such as mica flake is partially embedded in the back coating. The finished material after cooling is then cut into shingles.

The advantages which result from the practice of this invention as described above are several. By utilizing a felt of long fibers, the felt can be made considerably thinner than is the case with ordinary roofing felt. The short fibers of which ordinary roofing felt is composed have to be built up to substantial thickness in order to afford a base having adequate strength of the manufacture of a roofing product. When the thickness of the felt is reduced by employment of open textured long fiber felt according to this invention, we have found that the suitability of the product for roofing is greatly improved. The effective weather resistance of a roofing is afforded by the bituminous coating layer and not by the bituminized felt base; thus, by increasing the proportion of the bituminous coating layer to the felt, a roofing product having longer weathering life is afforded. Moreover, the entrapment of voids within the roofing induces the defect of blistering and, by utilizing a thin open textured felt which is readily filled with asphalt, the tendency to entrapment of voids is minimized. The open textured character of the felt also permits much higher saturation of the felt. The roofing felt which is used in conventional roofing is saturated to the extent of about 175% on the weight of the fibers, and the upper limit of saturation is about 225%. As pointed out above, much

higher saturation of the base felt is possible according to this invention, namely, saturation of the order of 300% to 600% and preferably greater than 400%. The felt base sheet is thus seen to be largely asphaltic and in effect constitutes a second layer of asphalt contained in a skeletal reinforcement of fiber. The result is that the base sheet in itself constitutes a second weather resistant membrane. In ordinary roofing, the felt goes to pieces rapidly upon exposure to weather, unless it is protected by a weather resistant coating layer on the weather exposed side and unless it is further protected by a water resistant coating layer on the non-weather-exposed side.

Due to the openness and very low density of the felt base sheet that is employed according to this invention, the saturant for the base sheet can be of considerably higher softening point than the saturant used for saturating ordinary roofing felt. In saturating ordinary roofing felt, the felt is passed repeatedly through a bath of the heat liquefied asphalt. The asphalt has to be heated until it is liquid, and in order to produce sufficient fluidity to permit penetration of the asphalt into the minute voids of the felt without burning the felt, the softening point of the saturating asphalt that has been used in the roofing industry has been of the range 115° F. to 165° F. When an open textured long fiber felt is used in the practice of this invention, the impregnation of the felt is much more rapid and saturating asphalts of higher softening point than heretofore employed may be used. In fact, an asphalt may be used which has substantially the same softening point as the asphalt used in the bituminous coating layer. We have found that one of the reasons for failure of bituminous roofing resides in a lack of compatibility of the asphalt used for impregnating the felt with the asphalt used in the coating layer, this lack of compatibility being due to the wide difference in softening point of the asphalts used. By employment of the open textured long fiber felt in the practice of this invention, this source of difficulty can be minimized or eliminated by employment of asphalt saturant in the felt which more closely resembles or is identical with the asphalt used in the coating layer. In the ordinary practice of this invention, the softening point of the bituminous saturant used for impregnating the felt is between 170° F. to 220° F. and it is better that the softening point of the impregnant be above about 180° F. It is preferable in preferred practice of this invention that the softening point of the bitumen used in saturating the felt differ by less than 30° F. from the softening point of the bitumen that is used in the bituminous coating composition. By employing a saturant that more closely approaches the softening point of the bitumen in the bituminous coating layer, a much stronger bond is afforded between the felt base and the coating layer, which fact serves to prevent blistering and other types of failure in the region of the union of the coating with the felt. This advantage is of particular value at summer sun roof surface temperatures, which in some localities are as high as 170° F. Moreover, by employment of an impregnating material of relatively high softening point of the order mentioned, we have found that the base sheet is made considerably more weather resistant due to the fact that the impregnating material has greater stability at summer sun temperatures and serves more effectively to seal the edges of the roofing and to

prevent erosion of bitumen and fibers from exposed edges of the felt. Moreover, the tendency for moisture and air to penetrate the base sheet and cause oxidation and embrittlement of the bitumen and also cause rotting of any organic fiber contained therein is greatly reduced. The bitumen employed in the bituminous coating generally has a softening point of the order of 200° F. to 240° F. However, other types of bitumen, particularly those which have been stabilized so as to be flow resistant may be employed.

When reference is made herein to "bitumen," the reference is to asphalt or other bitumen per se, while reference to a "bituminous" composition, or material, or coating, or layer is a reference to the composition, material, coating or layer in its entirety which may consist of bitumen per se or may comprise bitumen mixed with some filler material. This distinction is made since the addition of a filler to a bitumen usually tends to increase the softening point of the bitumen.

The fact that the open textured long fiber web can be very rapidly and completely impregnated is also of advantage in the commercial production of roofing, since the impregnation step can be accomplished more rapidly and using less cumbersome and costly equipment. In fact, the impregnation of the felt can be accomplished by a simple roll coater or by merely spreading the bituminous impregnating material on the surface of the open textured sheet material, the spreading action alone causing the impregnating material to penetrate throughout the thickness of the sheet material. Moreover, when the bituminous impregnating material is to be the same in composition as the coating material, the impregnation of the open textured sheet and the spreading of a coating layer on the surface thereof to desired thickness can be accomplished in a single operation.

The fact that the base sheet is composed of long fibers and is of open texture so as to be of very low density is likewise of advantage in that the coating layer of bituminous composition becomes more effectively anchored to the base sheet. When ordinary roofing felt is used it can only be saturated with asphalt of a relatively low softening point above mentioned and the body of the felt is necessarily quite thick and is of the dense character above mentioned. The result is that the bituminous layer of coating composition merely rests upon the surface of the felt. In warm weather the low softening point saturant tends to bleed to the surface of the felt and in effect forms a layer of lubricant which may induce sliding of the bituminous coating relatively to the felt. Summer sun roof surface temperatures may be as high as 170° F. and, since this temperature is above the softening point of the saturant used to impregnate ordinary roofing felt, it is apparent that the saturant becomes quite fluid and offers little resistance to sliding of the coating layer. When an open textured web of long fibers is employed, the surface is quite irregular. Moreover, even when this type of felt is saturated to the extent of 400% to 500% on the weight of the fibers, the surface interstices in the felt remain unfilled and the felt is still far from completely saturated so as to fill all of the voids. The result is that the bituminous coating layer becomes keyed into the felt and is much more resistant to sliding. Moreover, the felt being thinner and incompletely saturated with a high softening point bitumen, there is less tendency for the saturant to bleed to the surface. The

fact that a saturant having a higher softening point than is ordinarily employed may be used to impregnate the open textured long fiber felt minimizes the susceptibility of the saturant to excessive softening in warm weather and further counteracts the tendency to slide of the coating layer of the roofing.

The improved anchoring of the weatherproofing coating that results from the employment of the open textured long fiber base sheet is likewise of advantage in improving the fire resistive properties of bituminous roofings due to the fact, when the weatherproofing bituminous coating layer is applied to the open textured long fiber base sheet, its tendency to run and flow when exposed to flame temperature is much less than when the weatherproofing coating is applied to ordinary roofing felt. Conventional weatherproofing coating composition as applied to ordinary roofing felt tends to run and flow and to burn freely when exposed to flame temperature, whether the coating composition is composed essentially of bitumen or contains a considerable quantity of a conventional filler such as 20% to 35% by weight of limestone dust or slate flour. However, by employing an open textured long fiber base sheet according to this invention, the bituminous coating has a greatly reduced tendency to flow when exposed to flame temperature, and when the bituminous material of the weatherproofing coating layer contains at least 30% and preferably at least 40% by weight of a finely divided solid water insoluble filler material that has a softening point above 500° F., a marked improvement in fire resistance is obtained. Hereinbelow we have described certain special fire resistive coating compositions which afford roofing having extremely high fire resistive properties due to the very high flow resistance of the filler contained therein, and due to the greatly reduced flammability of the bituminous coating material. However, even in the case of bituminous coating compositions which exhibit little or no improvement in fire resistance when applied to ordinary roofing felts, a very considerable improvement in fire resistance is obtainable according to the present invention by the employment of an open textured long fiber base sheet. As in the case of the special fire resistive coating composition described below, it is desirable, however, that the filler contain heat resistant mineral filler, the ratio of the per cent. by weight of which to the per cent. by weight of bitumen in the bituminous coating material be at least 1 to 3 and preferably at least 1 to 2. Ordinarily, all of the filler consists of heat resistant mineral filler. A more full exemplification of suitable fillers is given hereinbelow.

In addition to glass fibers, other mineral fibers may be employed such as rock wool or slag wool when the molten mineral is blown out or otherwise produced in filamentary form, rock wool and slag wool being essentially glass-like and resembling glass fibers quite closely. Asbestiform mineral fibers may likewise be employed provided the fibers are long fibers, at least 50% by weight of them being at least ½ inch in length. Such asbestos fibers are to be contrasted with the ordinary asbestos fibers used in the manufacture of asbestos felt for roofing and which are of the order of 1/16 to 1/4 inch in length and produce a dense felt. The fiber web Densometer value of ordinary asbestos felt used for roofing is about 1000 to about 2000 seconds. The longer grades of asbestos fibers are considerably more

expensive than the shorter grades, but this expense factor is largely counteracted by the fact that in the manufacture of an open textured long fiber felt from asbestiform mineral fiber, the amount of fiber that is required is much less than is required to produce a felt of corresponding strength from the grades of asbestos used in the manufacture of ordinary asbestos roofing felt.

In addition to the mineral fibers which are preferred, organic fibers either natural or synthetic, which are of the length above mentioned can be employed, such as cotton, rayon, cellulose acetate, wool, jute, flax (linen), ramie, defibrated wood (particularly the kind produced by the well-known Asplund process). Those partially opened threads which occur in fiberized rags and the like are long and flexible and are to be regarded as fibers or filaments in the practice of this invention. The fiber ordinarily should be one that is flexible, those fibers, including mineral fibers, that are of the flexibility of ordinary commercial grades of jute, or are more flexible, being regarded as flexible fibers. Such fibers are not capable of being felted by ordinary wet felting operations due to the fact that they tend to form into clumps or clots. However, this difficulty can be obviated by employing as the liquid medium from which the fibers are felted, a liquid medium having considerably higher viscosity than water, the viscosity of the medium being of the order of at least about 100 centipoises and preferably considerably higher, e. g. of the order of 1000 centipoises. By employment of a viscosity-increasing agent such as bentonite clay, soluble silicates, glycerine, casein, pectins and pectates, starch, or the like, aqueous media of high viscosity can be afforded. If desired, non-aqueous media of high viscosity may be employed such as glycerine, ethylene glycol, mineral oils, or the like. The employment of such high viscosity media is not only of utility in wet felting long flexible organic fibers, but also is of utility in wet-felting long flexible mineral fibers.

In addition to the wet felting of the long fibers, the fibers may be individually disposed in felted arrangement in other ways. Thus those long fibers which can be handled in a carding machine or a garnetting machine may be produced in open textured sheet form in this way, such operations being regarded as dry felting of the fibers. Other methods of dry felting the long fibers may also be employed as by felting from a suspension in an air stream.

When long flexible organic fibers are employed in the production of an open textured web of low fiber web Densometer value, the fact that such web material can be much more highly saturated with a bituminous saturant than ordinary roofing felt is of especial advantage in the case of organic fibers, since the fibers are more completely waterproofed and protected from the weather, and a roofing product of longer weathering life is afforded.

When the flexible long fibers are disposed in an open textured felt, the resulting web generally has insufficient strength to be successfully run through a roofing machine. Therefore, in the practice of this invention the felt generally has a binder applied thereto. The only requirement for the binder is that it not be such that it is rendered so ineffective by contact with the heat-liquefied asphalt used in roofing that the felted fiber web will break in the roofing machine. The binder therefore should be a "heat resistant" binder, namely, should not become excessively

weakened because of melting or decomposition during impregnation of the felt with the bituminous impregnating material. There are a large number of binders that may be employed such as starch, water soluble gums, rubber latex, synthetic resin solutions or emulsions (melamine-aldehyde, phenol aldehyde, urea-formaldehyde, polyvinyl acetate, etc.), synthetic rubber emulsions, viscose, cellulose esters or ethers, and the like. In addition, the binder may be developed from the fiber substance itself. Thus, in the case of organic fibers, the surfaces of the fibers may be peptized with a substance such as sulphuric acid, phosphoric acid, zinc chloride or the like, the peptized fiber surface being thereafter quenched with water, and hardened by drying.

Even the gelatinization of the surface of cellulosic fibers as a result of sufficient heating in water suspension will provide a binder that will bond the fibers together when the fibers are felted and dried in felted sheet form. Alternatively, fibers that are peptized by heat or by means of a solvent, e. g. cellulose esters, rayon, and the like, may be employed, the felt being bonded by the application of heat or a brief application of a solvent to peptize the fibers and by permitting the peptized fiber substance to harden. Any such binder, whether added to the felt, or produced from the substance of the fibers themselves, is to be regarded herein as a binder which bonds the individual fibers together at the points of contact of the fibers in the felt.

As mentioned above, we prefer to employ long flexible mineral fibers as the base sheet for our new roofing, since such fibers are not adversely affected by water (do not deteriorate or swell) and since such fibers are non-inflammable and afford roofings of higher fire resistance. While it is preferable to employ a base sheet consisting substantially entirely of mineral fibers, many of the advantages which result from the employment of mineral fibers can be attained when the major proportion by weight of the fibers in the open textured long fiber felt consists of mineral fibers, although preferably at least 65% by weight of the fibers consists of mineral fiber. From the point of view of improving the fire resistance of the roofing, it is of advantage in the practice of this invention to treat any organic fibers with a combustion retarding material such as chlorinated paraffin having a high chlorine content, chlorinated diphenyl, polyvinyl chloride, and polychloroprene, which has the effect of preventing the organic fibers from giving off combustible gases and vapors when highly heated. Any fiber which does not burn with a flame when exposed to a temperature of about 900° F. is to be regarded as a fire resistant fiber and the employment of felt consisting in major proportion of fire resistant fiber, and preferably consisting substantially entirely of fire resistant fiber, is desirable in the practice of this invention.

Regardless of the kind of fiber that is employed, the felted sheet, which contains the binder, should be such that the fiber web Densometer value is not greater than about 50 and preferably not greater than about 10. The thickness of the felt is preferably low, namely, in preferred practice, of the order of 0.01 to 0.03 inch. However, it is not without the scope of this invention to employ the open textured long flexible fiber felt in any thickness, e. g. from about 0.005 to 0.1 inch. As mentioned above, variation in the thickness of the felt produces very little change in fiber density value and the fiber density value will

remain low provided the felt is loosely arranged and does not contain an undue amount of fine material or filler that tends to fill the interstices and render the felt of low porosity. In the roofing product, it is desirable that the felt constitute the minor proportion of the total thickness of the felt plus the coating and preferably constitutes not more than about 30% of the total thickness of the felt plus the coating.

While the fibers in the base sheet ordinarily are felted either dry or wet, any other disposition of fibers or filaments in essentially individual condition in a web or sheet that has the open texture and low fiber web Densometer value referred to may also be employed. By stating that the fibers or filaments are arranged in essentially individual condition, the discrete and individual condition that occurs in felt is intended as distinguished from woven fabrics and from the use of cords or the like which are unsuitable due to the fact that the spun condition of the fibers in the threads or cords reduces the over-all absorptiveness and porosity of the sheet material and creates discontinuities in the sheet material due to the fact that the spun strands do not become properly saturated with the asphalt and the asphalt tends to concentrate at the interstices in the fabric. However, as pointed out above, soft flexible partially fiberized threads such as occur in fiberized rags are regarded as fibers or filaments and may be comprised in the open textured long flexible fiber sheet of low fiber web Densometer value that is employed in the practice of this invention.

The employment of the open textured long flexible fiber felt is not only of advantage in the manufacture of shingles, but also is of advantage in the manufacture of other types of roofing such as roll roofing, cap sheets, etc., and the advantages above mentioned are likewise incident to these types of roofing. The open textured long flexible fiber felt is likewise of advantage in the construction of built-up roofings. In the construction of built-up roofings, considerable trouble has been encountered due to entrapment of air pockets underneath the felt as it is laid in place on mopping asphalt. When a long fiber felt of high porosity and low fiber web Densometer value is employed in the practice of this invention, this difficulty is eliminated, since the air passes freely through the porous felt. Moreover, the special felt provides a better bond between the plies and permits the fabrication of a more homogeneous roof covering wherein the bond between the plies is improved and wherein the bulk of the felt contained in the built-up roof covering can be greatly reduced.

As mentioned above, we have found that the fire resistive properties of bituminous roofings can be very greatly increased by special formulation of the weatherproofing bituminous coating. When the bituminous coating composition of the weatherproofing coating layer is formulated within narrow limits imposed by the minimum flow resistance coefficient of the composition under flame exposure, on the one hand, and the maximum Wagner-Bowen plasticity value at 400° F., on the other hand, roofings having extremely high fire resistive properties can be obtained. The flow resistance coefficient and the Wagner-Bowen plasticity value at 400° F. are measurable properties which are fully defined hereinbelow and which are analogous in many respects to properties such as softening point, viscosity, boiling point, etc., that are commonly resorted to

for the purpose of defining particular bituminous or other compositions wherein such properties are critical.

We have found that, where conventional filler materials such as slate flour or limestone dust are incorporated in a bituminous composition, even in very considerable quantities, of the order of 40% to 50% by weight of the bituminous composition, such filler material has very little effect upon the tendency of the bituminous composition, when applied to ordinary roofing felt as the weather resistant coating layer, to burn and at the same time to freely run and flow when the roofing comprising the coating is exposed to flame temperature either in the case of an actual conflagration or in the case of the fire resistive tests which are prescribed by Underwriters Laboratories, Inc., of Chicago, Illinois, for testing the fire resistive properties of roofings. However, there is a critical point at which further addition of only a few per cent, of a particular filler effects a very great increase in flow resistance of the composition and has the effect of providing in the composition a stable skeletal mat that is highly resistant to running and flowing when the bituminous composition disposed as a coating layer is exposed to flame temperature. When such critical condition is attained, the composition, upon exposure to flame temperature remains in place and the bitumen in the composition, instead of inducing combustion, tends to carbonize to provide a cementitious bonding material that binds the skeletal mat of filler to form a coherent, mat-like mass that is highly resistant to combustion and that provides a very effective layer of thermal insulation material and protects an underlying roof deck from the heat of the flame. Due to the special properties of the open textured long fiber base sheet, such base sheet contributes to the fire resistance of the roofing product by virtue of its specific effect in tending to reduce flow of bituminous coating material when exposed to flame temperature; and the special fire resistant coating composition which is employed according to preferred practice of this invention is described and defined herein as being appropriate for use in combination with an open textured long fiber base sheet for the purpose of providing a roofing having very high fire resistance.

The critical point at which the flow resistance coefficient becomes greatly increased is different for different fillers and is a specific property of the filler. Thus in the case of limestone flour (about 85% by weight of which passes a 200 mesh testing sieve), the critical point is in the neighborhood of 75 to 80% by weight of the bituminous coating composition. For slate flour (about 85% by weight of which passes a 200 mesh testing sieve), the critical point is in the neighborhood of 65% by weight of the bituminous coating composition. The critical amount in the case of silica flour (about 85% by weight of which passes a 200 mesh testing sieve) is in the neighborhood of 70% and the same is true of one variety of talc. In the case of one variety of Kaolin clay, the critical point is around 65%. The foregoing fillers are non-fibrous in character. In the case of a fibrous filler such as asbestiform mineral fibers, the critical point is considerably lower and may be of the order of 35%, but this depends on the state of subdivision of the fiber, for, as will be pointed out more in detail hereinbelow, different screen fractions of a fibrous filler material differ very greatly in their property of imparting

flow resistance and of affording high fire resistance.

We have discovered that the property of greatly increasing the flow resistance and fire resistive properties does not parallel the consistency and viscosity characteristics of the composition as determined at 400° F. by a Wagner-Bowen plasticimeter. In order to provide a satisfactory fire resistive roofing, the Wagner-Bowen plasticity value should not be excessive, if the mixture is to be spreadable in a heat plasticized condition to provide a coating layer which is uniform in consistency and in thickness. The Wagner-Bowen plasticity value also has a bearing upon the fire resistive properties of the composition, for, if it is too high, the bitumen does not form the desired cementitious bond when the composition is exposed to flame so as to provide a continuous coherent mat-like residue and so as to remain adherent to the underlying sheet-like base. The point at which the Wagner-Bowen plasticity value rapidly increases is also critical for any given filler, but is different from the critical point at which the flow resistance coefficient rapidly increases. There is, therefore, for each filler or filler mixture a narrow zone determined by the minimum flow mixture coefficient and the maximum Wagner-Bowen plasticity value, within which special bituminous coating compositions having high fire resistive properties are obtainable. It is one of the purposes of this invention to employ such highly fire resistive coating compositions in combination with open-textured long fiber felt base sheet material of the character above defined.

Since the existence and criticality of the aforesaid properties have been the result of our discoveries, the method and apparatus for accurately measuring the properties have been especially designed by us and in order that the values referred to may be determined and reproduced by others, the description of the testing apparatus and procedures is given hereinbelow in connection with the following drawings wherein

Fig. 1 is a side elevation partly in section of a testing device for determining the flow resistance coefficient of filler material as incorporated in a bituminous composition;

Fig. 2 is a plan view partly in section of the testing device;

Fig. 3 is a front elevation of the test panel assembly;

Fig. 4 is a side elevation of the test panel assembly;

Fig. 5 is a plan view of the test panel assembly with parts thereof broken away;

Fig. 6 is a perspective view of the frame and guard plate used in the test panel assembly;

Fig. 7 is a longitudinal sectional detail view on an enlarged scale of the orifice in the gas line leading to the burner of the testing device;

Fig. 8 is an end elevation of the Wagner-Bowen plasticimeter testing device;

Fig. 9 is a plan view of the plasticimeter;

Fig. 10 is a front elevation partly in section of the plasticimeter;

Fig. 11 is a side detail elevation of the drag tool and drag tool support of the plasticimeter;

Figs. 12a, 12b, and 12c are views of the smoothing blade of the plasticimeter taken respectively in elevation normal to the line A—A of Fig. 9, from the bottom, and in elevation from the back in the direction of the line A—A of Fig. 9;

Figs. 13a, 13b, and 13c are views of the mixing blade of the plasticimeter taken respectively in

elevation normal to the line B—B of Fig. 9, from the bottom, and in elevation from the back in the direction of the line B—B of Fig. 9.

The flow resistance coefficient of a filler material is determined by measuring the amount of flow of a bituminous composition containing the filler, when the bituminous composition in the form of a coating of given thickness applied to a standard base is subjected to actual flame exposure under accurately controlled conditions. The testing apparatus is shown in Figs. 1 to 7.

The bituminous composition to be tested is applied to a 30 gauge "black iron" sheet, so that the resulting coating will be applied at the rate of 30 pounds, plus or minus 2 pounds, per 100 square feet.

The test is made in a wind tunnel 50 having a fan 51 at one end and a stack 52 at the other end. The tunnel is made of ½ inch thick asbestos-cement lumber and has two windows 53 and 54 therein which can be opened and closed by any suitable means (not shown).

Within the tunnel are the burner and testing deck which are located between two shields 55 and 55¹ of the asbestos-cement lumber spaced 12½ inches apart, and which are rigidly mounted on the asbestos-cement slab 56. The inclined test deck is indicated generally by the reference character 57 and comprises a lower frame-like member 58 having ¼ inch pegs 59 projecting from the face adjacent the upper and lower margins. Between the pegs, strips 60 of asbestos-cement boards 1¾ x 12 x ¼ inches are placed. One ply 12 x 12 inches of the prepared coated sample 73 to be tested is placed on the asbestos-cement boards 60 followed by an L-shaped guard plate 61 which guards the bottom edge of the sample. The assembly is held down by an iron frame 62 and held in place by thumb screws 63.

After the test deck has been assembled, it is placed on the inclined support 64 which has an opening in the back underneath the strips 60 and which has side flanges 65 to protect each side of the test panel. The support 64 comprises a baffle 66 to prevent the flame licking around behind the test deck. The support, which is made of iron, is mounted on the asbestos-cement slab 56 which measures 12 x 40 x 1 inches. The parts for carrying the test deck are also made of iron. An iron bar 67, 12 x 1 x ¼ inches is placed across the top edge of the deck to protect the sample at this point.

In front of the test deck is the burner 68 comprising an iron pipe having an inside diameter of .472 inch and an outside diameter of .675 inch, with 17 holes .073 inch in diameter and ½ inch apart disposed at an angle that is parallel with the plane of the test deck. The burner is provided with an inlet line 69 controlled by a shut off valve 70. In the line 69 is an orifice 76, ⅝ inch in diameter, that is located in the housing connections 71. Between the valve 70 and the orifice is a manometer 72. By this arrangement, a supply of gas under constant pressure can be obtained, thereby obtaining a steady flame of constant intensity during the test.

At the base of the test deck and between the burner and the deck holder is placed a pan 74, ¾ x 1 x 10 inches, which collects any bituminous coating material that flows from the sample during the exposure to the flame. This pan is provided with a removable cover 75.

The dimensions of the different parts of the

testing apparatus shown in the drawings as used by us are as follows:

a—24 inches	o—8 inches
b—38 inches	p—2 inches
b'—6 inches	q— $\frac{3}{4}$ inch
c—18½ inches	r—1 inch
d—72 inches	s—12 inches
e—16½ inches	t—7 inches
f—10 inches	u—10½ inches
g—12¼ inches	v—12 inches
h—38 inches	w—12 inches
i—64 inches	x—1 inch
j—32 inches	y—12 inches
k—24 inches	y'—1 inch
l—24 inches	z—6 inches
m—40 inches	aa—12 inches
n—27 inches	bb—1 inch

In carrying out the test, the apparatus is first assembled and the burner is lighted so as to provide a pilot flame that is about ½ inch in length when the fan is operating. The windows 53 and 54 are then closed and the room in which the apparatus is placed is arranged so that there will be relatively constant conditions during the test. The temperature and relative humidity of the room should be approximately 80° F. and 40%, respectively. The fan should generate a wind velocity of about 150–155 feet per minute at the portion of the deck exposed to the flame. The valve 70 is then opened until a flame about 8 to 9 inches long is produced having a temperature of about 1325–1370° F. When the valve 70 is opened to produce such flame, a timing device is started. After the sample has been exposed to the flame for a period of five minutes the flame is turned off and simultaneously the cover 75 is placed over the top of pan 74 so as to prevent any further bituminous composition that may flow down the test deck from collecting in the pan. Before the commencement of the test the part 74 is carefully weighed and at the conclusion of the test is again weighed, the difference in weight being the number of grams of bituminous coating material that has flowed into the pan. Before making the test the weight of the bituminous coating on the test sample is determined by weighing the base sheet material before the bituminous coating is applied thereto and weighing the complete sample, the difference being the weight of the coating.

The coefficient of flow resistance is computed according to the following expression:

$$100 - \frac{\text{weight in grams of coating collected in pan} \times 100}{\text{total weight in grams of coating prior to test}}$$

For example if the bituminous coating on the sample weighed 1000 grams prior to testing and 50 grams of the coating were collected in the pan during the test the coefficient of flow resistance would be

$$100 - \frac{50 \times 100}{1000} = 95$$

If no coating composition flows into the pan then the flow resistance coefficient is 100 which is the maximum attainable.

In order to afford high fire resistance, the flow resistance coefficient of the filler should be 50 or greater, and it is preferable that the flow resistance coefficient be 75 or greater. For optimum fire resistance the flow resistance coefficient should be 100, and compositions of such high flow resistance coefficient can readily be afforded in accordance with the principles dis-

closed herein. The foregoing is to be contrasted with ordinary coating asphalt having a softening point of about 230° F. which has a flow resistance coefficient of only 30. The foregoing is also to be contrasted with a conventional "filled" asphalt, e. g., containing about 30% by weight of limestone dust which has an even lower flow resistance coefficient, namely, a flow resistance coefficient of only 26. In the case of a filler material such as limestone dust, it has been mentioned above that even further additions up to about 70% by weight have little effect, and that it is not until 75%–80% of filler is reached that the flow resistance coefficient is greatly increased, and at such point the flow resistance coefficient will be in the neighborhood of 60 to 65.

For any given bitumen, the flow resistance coefficient is a function of the particular filler that is employed. However, there is some difference in flow resistance coefficient depending upon the bitumen that is employed in the bituminous composition. Thus, the critical amount of filler for a so-called "cracked asphalt," namely, oxidized asphaltic cracking still residue, is usually somewhat less than for a straight run residue.

As mentioned above, fibrous filler material such as asbestiform mineral fiber is much more effective than a filler such as slate flour or limestone dust. For this reason, in order to obtain a high flow resistance coefficient while maintaining the total filler content relatively low (about 35% to 55% by weight of the bituminous composition) we prefer to employ fibrous mineral as all or part of the filler. A typical example of a highly fire resistive composition which may be employed in the practice of this invention is as follows: The bitumen used in the special coating composition preferably is a residual asphalt flux obtained from the refining of Mid-Continent petroleum which has been air-blown until it has a softening point of about 200 to 205° F. The asphalt constitutes about 45% by weight of the coating composition. With this asphalt there is admixed and uniformly distributed finely-divided solid water-insoluble heat-resistant mineral filler which constitutes about 55% by weight of the composition. The filler consists of slate flour which constitutes about 20% by weight of the composition, and asbestos dust which constitutes about 35% by weight of the composition. The screen analysis of the asbestos dust of this example of the practice of this invention follows, the percentages being percentage by weight of the bituminous composition as a whole. The screen grading of asbestos fiber passing a 6 mesh testing sieve and retained on an 8 mesh testing sieve is indicated (–6+8). Other screen gradings are indicated similarly below and elsewhere herein.

Asbestos dust screen gradings:

	Per cent
–6+8	.5
–8+10	.5
–10+14	1.0
–14+20	1.5
–20+28	1.5
–28+35	2.0
–35+48	2.0
–48+65	3.0
–65+100	5.0
–100+150	5.0
–150+200	5.0
–200	8.0
Total	35.0

Such asbestos dust is considerably finer in particle size than the asbestos which is ordinarily placed on the market and which is known in the art as fiber. Heretofore such asbestos dust has generally been discarded in enormous quantities in dumps at the mines as a waste product while the usual asbestos fibers of commerce have been recovered and sold.

In preparing the special coating material, the asphalt is heated to a heat-liquefied condition and the asbestos dust and slate flour are thoroughly mixed therewith. The mixing preferably is carried out at a temperature of about 375° to 475° F. It is important that the bitumen and finely-divided filler be thoroughly mixed together so that the filler becomes intimately commingled and distributed uniformly throughout the mass and so that the composition of the coating with respect to the distribution of the filler therein is uniform throughout.

The coating composition as thus prepared is then applied by a hot-spread coating operation to a base sheet which, according to the present invention, is in the form of an open textured long fiber felt of low fiber web Densometer value. The glass fiber felt above described for purposes of exemplification may be used. The base sheet is, as in the above-mentioned example, impregnated to the extent of about 450% with an asphaltic bitumen having a softening point of about 190° F. The special coating composition is applied to the base sheet so as to provide a layer that is substantially uniform in consistency and in thickness at the rate of about 80 pounds per 100 square feet. Conventional granular surfacing may be embedded in the coating. The asphaltic back coating preferably consists of the special coating composition.

The special bituminous coating composition of the foregoing example has a flow resistance coefficient of 100. The character of the special base sheet contributes to the high fire resistive properties of the roofing, for the open textured base sheet co-operates with the highly flow resistant coating to maintain the coating composition in place during exposure of the roofing to fire. This advantage is obtained without regard to the employment or non-employment of mineral fiber, namely, glass, in the felt base sheet and constitutes the principal factor in attaining high fire resistance. However, by employment of a mineral fiber such as glass in the base sheet, the fire resistance of the roofing is further increased.

As aforesaid, the different screen gradings of a fibrous mineral filler, such as asbestos dust, have been found by us to differ considerably in their effectiveness in imparting flow resistance to a bituminous coating composition. In view of these differences in effectiveness of the different screen gradings of a mineral fiber, such as asbestos dust, we have, in order to indicate more definitely the amount of mineral fiber of given screen gradings that is used to effect stabilization of a bituminous composition, assigned to the different screen gradings what we have called the "screen factor" for each of the different gradings. Thus, if the screen grading (—28+35) is taken as having a screen factor of unity, any screen grading that requires half the amount as compared with the screen grading (—28+35) will be twice as effective and will have a screen factor of 2. On the other hand, a screen grading that requires twice the amount as compared with the screen grading (—28+35) will only have half the effectiveness and will have a screen factor of 0.5.

In like manner, screen factors can be assigned to each of the other screen gradings.

The above described test for measuring the flow resistance coefficient of a filler in a bituminous composition affords a convenient basis for setting up the screen factors of fibrous mineral fillers on a definite scale, and, when the screen factor of a given screen fraction of a fibrous mineral is referred to herein the screen factor as determined in the following manner is intended. The test for determining the flow resistance coefficient is carried out using the test apparatus and procedure above described, except that in all cases the total filler is 55% by weight of the bituminous composition. The asphalt in all cases is oxidized straight run asphalt having a softening point of about 205° F. Slate flour (85% by weight of which passes a 200 mesh testing sieve) is used in all cases as the standard diluent filler, and the amount by weight of the particular screen grading of fibrous mineral to achieve a flow resistance coefficient of about 75, as ascertained by the average of a plurality of test runs, is determined. Each sample is prepared using a 30 gauge "black iron" sheet as the base sheet. As a standard, chrysotile asbestos dust of the grading (—28+35) is taken as having a screen factor of unity and the factor for each of the other gradings is calculated to this standard. By way of concrete example, it being the case that substantially 9.5% by weight of the bituminous composition of chrysotile asbestos dust having the screen grading (—28+35) (the total filler being 55% as aforesaid), is required to afford a flow resistance coefficient of substantially 75, and it also being the case that only about 1% by weight of the bituminous composition of the screen grading (—6+8) (the total filler again being 55%), also affords a flow resistance coefficient of about 75, the screen factor of chrysotile asbestos dust having the screen grading (—6+8) is seen to be about 9.5.

By way of further illustration, the "screen factors" of the different screen gradings of a typical chrysotile asbestos dust on the basis above mentioned are as follows:

Screen grading:	Screen factor
—6+8	9.5
—8+10	8.0
—10+14	6.0
—14+20	4.0
—20+28	1.9
—28+35	1.0
—35+48	.6
—48+65	.4
—65+100	.3
—100+150	.2
—150+200	.2
—200	.1

It may be noted that in the case of the screen grading (—200), a flow resistance coefficient of 75 was not attained even when the total filler (55% by weight) consisted of this particular grading of asbestos dust. However, since this particular screen grading (—200) is somewhat effective when combined with the more effective screen gradings, a screen factor of .1 has been assigned to this particular screen grading. Similarly, in the case of any other fibrous minerals, a screen grading which, when employed to the extent of 55% by weight, does not attain a flow resistance coefficient of 75 is to be regarded as having a screen factor of .1. While the screen factors of the different screen gradings are determined on

the basis of a flow resistance coefficient of 75 as a standard of comparison, it is not necessarily the case that the flow resistance coefficient be as high as 75 in order to afford a roofing having very fire resistive properties when an open textured long fiber base sheet is used in combination with the weatherproofing coating layer.

The foregoing is seen to afford a convenient way of knowing and defining the flow resistance effectiveness of a fibrous mineral filler. Given the percentage by weight of each of the screen gradings of mineral fiber contained in a bituminous composition, the percentage by weight of each multiplied by the screen factor for each gives what we term herein and in the claims the "grading index" for each of the screen gradings; and, by adding the grading indices for the several screen gradings together, the grading index for the total mineral fiber is readily determined. By way of illustration, the grading index of the mineral fiber component of the above-mentioned typical embodiment of this invention is as follows:

Screen Grading	Per cent by Weight of Composition	Screen Factor	Grading Index
-6+8	.5	×	9.5
-8+10	.5	×	8.0
-10+14	1.0	×	6.0
-14+20	1.5	×	4.0
-20+28	1.5	×	1.9
-28+35	2.0	×	1.0
-35+48	2.0	×	.6
-48+65	3.0	×	.4
-65+100	5.0	×	.3
-100+150	5.0	×	.2
-150+200	5.0	×	.2
-200	8.0	×	.1
Total grading index for mineral fiber			32.3

In determining the screen fractions or gradings of fibrous or other mineral filler that are referred to herein, 8 inch diameter testing sieves of the W. S. Tyler Company sieve series, meeting American Society for Testing Materials Standard E11-39 for Sieves for Testing Purposes have been used. A quantity of filler to be screened, ranging from 200 to 300 grams, is placed in the uppermost of a series of testing sieves and subjected to suitable agitation as in a standard W. S. Tyler Company Ro-Tap sieve shaking machine for a period of five minutes in order to separate the original filler roughly into fractions retained in the different testing sieves. This operation is repeated, if necessary, in order to obtain about 100 to 200 grams of the desired screen fraction, which fraction is then individually rescreened for 15 minutes, using the Ro-Tap screening machine or the equivalent in order to remove any fines contained therein. The resulting screen fraction is material that has passed the coarser screen and is retained on the finer screen.

The mineral fiber filler that is preferred in the practice of this invention is asbestiform mineral fiber, chrysotile asbestos dust being especially desirable. Other asbestiform mineral fibers of the particle size mentioned may likewise be employed, such as Canadian picrolite, amosite, anthophyllite, tremolite and actinolite.

Another suitable fibrous material is a comminuted mixture of hydrated Portland cement and asbestos fiber, the hydrated Portland cement having become set with the asbestos fiber distributed therethrough. A convenient source of such material is asbestos-cement roofing scrap which usually contains about 20% to 35% by weight of

asbestos fiber and about 65% to 80% of hydrated Portland cement. Heretofore such scrap has been regarded as an unavoidable waste of no commercial value. However, by subjecting the scrap to a disintegrator such as a hammer mill, the resulting mass contains a multiplicity of short asbestos fibers to which the hydrated Portland cement adheres as nodules and for this reason this material is fibrous and is to be included in the term fibrous mineral.

Another material which is somewhat similar to asbestos-cement in that the material contains mineral fibers together with non-fibrous material, is disintegrated fiber-bearing serpentine rock. Since disintegrated fiber-bearing serpentine rock contains fibrous particles, such disintegrated serpentine rock is regarded as one form of fibrous mineral which is suitable for use in practicing this invention. However, depending upon the physical structure and fibrous mineral content of the particular serpentine rock that is used, the proportion of fiber contained therein is subject to some variation, but, as pointed out below, this merely has the effect of varying somewhat the screen factors of the various screen gradings of the disintegrated serpentine rock as calculated to chrysotile asbestos dust of the screen grading (-28+35) which has a screen factor of unity. Moreover, disintegrated fiber-bearing serpentine rock is of such character that in order to liberate the fibrous material contained therein it should pass a 20 mesh testing sieve, and, when reference is made herein to disintegrated fiber-bearing serpentine rock, only that serpentine rock which has been disintegrated so that it passes a 20 mesh testing sieve is intended, since the individual particles of coarser gradings are not fibrous in character but granular (are not fibrous mineral as this term is used herein), and since such coarser gradings are ineffective in producing highly fire resistant roofings.

Other mineral fibers may likewise be employed, such as mineral wool and glass fibers. The term "mineral wool" includes various products obtained by attenuating into fibrous form suitable fused materials such as rock or slag.

While the fiber component of the bituminous coating composition is preferably fibrous mineral, fibrous filler other than mineral fibers may be employed including animal, vegetable, and synthetic fibers. Thus one may employ cotton fibers. Wood fibers such as fine sawdust are suitable, or defibrated wood or paper and paper fibers such as ground wood, sulphite, and kraft paper pulps. Finely-divided wool fiber is suitable but is much more costly. Synthetic fibers such as regenerated cellulose (rayon) and cellulose acetate are likewise suitable.

In addition to fibrous fillers, another filler material that is similar to the fibrous filler in that it is effective in varying degrees depending upon the screen grading or mixture of screen gradings that is employed, is mica. Mica is characterized by its occurrence in the form of small plates which are thin relative to their lateral extent. Mica and fibrous materials both are characterized by the fact that at least one dimension is very small relative to another more extended dimension. Such materials are therefore referred to generally as planar-extended filler materials, thereby indicating that the particles are in the form of fibers or are in the form of plates having an extended dimension that is confined essentially to one of the three dimensional planes. In the case of a filler such as mica that exists in the form of

plates, particles which are retained in a 14 mesh testing sieve are undesirable in the composition.

With regard to planar extended fillers other than asbestiform mineral fibers, it is usually the case that a particular screen grading of, for example, asbestos-cement, cotton, wood, fiber, mica, etc., will not have the same screen factor as chrysotile asbestos fibers. In fact, even as among the different varieties of asbestiform mineral fibers there are some variations in this regard. However, utilizing chrysotile asbestos dust as the standard chrysotile asbestos dust of the screen grading $(-28+35)$ having a value of unity, the screen factor of this screen grading of other planar extended filler materials is determinable in the same way that the screen factors of the different screen gradings of chrysotile asbestos fiber are determinable. The foregoing with respect to the screen factors of other planar extended fillers also applies to the screen factors of mixtures of planar extended fillers, calculated to chrysotile asbestos dust of the screen grading $(-28+35)$ which has a screen factor of unity.

As has been mentioned above, the employment of a filler of the planar extended type is preferable, and is of importance particularly when the total filler content is of the order of 35% to 55% by weight of the bituminous composition. Generally in the formulation of a fire resistive bituminous composition for use in the practice of this invention, it is desirable to employ filler of the planar-extended type having a grading index the ratio of which to the percent by weight of waterproofing bitumen in the bituminous composition is at least 1 to 8 and preferably is at least 1 to 5.

When the filler content of the bituminous coating composition is above 55% the grading index of the planar extended filler may be somewhat less. Thus, for filler contents of the order of 55% to 65% by weight, the ratio of the grading index of the planar extended filler to the waterproofing bitumen is desirably at least 1 to 12 and preferably is at least 1 to 8.

When reference is made herein to "screen factor" or "grading index" of a filler of the planar extended type, the reference is to these values as hereinabove described.

When a filler of the planar-extended type is used, such filler should be used in the finely-divided or dust-like form as distinguished from long cotton, wood or asbestos fibers. Fibrous fillers that are retained on a 6 mesh testing sieve are undesirable in the coating composition. It has been mentioned above that mica retained on a 14 mesh testing sieve is undesirable. Accordingly, and somewhat more generally, it is desirable that the filler material of the planar-extended type be confined substantially to that which passes a 6 mesh testing sieve and which does not have more than one dimension greater than about .046 inch, which is the width of the opening in a 14 mesh testing sieve. When a filler of the planar-extended type is used that is coarser than has been mentioned above, such filler material tends to form into clumps or clots which render the bituminous composition non-spreadable to the uniformity that is required in a commercial product and also impairs the fire resistance due to occurrence of zones of insufficient protection and due to poor adhesion to the underlying base sheet upon exposure to fire. For this reason, it is highly desirable to limit the quantity of the coarser planar-extended filler to that which can be incorporated in a bituminous

composition that, when heat plasticized, is spreadable to form a layer of uniform thickness and uniform consistency. In this connection, a slight pebbled appearance of the surface is permissible provided the coating as a whole provides a good waterproof and weatherproof layer. It is usually desirable that less than 10% by weight of the total content of the bituminous coating composition consist of a filler of the planar-extended type which is retained on a 14 mesh testing sieve, although in the case of mineral wool a substantially greater proportion of such coarse material can be incorporated in a spreadable composition. While the presence of any fiber retained on a 6 mesh testing sieve is regarded as undesirable and while the presence of any mica retained on a 14 mesh testing sieve is regarded as undesirable, any small quantity of such excessively coarse planar-extended filler that may be present while still retaining spreadability is to be regarded as having the same screen factor as that of the fraction $(-6+8)$ in the case of fibrous materials and the fraction $(-14+20)$ in the case of mica. Usually, it is desirable that the ratio of the grading index of filler of the planar-extended type to the percent by weight of bitumen in the composition be less than about 1 to 1 and preferably less than about 1 to 1.5, so as to avoid the inclusion of an excessive amount of the coarser screen fractions.

When the coating composition is of the special fire resistive character aforesaid, the bitumen that is employed is of the order of 20% to 65% by weight of the composition, the filler being of the order of 35% to 80% by weight of the bituminous composition. Preferably all of the filler including the fibrous filler is finely-divided solid water-insoluble heat resistant mineral fiber. By "heat-resistant" any material is intended which is sufficiently heat resistant to retain structural integrity when subjected to flame temperature while incorporated in the roofing. In this connection, substances such as chrysotile asbestos, Canadian picrolite, hydrated Portland cement, etc., are regarded as heat resistant even though they contain water of constitution which may be driven off at temperatures below flame temperatures. It is not essential, however, that the filler material be heat resistant, although it is preferable that the percent by weight of heat resistant mineral filler to the percent by weight of bitumen in the composition be at least 1 to 3 and it is still better if this ratio is at least 1 to 2. Other examples of heat resistant fillers are fly ash, dead burned calcium sulphate, land plaster, precipitated calcium silicate hydrate, and the like.

It has been mentioned above that organic fibers may be employed. In addition other organic filler materials may be employed such as ground cork, coal, coke, ground hardened resins of the phenol aldehyde type, lignin resins etc. It is very surprising, but such organic filler materials may be employed in highly fire resistive compositions provided the flow resistance coefficient is at least 60. Where such is the case the coating tends to carbonize to a heat insulating mat, but if the flow resistance coefficient is low the coating flows and the organic material adds to the combustion rather than otherwise. In any event, both for organic and inorganic or mineral fillers the softening point of the filler as determined by the standard cube softening point test (carried out in air) should be at least 500° F. and preferably should be at least 700° F.

In the case of filler materials such as slate

flour, limestone dust, and the like, which are not of the planar-extended type it is preferable that at least 90% by weight pass a 100 mesh testing sieve. Referring to the filler as a whole in the bituminous composition, including the presence of planar extended filler, it is desirable that at least half by weight of the total filler pass a 100 mesh testing sieve and preferable that at least $\frac{2}{3}$ by weight of the total filler pass a 100 mesh testing sieve. This is especially the case when the bituminous composition contains 55% by weight or more of filler. In the ordinary case, if the total filler is between 35% and 55% by weight or even between 55% and 65% by weight of the bituminous composition, the major proportion of the total filler passes a 100 mesh testing sieve and when the total filler is over 65% by weight of the bituminous composition about $\frac{2}{3}$ or more of the total filler passes a 100 mesh testing sieve. In order to prevent the inclusion in the bituminous composition of undesirable granular material it is ordinarily desirable that the coating composition contain less than about 10% by weight of the total filler in the bituminous composition of particles retained on a 14 mesh testing sieve. While it is preferable to employ filler comprising filler of the planar-extended type, it is not necessary to do so provided the flow resistance coefficient is at least 50. However, when planar-extended filler is omitted the amount of filler is usually quite high, namely, of the order of 55% to 80% by weight of the bituminous composition.

As aforesaid, it is essential that the high flow resistance coefficient be attained in a bituminous composition that does not exceed a predetermined plasticity value at 400° F. The fire-resistant bituminous compositions fall within the narrow range wherein the flow resistance coefficient exceeds the minimum above mentioned, but have a plasticity value at 400° that does not exceed 1500 grams and that preferably does not exceed 1000 grams. The Wagner-Bowen plasticity value at 400° F., as aforesaid, imposes an upper limit on the total filler material that is employed for at a critical point the plasticity value begins to increase very rapidly and there is only a narrow operative zone within which fire resistive spreadable coatings are obtainable. The Wagner-Bowen plasticity value also imposes a limit upon the amount of planar extended filler material such as asbestos, cotton, mica or the like which can be employed particularly with regard to the coarser grades of fibrous materials which are commonly referred to as "fiber." In other words, such fibrous filler materials should be dust-like if the Wagner-Bowen plasticity value at 400° F. is not to be exceeded.

The plasticity value of the bituminous composition has been determined by us, using a Wagner-Bowen mixing bowl plasticimeter, manufactured by E. E. W. Bowen, Bethesda, Maryland. For testing bituminous compositions of the character mentioned herein, we have made certain modifications in this test apparatus. In view of this fact and further in view of the fact that we do not know of any available publication wherein this type of apparatus is described in detail, we have shown in the drawings, Fig. 8 to Fig. 13C, the testing apparatus which we have employed and a description of the apparatus and its operation follows. When reference is made herein or in the claims to Wagner-Bowen plasticity value, the plasticity value as determined by this test is intended.

The Wagner-Bowen plasticimeter consists of a

suitable base 100 which comprises in housing 143 suitable gear means (not shown) for rotating the vertical shaft 101 by power supplied from the motor 102. The shaft 101 carries for rotation at the upper end thereof the horizontally-disposed disk-shaped support 103 to the top of which is secured the bowl 104 by means of screws 105. By this arrangement, the bowl 104 can be rotated by operation of the motor 102, and any suitable means can be provided for maintaining the rotation of the bowl at a predetermined constant speed.

Emanating from the base 100 are arms 106 and 106' which carry on the upper ends thereof the cross bar 107. At the upper end of each arm 106 and 106' is a yoke 108 which is pivoted to lug 109 and which is provided with a thumb screw 110 in threaded engagement therewith so that, by loosening the thumb screw 110, the yoke 108 can be swung outwardly, permitting removal of the cross bar 107. At each end of the cross bar 107 is a positioning pin 111 by which the position of the cross bar is determined.

The cross bar 107 carries the means determining the plasticity of material in the bowl 104. Carried at the lower end of shaft 112 is the mixing blade 113, the vertical position of which relative to the bowl 104 is adjustable by nuts 114 which secure the shaft 112 to the cross bar 107. Adjacent the end of the mixing blade 113 is the thermometer 115 carried in a guard 116 attached to bracket 117 which in turn is secured at the upper end to the cross bar 107. Carried by the lower end of the shaft 118 is the smoothing blade 119. The shaft 118 and smoothing blade 119 are urged downwardly by the expansion spring 120 between the annular washer 122 and the sleeve 121 in which the shaft 118 is vertically slidable. The vertical position of the smoothing blade 119 is determined by the thumb screw 123 which rests against the upper end of sleeve 121 and which is in threaded engagement with the upper end of the shaft 118. Also rigidly secured to the cross bar 107 is the rod 124, the lower end of which can be adjusted so as to be a predetermined distance from the bottom of the bowl 104 by nuts 125.

At the center of the cross bar 107 is the bearing 126 for the vertical shaft 127 that is mounted for rotation therein at a given vertical position. Rigidly secured to the shaft 127 by means of the collar 128 is the arm 129 which carries the small cylindrical drag tool 130 at the lower end thereof. Carried by the upper end of shaft 127 and rotatable therewith is the arm 131, the outer end of which has a small amount of play for angular movement with the shaft 127 between opposed adjustable stop screws 132.

A horizontally-disposed stub shaft 133 is fixedly carried by arm 106' and pivotally mounted thereon is the T-shaped scale beam member comprising the vertically-extending arm 134 and the arms 135 and 136 extending horizontally on opposite sides of the stub shaft 133. Pivotally secured at the end of the arm 135 is the weight pan 137 and pivotally secured at the end of arm 136 is the counterweight 138 which substantially counterbalances the weight pan about stub shaft 133. Between the end of arm 131 and the end of arm 134 is the tie rod 139. One end of the tie rod 139 has a turned-down portion 140 which slips through an opening of corresponding size in the end of arm 131 to provide pivotal movement with respect thereto. The other end of tie rod 139 is in threaded engagement with a yoke member 141 which is pivotally mounted with respect to the

upper end of arm 134. If desired, a fixed stop arm 144 can be provided for convenience in restraining movement of arm 134 when the tie rod 139 is disengaged from arm 131. The contents of the bowl 104 can be heated as by the gas burners 142.

In the operation of the plasticimeter, the bowl 104 is rotated in a clockwise direction, and, as the bowl continues to operate, the mixing blade 113 scrapes the heated contents of the bowl away from the outside bottom portion thereof and the thermometer 115 records the temperature of the contents of the bowl at this point. The contents of the mixing bowl are next carried under the smoothing blade 119 which smooths the contents of the bowl at this point. The contents of the mixing bowl are next carried under the smoothing blade 119 which smooths the contents of the bowl to a predetermined level above the outer portion of the bottom of the bowl. The lower end of the rod 124 serves as a check to determine whether the smoothing blade 119 is smoothing the contents of the mixing bowl to the desired level, and, if it is not, the vertical position of the smoothing blade 119 is adjusted by thumb screw 123. The heated contents of the bowl at the predetermined desired level therein are then carried past the drag tool 130 and this tends to rotate the shaft 127 in a clockwise direction and to pull the weight pan 137 upwardly through the system of lever arms and tie rod hereinabove described. The greater the plasticity (i. e., the heavier the consistency) of the contents of the bowl 104, the greater will be the drag on the drag tool 130 that tends to elevate the weight pan 137. By placing weights on the weight pan 137 and while continuing to rotate the bowl 104, the device can be brought into a state of equilibrium so that the end of the arm 131 will be approximately midway between stop screws 132—132, and the weight in grams required to achieve this condition of equilibrium for a particular composition contained in the mixing bowl is the Wagner-Bowen plasticity value. This value is dependent upon the dimensions of the apparatus and the manner of use of the apparatus in making the determination. The essential dimensions of the apparatus shown in the drawings are:

a—3½ inches	r—4½ inches
b—9½ inches	s—1¼ inches
c—½ inch	t—2½ inches
d—8° angle	u—2⅝ inches
e—7⅞ inches	v—1⅞ inches
f—4° angle	w—9° angle
g—3⅝ inches	x—5⅞ inch radius
h—2⅝ inches	y—11° angle
i—2⅝ inches	z—1 inch
j—2¼ inches	aa—3⅝ inches
k—4¾ inch radius	bb—2¾ inches
l—1⅞ inch radius	cc—¾ inch
m—1⅞ inch	dd—⅝ inch
n—2⅝ inches	ee—¼ inch
o—1⅞ inches	ff—24° 15' angle
p—2⅝ inches	gg—⅝ inch radius
q—3⅜ inches	hh—⅛ inch

The dimensions of the mixing blade 113 and of the smoothing blade 119 are indicated on the face of the drawings.

The Wagner-Bowen plasticity values at 400° F. given herein are determined in the following way. The height of the lower end of the measuring rod 124 from the bottom of the bowl 104 is adjusted so as to be ⅞ inch. The height of the bottom of the drag tool 130 from the bottom of the bowl

is set so as to be ¼ inch. The weight of the composition to be tested is approximately 1500 grams. The weight is not critical provided the thickness of the composition as it leaves the smoothing blade just clears the lower end of the measuring rod 124. The composition is mixed while in the bowl 104. Throughout the test the bowl 104 is rotated at the rate of 60 rotations per minute. The bitumen that is used in the composition while in a heat-liquefied condition is poured into the bowl while the bowl is rotating. The bitumen is brought to a temperature of 400° F. by the burners and, while maintaining the temperature at 400° F., the filler material is added in small increments until it has all been incorporated. The smoothing blade 119 is then adjusted so that the composition as it leaves the smoothing blade just clears the lower end of the measuring rod 124. Weights are then placed on the weight pan 137 until the arm 131 is brought to a position between the stop screws 132, the weight to bring about this condition of equilibrium being recorded. Such recordings are made every three consecutive readings. The weight in grams for the last three consecutive readings is taken as the Wagner-Bowen plasticity value of the composition at 400° F.

With regard to the waterproofing bitumen that is used in the bituminous coating composition, any suitable waterproofing bitumen may be used. In preferred practice, the softening point will be between 190 and 230° F. Bitumens of high softening point may, however, be employed up to about 300° F. In the fire resistive type of coating composition, the softening point of the bitumen is desirably kept below about 275° F. At the other extreme bitumens having a softening point of down to about 160° F. may be used for a variety of roofing products especially when stabilized by inclusion of filler so as to provide a high flow resistance coefficient. In the latter type of coating bitumens having a softening point as low as 80° F. may be employed in the manufacture of flexible type roofings such as roll roofing although it is normally desirable that the bitumen have a softening point of at least about 120° F. The softening point of the bitumen is determined by the standard Ring and Ball softening point test. A particularly desirable bitumen is asphaltic bitumen derived from Mid-Continent crudes, although other asphalts derived from Mexican, Venezuelan and Colombian crudes also may be employed. Moreover, other bitumens such as pitches, coal tar and the like may also be used in the practice of this invention. Moreover, any modifier, e. g., of a resinous or oily character that may be mixed and blended with the bitumen is to be regarded as part of the bitumen content of the composition. While the flow resistance of a filler is usually somewhat higher in the case of cracked asphalts as compared with other asphaltic materials, cracked asphalts do not have as long weathering life and for this reason it is usually preferable to use the usual type of roofing asphalt where the roofing is to be exposed directly to the sun and to weather.

There are certain bituminous materials consisting wholly or partially of bitumens having a softening point above 500° F. e. g. pyrobitumens such as grahamite, albertite, and the like or even blown asphalts. These materials, if present in finely-divided condition, are to be regarded as part of the filler rather than part of the waterproofing bitumen.

The coating composition that is applied to the

open textured long fiber felt base sheet may be applied in any desired thickness. In preferred roofing materials, it is usually applied as a coating ranging from about 20 to about 100 pounds per 100 square feet, although for certain purposes coating up to about 120 pounds per 100 square feet may be employed.

It is a further feature of preferred embodiments of this invention that a bituminous material conforming to the requirements for the special fire-resistive coating layer is introduced into the open textured long fiber base sheet so that the filler material contained in the coating composition will be caused to occur in the body portion of the base sheet. This may be done in any suitable way as by applying the special coating composition in a heat plasticized condition to the open textured sheet material while the sheet material is in an open porous condition, and passing the sheet material between rolls so as to force the bituminous material into the base sheet. During the operation, a considerable proportion of the filler material is carried into the body of the base sheet and will occur throughout the body of the base sheet. In preferred practice, the filler material is caused to occur in greater amount adjacent the surface of the base sheet which in the finished product carries the water-proofing coating layer, while the mid-portion of the base sheet is caused to contain a lesser quantity of the filler material and thereby afford a roofing product of greater flexibility. Such non-uniform introduction of filler material into the body portion base sheet can be promoted by employing a combination of filler materials, e. g., a fibrous filler in combination with a pulverulent filler such as limestone dust or slate flour. In such case, the longer portion of the fibrous filler will tend to become entangled with the fibers of the base sheet adjacent the surface of the base sheet while only the more pulverulent and finely divided filler will penetrate more deeply into the base sheet. Moreover, when the filler-containing bituminous impregnating material is applied to an open textured web so as to cause both the bitumen and the filler to penetrate into the body portion of the web, some of the filler material which is not carried into the body portion of the web becomes deposited at the surface of the web so as to produce at the zone of the interface between the web and the weatherproofing coating layer, that is, either simultaneously or subsequently applied, a stratum wherein the proportion of filler is substantially greater than it is in the body portion of the base sheet and in the body portion of the weatherproofing coating layer. This is of advantage in anchoring the weatherproofing bituminous layer to the web material in a manner that is especially effective in preventing movement of the weather resistant coating layer relatively to the base sheet either because of tendency to slide at summer sun roof surface temperatures or because of tendency to flow upon exposure of the roofing to flame temperatures.

When the bituminous impregnating material comprises filler which is introduced into the body portion of the base sheet, the bond between the base sheet and the coating layer is particularly effective. Moreover, the stability and weather resistance of the base sheet is increased, which is important particularly with regard to edge portions of the roofing that are exposed to the weather. If desired, the same fire-resistive bituminous material that is employed as the water-proofing coating layer may be employed to im-

pregnate the open textured web and in such case the roofing will be very homogeneous throughout except for the occurrence of the open textured base sheet adjacent the back surface of the roofing and except that in the mid-portion of the base sheet the filler content of the bituminous impregnating material will be somewhat less than the filler content of the coating. The employment of filler, particularly heat resistant mineral filler, in the bituminous impregnating material for the base sheet is also of advantage in affording a higher degree of fire resistance, since the filler acts to increase the bulk and impermeability of residual carbonized mat that serves to protect an underlying roof deck upon exposure of the roofing to flame.

The advantages which result from the occurrence of a filler material in the impregnating material used to impregnate the open textured long fiber base sheet may also be realized, even when the bituminous impregnating material is not formulated according to the requirements for a highly fire-resistive coating. Thus the bituminous impregnating material may merely contain some conventional filler such as 25% to 40% by weight of limestone dust or slate flour or any other of the finely divided solid water insoluble filler materials having a softening point above 500° F. that have been mentioned hereinabove. In any event, it is desirable that bituminous impregnating material that has been introduced into the body of the open textured long fibered base sheet contain filler which constitutes at least 5% by weight of bituminous impregnating material and preferably at least 10% by weight of the bituminous impregnating material. Moreover, the introduction into the base sheet of bituminous impregnating material comprising filler is of general advantage whether or not the coating layer is specially formulated so as to have high fire resistance.

When the impregnating material is introduced into the open textured long fiber sheet material, the fibrous sheet is preferably dry, namely, has not previously been impregnated with a bitumen that is free of filler. Such conditions favor maximum penetration of the filler contained in the impregnating material into and throughout the thickness of the open textured sheet material. However, even when the open textured sheet material has previously been impregnated with a bitumen that is free of filler, the sheet material may remain quite porous notwithstanding the fact that the sheet material may have been impregnated to the extent of, for example, 300% or greater, by weight on the weight of the fibers in the sheet material. By thereafter applying a filler-containing bituminous impregnating material to the sheet material so as to introduce the bituminous material into the remaining pores and interstices in the sheet material, a substantial quantity of the filler can be introduced into the body portion of the sheet material. By varying the initial impregnation of the open textured sheet material with filler-free bitumen, the amount of filler introduced into the body of the sheet material can be controlled. Filler likewise can be introduced into the body of the sheet material merely by applying a filler-containing bituminous composition for the primary purpose of providing a coating layer, when the coating layer is applied to an open textured base sheet that still retains considerable porosity, for the coating will penetrate into the pores and will carry filler particles into the base sheet while at

the same time becoming spread as a coating layer. When the base sheet is caused to be impregnated with a filler-containing bituminous impregnating material, the base sheet preferably has a Densometer value of at least 25 seconds. In this connection, it may be mentioned that when reference is made to "fiber web Densometer value" herein or in the claims, the reference is to the Densometer value of the fibrous web as it occurs prior to the application of bituminous impregnating material thereto. In the finished roofing, such value can readily be determined by extracting the bitumen from the web by means of a suitable solvent such as carbon tetrachloride. When the reference is merely to "Densometer value," the Densometer value of the sheet material as it stands is intended, whether it is unimpregnated or is impregnated with a bituminous impregnating material.

It is desirable to employ as the filler material that is used in the bituminous material of the weatherproofing layer or that is caused to occur in the body portion of the base sheet, and preferably both, a filler such as chrysotile asbestos fiber which contains water of constitution liberatable at or below flame temperature. Chrysotile asbestos contains about 12% to 15% of water of constitution. Canadian picrolite and serpentine rock are similar to chrysotile asbestos in this regard. Other materials such as kaolin clay, hydrated Portland cement and calcium silicate hydrate contain 10% or more by weight of water of constitution. When the filler material contains water of constitution liberatable at or below flame temperature, the liberation of moisture causes the bituminous composition to develop pores to a greater extent than otherwise when the bituminous material is exposed to flame temperature and this is desirable since the pores tend to augment the heat insulating efficiency of the residuum that is formed upon exposure of the roofing to flame temperature. It is desirable that the bituminous material have incorporated therein a filler containing water of constitution, which water of constitution that is liberatable at or below flame temperatures amounts to at least 5% by weight of the filler contained in the bituminous material, whether the bituminous material constitutes the weatherproofing coating layer or occurs as the impregnating material with which the body portion of the base sheet of the roofing is impregnated.

For most purposes, it is desirable that the roofing be pliable. It is desirable that a roofing having a bituminous waterproofing coating such as shingles be sufficiently pliable to be bent 180° in two seconds about a mandrel 10 centimeters in diameter at 77° F. with the coating on the outside without cracking the coating through to the base on which it is applied, and any such coating or layer is referred to herein as "pliable" at ordinary temperatures.

While this invention has been described in connection with certain illustrative embodiments thereof, it is to be understood that this has been done for the purpose of exemplification. Accordingly, the scope of this invention is to be governed by the language of the following claims construed in the light of the foregoing description of our invention.

We claim:

1. A prepared roofing material comprising a fibrous sheet-like base and integrally bonded with said sheet-like base a weatherproofing layer of bituminous material applied at the rate of about

20 to about 120 pounds per 100 square feet of the roofing material, said bituminous material comprising at least 30% by weight of finely-divided solid water-insoluble filler having a softening point above 500° F., the fibers in said sheet-like base consisting in major proportion by weight of fibers at least ½ inch in length and being disposed in open textured essentially individual contacting arrangement to provide a sheet-like body between about 0.005 and about 0.1 inch in thickness and having a fiber web Densometer value not greater than about 50 seconds, and said sheet-like base being impregnated with a bituminous impregnating material comprising a finely-divided solid water-insoluble filler having a softening point above 500° F. that is contained within the body portion of said sheet-like base to the extent of at least 25% by weight of said bituminous impregnating material within said body portion of said sheet-like base and with the filler content of said impregnating material substantially greater adjacent the surface of said sheet-like base than adjacent the mid-portion of said sheet-like base.

2. A roofing comprising a fibrous sheet-like base and integrally bonded with said sheet-like base a highly fire resistant weatherproofing layer of bituminous material, the fibers in said sheet-like base consisting in major proportion by weight of fibers at least about ½ inch in length and being disposed in open textured essentially individual contacting arrangement to provide a sheet-like body about 0.005 to about 0.1 inch in thickness and having a fiber web Densometer value not greater than about 50 seconds, said sheet-like body being impregnated substantially throughout with a bituminous impregnating material containing a finely-divided solid water-insoluble filler having a softening point above 500° F. which constitutes at least about 25% by weight of said bituminous impregnating material that is contained within said sheet-like body, said bituminous material of said weatherproofing layer containing bitumen having a softening point from about 80° F. to about 275° F. and of the range 20% to 65% by weight of said bituminous material, and said bituminous material of said weatherproofing layer containing intimately commingled and distributed uniformly throughout finely-divided solid water-insoluble filler of the range 35% to 80% by weight of said bituminous material and having a softening point above 500° F., said filler having a flow resistance coefficient of at least 50 when said bituminous material is exposed to flame temperature under the flow resistance test defined herein, said bituminous material providing a weather resistant layer of the hot spread coating type integrally bonded with said sheet-like base and having a Wagner-Bowen plasticity value at 400° F. not substantially greater than 1500 grams.

3. A roofing according to claim 2 wherein the filler contained in said bituminous material of said weatherproofing layer comprises planar extended filler consisting of particles passing a 6 mesh testing sieve and not having more than one dimension greater than .046 inch, the ratio of the grading index of which particles to the percent by weight of bitumen in said bituminous composition being at least 1 to 8.

4. A roofing according to claim 2 wherein the filler contained in said weatherproofing layer of bituminous material comprises heat resistant mineral filler the ratio of the percent by weight

of which to the percent by weight of the bitumen in said bituminous material is at least 1 to 3.

5. A roofing according to claim 2 wherein the filler contained in said weatherproofing layer of bituminous material comprises mineral fiber passing a 6 mesh testing sieve, the ratio of the grading index of which to the percent by weight of bitumen in said bituminous material is at least 1 to 12.

6. A highly fire resistant prepared bituminous roofing material comprising a fibrous base sheet and a layer of thermoplastic bituminous material carried by said base sheet, the fibers in said base sheet consisting in major proportion by weight of flexible fibers at least about 1/2 inch in length and being disposed in open textured felted arrangement to provide a sheet the thickness of which is between about 0.01 and about 0.03 inch and having a fiber web Densometer value not greater than about 10 seconds, said base sheet comprising a heat-resistant binder and having a tensile strength of at least 10 pounds per linear inch of width, said base sheet being impregnated substantially throughout with a bituminous impregnating material containing a finely-divided solid heat resistant mineral filler which constitutes at least about 25% by weight of said bituminous impregnating material within the body portion of said fibrous base sheet, said thermoplastic bituminous material containing bitumen having a softening point above 120° F. and not above 275° F. and of the range 20% to 65% by weight of said bituminous material, said bituminous material containing intimately commingled and distributed uniformly throughout finely divided solid water-insoluble filler having a softening point above 500° F. and of the range 35% to 80% by weight of said bituminous material, said filler having a flow resistance coefficient of at least 50 when said bituminous material is exposed to flame temperature under the flow resistance test as defined herein, said bituminous material having a Wagner-Bowen plasticity value at 400° F. not greater than about 1500 grams, said layer of bituminous material being of the hot-spread coating type integrally bonded with said base sheet and being applied at the rate of about 20 to about 120 pounds per 100 square feet of roofing material, said roofing material being pliable at 77° F.

7. A highly fire resistant prepared bituminous roofing material according to claim 6 wherein the filler contained in said thermoplastic bituminous material comprises planar extended filler consisting of particles passing a 6 mesh testing sieve and not having more than one dimension greater than .046 inch, the ratio of the grading index of which particles to the percent by weight of bitumen in said bituminous composition being at least 1 to 12, and the ratio of the grading index of the planar extended filler contained in said thermoplastic bituminous material to the percent by weight of the bitumen in said thermoplastic bituminous material being not greater than 1 to 1, and wherein the filler contained in said thermoplastic bituminous material comprises heat resistant mineral filler the ratio of the percent by weight of which to the percent by weight of bitumen in said bituminous material is at least 1 to 3.

8. A highly fire resistant prepared roofing material according to claim 6 wherein said impregnated open textured base sheet is the product of impregnating said open textured base sheet with bituminous impregnating material conforming to the requirements for said thermoplastic bitu-

minous material of said layer carried by said base sheet.

9. A roofing according to claim 2 wherein the fibers of said sheet-like base consist in major proportion by weight of fire resistant fibers.

10. A roofing according to claim 2 wherein the fibers of said sheet-like base consist to the extent of at least 65% by weight of mineral fibers.

11. A roofing comprising a fibrous sheet-like base and integrally bonded with said sheet-like base a weatherproofing layer of bituminous material, said roofing being characterized in that said fibrous sheet-like base is about 0.005 to about 0.1 inch in thickness and consists in major proportion by weight of fibers at least about 1/2 inch in length which are disposed in open-textured essentially individual contacting relation and are co-bonded by a heat-resistant binder material to provide a sheet-like body having a fiber web Densometer value not greater than about 50 seconds and having a tensile strength of at least 10 pounds per linear inch of width, said sheet-like base being impregnated substantially throughout with a bituminous impregnating material which contains at least 25% by weight of finely-divided solid water-insoluble heat-resistant mineral filler disposed within the body portion of said sheet-like base, and said waterproofing layer of bituminous material containing at least 30% by weight of finely-divided solid water-insoluble heat resistant mineral filler.

12. A roofing according to claim 11 wherein the thickness of said sheet-like base constitutes not more than about 30% of the thickness of said sheet-like base plus the thickness of said layer of bituminous material.

13. A roofing according to claim 11 wherein said fibrous sheet-like base has a percent elongation at 77° F. not greater than about 5%.

14. A roofing according to claim 11 wherein the fibers contained in said sheet-like base consist in major proportion by weight of glass fibers.

15. A roofing according to claim 11 wherein the heat-resistant binder material by which the fibers in said sheet-like base are co-bonded is peptized substance of said fibers hardened in situ to bond contacting fibers in the sheet-like base together.

16. In a method of making roofing the steps comprising depositing fibers a major proportion by weight of which are over 1/2 inch in length in essentially individual contacting arrangement to form a web, applying a heat-resistant binder to said web, said web with said binder applied thereto being from about 0.005 inch to 0.1 inch in thickness, having a fiber web Densometer value not greater than 50 seconds, and having a tensile strength of at least 10 pounds per linear inch of width, and thereafter impregnating said web with bituminous material containing at least 25% by weight of a finely-divided solid water-insoluble filler having a softening point above 500° F. with incorporation of said filler in at least said percentage of said impregnating material within the body portion of said web and spreading a layer of bituminous material containing at least 30% by weight of a finely-divided solid water-insoluble filler having a softening point above 500° F. on said impregnated web while said bituminous material is in a heat liquefied condition as a weather-proofing coating.

17. A roofing comprising a fibrous sheet-like base impregnated substantially throughout with bituminous material and coated with a weather-proofing coating layer of bituminous material in-

tegrally bonded with said sheet-like base, said fibrous sheet-like base being about 0.01 to about 0.03 inch in thickness and consisting in major proportion by weight of fibers at least $\frac{1}{2}$ inch in length which are disposed in open-textured essentially individual contacting relation, said fibers in said sheet-like base being bonded together by a heat-resistant binder which has a softening point substantially higher than the bituminous material that impregnates said sheet-like base and which per se imparts to said sheet-like base a tensile strength of at least 10 pounds per linear inch of width, said sheet-like base with said fibers thereof bonded by said heat-resistant binder having a fiber web Densometer value not greater than about 50 seconds, said bituminous material that impregnates said sheet-like base containing a finely-divided solid water-insoluble heat resistant mineral filler which constitutes at least 25% by weight of said bituminous material that impregnates said sheet-like base and is within the body portion thereof, said coating layer being of substantially greater thickness than said sheet-like base, and said bituminous material of said coating layer comprising bitumen which has a softening point of from 80° F. to 275° F. and which constitutes from 20% to 70% by weight thereof and comprising from 30% to 80% by weight of finely-divided solid water-insoluble filler which has a softening point above 500° F. and which comprises heat-resistant mineral filler the ratio of the percent by weight of which to the percent by weight of the bitumen in said bituminous material is at least 1 to 3.

18. A roofing according to claim 17 wherein the fibers of said fibrous sheet-like base consist essentially of glass fibers and wherein the fiber of said

sheet-like base as bonded by said heat-resistant binder has a percent elongation at 77° F. of less than about 5%.

19. A roofing according to claim 17 wherein said sheet-like base consists essentially of glass fibers bonded with a heat-resistant synthetic resin.

20. A prepared roofing material according to claim 1 wherein said weather proofing layer of bituminous material contains finely-divided heat resistant mineral filler the ratio of the percent by weight of which to the percent by weight of bitumen in said bituminous material is at least 1 to 3, wherein the thickness of said water-proofing layer of bituminous material is substantially greater than the thickness of said sheet-like base, and wherein said sheet-like base has a tensile strength of at least 10 pounds per linear inch of width and has a percent elongation at 77° F. not greater than about 5%.

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