

[54] **ENCAPSULATING COMPOUND AND CLOSURE**

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[57] **ABSTRACT**

Articles, and particular telephone splices, are protectively encased in a material consisting of a low viscosity oil gelled by a block copolymer such as styrene-isoprene-styrene. Addition of polyethylene to the blend reduces the yield shear stress in the gelled compound and also advantageously supplies a desirable high temperature property of nonslump. Component ranges are described. Stratifying the compound within the closure so that the portion contacting splices is soft and plastic while the remainder is stiff and hard, meets a stringent encasement requirement while minimizing oil separation at high operating temperatures.

6 Claims, 4 Drawing Figures

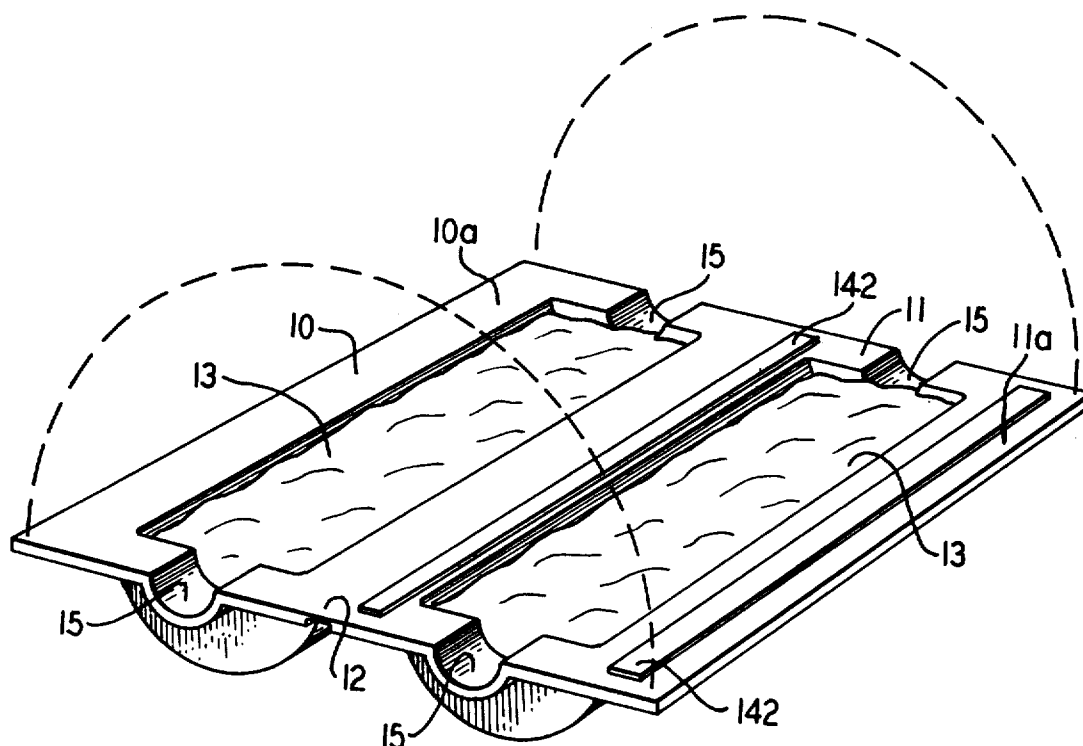


FIG. 1

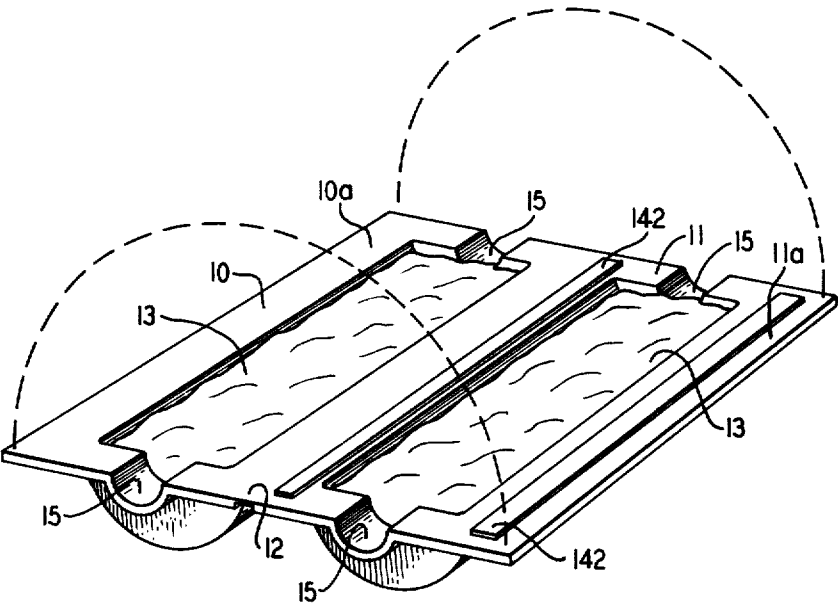
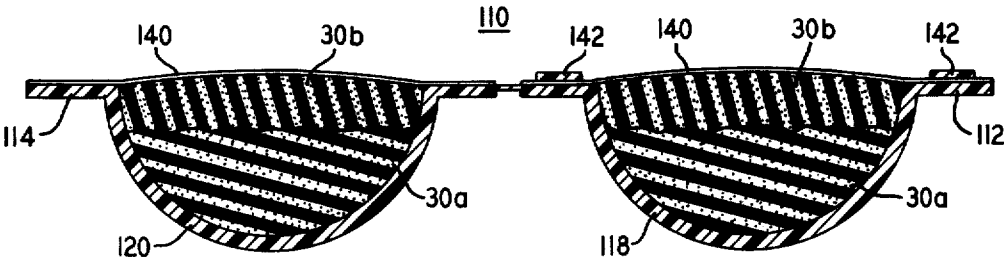


FIG. 3



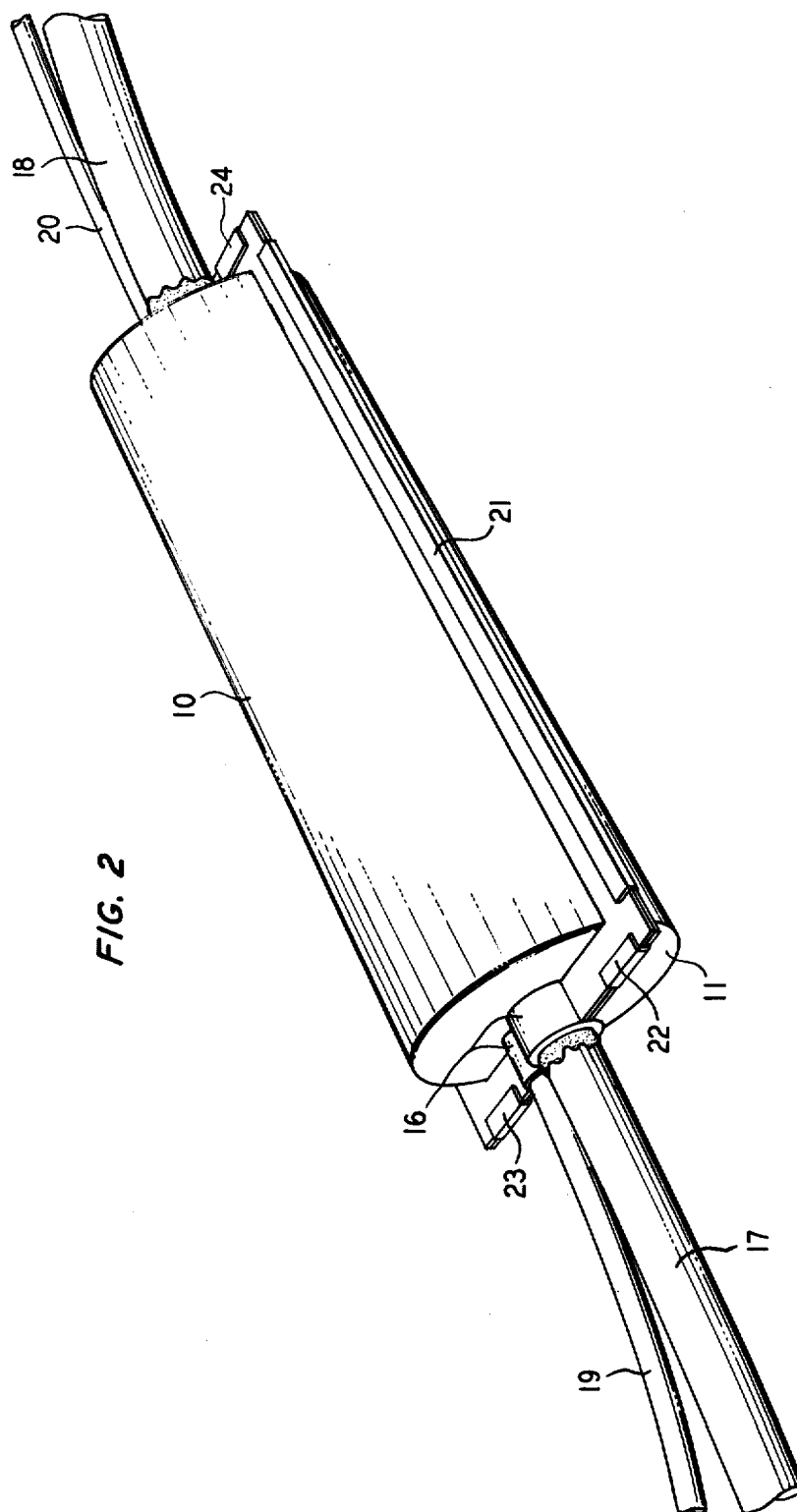
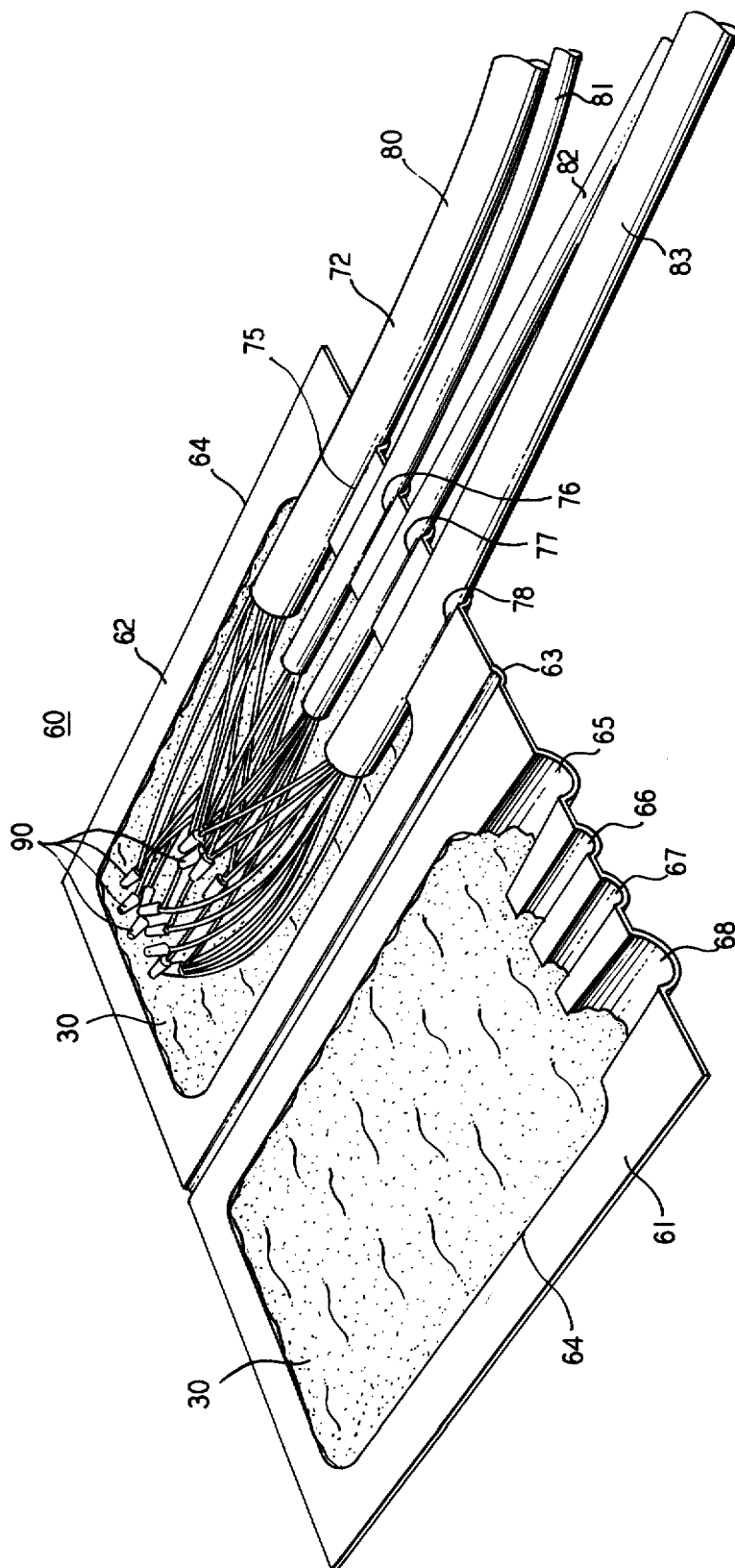


FIG. 4



ENCAPSULATING COMPOUND AND CLOSURE**FIELD OF THE INVENTION**

This invention relates in general to protection of articles from hostile ambients by encasing them in a protective fill. In an important specific application, this invention relates to moisture-proofing compounds for protecting electrical conductor splice points. Still more specifically, the invention relates to a system for protecting a multiplicity of telephone conductor splices and includes a class of waterproofing compounds predisposed in a closure for fast application in the field to splice points. In another aspect, the invention relates to a class of compounds for field pumping into closures around splice points.

BACKGROUND OF THE INVENTION

It is well known that one of the largest causes of telephone service disruption and degradation is the entry of water into or around the electrical connections at a cable splice point. Many systems are used to combat the problem, including encapsulating splicing connectors, elaborate and supposedly waterproof closure designs to envelop the splice points, and circulating a drying gas within the splice region.

More recently, systems of waterproof filler for telephone splice closures are finding use. The purpose of the filling material is to envelop the entire splice point in a waterproof mass, without in the process entrapping any liquid water. Certain currently used materials form nonreenterable plugs. A compound which allows repeated reentry of the closure and access to the splice point is more useful, however.

Design of a fully acceptable waterproof filling compound is not just a matter of assuring its dielectric and physical compatibility with the metal and plastic of the insulated conductors and splicing connectors. Account must also be taken of the temperature-dependent properties and the elasticity of the material. For example, the flow temperature of the filler predisposed in the closure should be at least greater than 140°F and should ideally be greater than 150°F. This high flow temperature is needed to assure that under extreme field conditions the filler will not slump or leak out of the closure (since these rarely are reliably liquid-tight).

In successfully applying the general invention to protecting telephone splices, another important consideration has been discerned. Applying the two factory-prefilled halves of the splice closure to a splice in the field, involves bringing the two halves together around the splices. In this process it is essential that the compound "knit" or inelastically flow around each individual connector splice, without becoming elastically strained. There must be no elastic force buildup in the compound that would resist a full enveloping of each splice.

A prime inventive object, therefore, is to realize a soft encapsulating material that inelastically flows around splices over a wide temperature range.

A further principal object of the invention is to realize a waterproofing compound that has substantially the temperature characteristics defined in the preceding paragraphs.

A general inventive object is to more effectively protect electrical splice points from the corrosive effects of water, particularly telephone conductors at splice

points where the corrosion can degrade or eliminate telephone service.

Another general inventive object is to provide an electrically and chemically inert flowable material that will protect articles from many adverse ambients.

SUMMARY OF THE INVENTION

In a broad sense the invention involves a protective encasing material consisting of a low viscosity oil gelled by a block copolymer.

Advantageously for telephone cable splices, the oil to be gelled is a mineral oil because of its low viscosity at ambient temperatures (360 in SUS units), and its low pour point of about 0°F to -15°F. Also, mineral oil is a paraffinic/naphthenic material and therefore will not stress crack splicing connectors which increasingly are made of polycarbonate.

Pursuant to an important aspect of the invention, the filling material is gelled using a block copolymer composed of styrene-isoprene-styrene (hereinafter called SIS).

SIS when used alone in mineral oil in the range 0.5 to 5.0 weight percent of the final product creates a substance ranging from an elastic fluid to an elastic semi-rigid gel.

However, pursuant to this invention the undesirable elastic properties of the foregoing are reduced by a blend of mineral oil, SIS, and polyolefin wherein the SIS is present in from 0.5 to 5.0 weight percent of the total produce and the polyolefin is present in from 6.0 to 15.0 weight percent of the total product. The polyolefin performs the further desirable functions of substantially reducing the yield shear stress in the final gelled compound and also supplying high temperature stability or resistance to slump up to substantially 150°F.

An advantageous encapsulating gel within the above system consists of mineral oil with from 1.5 to 3.0 weight percent of SIS and from 10.0 to 13.25 weight percent polyolefin stated in weight percent of the total product. Compositions within these ranges exhibit gel stability up to temperatures of 140°-160°F., as well as substantial nonelasticity.

A preferred encapsulating gel consists of mineral oil, SIS and polyolefin in which the SIS is present in from 2.0 to 2.25 weight percent of the total product and the polyolefin is present in from 13.0 to 13.25 weight percent of the total product. Compositions within these ranges are most desirable for nonelastic character and high temperature slump resistance.

Within the broad invention, an inelastic gel which is sufficiently fluid to be pumped at temperatures near 0°F is achieved in a blend of mineral oil, SIS and polyolefin in which the SIS is present in from 1.5 to 2.0 weight percent of the final product and the polyolefin is present in from 6 to 8 percent of the final product.

A specific preferred encapsulant composition pursuant to the invention, which exhibits both a high degree of inelasticity and substantial temperature stability, consists of 84.5 weight percent mineral oil, 2.25 weight percent SIS and 13.25 weight percent polyethylene.

A specific preferred encapsulant composition useful for field filling closures at temperatures near 0°F, consists of 92.0 weight percent mineral oil, 2.0 weight percent SIS and 6.0 weight percent polyethylene.

The preferred polyolefin is polyethylene because of its ready processability. Either high density or low den-

sity polyethylene is usable to substantially equal advantage in the invention. The most advantageous number average molecular weight range within which the polyolefin component of the present invention lies is substantially from 1,000 to 15,000. A preferred range for ease of blending is from 1,000 to 5,000. More specifically, a preferred range for blending high density polyethylene is from 2,000 to 5,000.

The SIS component of the fill desirably is constituted of at least two styrene blocks per molecule. The molecular weight distribution advantageously, although not necessarily, falls within the range of 1-2. The weight average molecular weight of the styrene unit advantageously falls within the range of 5,000 to 10,000. The weight average molecular weight of the isoprene unit advantageously falls within the range of 40,000 to 60,000. Desirably, the isoprene unit weight average molecular weight is substantially 50,000. Also advantageously, on a weight-percent basis, each isoprene block of an average SIS molecule is about 60%, and each styrene block is about 20%.

White mineral oils are a complex mixture of paraffin and naphthene hydrocarbons which, for use in the present invention, preferably include approximately 18 to 36 carbon atoms with the hydrocarbons substantially saturated. Importantly these oils undergo no phase changes in the 0°F through 160°F temperature range of present interest.

Pursuant to a further aspect of the invention the yield shear stress of the gels is further reduced by preshearing the materials. The original properties may be recovered, if desired, by reheating the presheared material above about 194°F.

Pursuant to another aspect of the invention, the requirement of storability without leakage at 140°-160°F is met concurrently with the requirement of "knit-around" when the compound is applied to splices, by stratifying the compound within the closure. With stratifying, the portion of the compound contacting the splice is made relatively soft and plastic, while the remainder is stiff and hard. The softening of the material is achieved by subjecting it to shear forces, which break down some of the bonds creating the gel.

The invention and its further objects, features, and advantages will be made more readily apparent by the descriptions to follow of illustrative embodiments.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a schematic perspective diagram of a closure suitable for receiving filling material of the invention;

FIG. 2 is a side schematic view showing the closure of FIG. 1 installed on a cable or cables;

FIG. 3 is a sectional front view of a closure similar to that of FIG. 1; and

FIG. 4 is a further variety of closure suitable for use with the filling material of the invention.

DETAILED DESCRIPTION OF ILLUSTRATIVE EMBODIMENTS

The invention will first be described by way of several illustrations of materials systems consisting of various amounts of mineral oil, SIS, and polyolefin (principally low density polyethylene, and their preparation).

Example 1

A blend of 85 parts by weight mineral oil, 2 parts by

weight SIS and 13 parts by weight low density polyethylene with appropriate stabilizer was produced. The polyethylene had a number average molecular weight of substantially 2,000. Because the isoprene units will oxidatively degrade by chain scission, the blending temperature was kept between 316°F and 364°F. Due to the compatibility of the materials, simple stirring with magnetic bars was sufficient to bring all materials into solution. On cooling substantially below the glass transition of polyethylene, a gel was formed. The ability of the blend to remain cohesively together under its own weight was tested. A 300 gram sample of the blend was poured hot into a 400 ml circular beaker. The sample was allowed to cool for a period of 17 hours after which its temperature was about 70°F. The sample was then turned upside down and exposed to a temperature of about 140°F for 48 hours. During this period mineral oil separation totaling less than 1 percent of that originally present was measured, thus establishing the ability of the blend to resist slumping.

Example 2

A composition consisting of 84.5 parts by weight mineral oil, 2.25 parts by weight SIS, and 13.25 parts by weight polyethylene was made by the procedure of Example 1. A gel having substantially the same characteristics of Example 1 was obtained.

Example 3

A blend of 85 parts by weight mineral oil, 3 parts by weight SIS and 12 parts by weight polyethylene was made by the method of Example 1. A sample was applied to a test splice connector and visually found to exhibit only slight—and for most purposes insignificant—drawback, signifying acceptable inelasticity. Slump temperature properties were observed to be substantially as found in the Example 1 composition.

Example 4

A blend of 85 parts by weight mineral oil, 5 parts by weight SIS and 10 parts by weight polyethylene was prepared by the method of Example 1. The blend exhibited no significant temperature slump dissociation until the 140°-160°F range. Its inelasticity was marginally acceptable at 70°F. By hot-melt encapsulation—that is, heating the blend to about 390°F and pouring the blend over the article to be encapsulated, and then allowing the blend to gel on cooling—drawback resulting from the relative elasticity is greatly minimized or eliminated.

Example 5

A blend of 87-3-10 parts by weight mineral oil, SIS, and polyethylene respectively was prepared by the method of Example 1, cooled to about 70°F and applied to a test connector. Its inelasticity was observed to be acceptable. On heating, no significant temperature slump was encountered until the range of 140°-160°F. Use of this mix as a hot-melt encapsulant is preferred.

Example 6

A blend of 86.5-3.5-10 parts by weight mineral oil, SIS, and polyethylene respectively was prepared as in Example 1 and found to exhibit substantially the same characteristics as did the Example 5 blend. Use as a hot-melt encapsulant is preferred.

Example 7

A blend of 85.5-1.5-13 parts by weight mineral oil, SIS, and polyethylene respectively was prepared pursuant to the method of Example 1. The blend exhibited the desirable high temperature properties of the Example 1 blend.

Example 8

A blend of 84.5-0.5-15 respective parts by weight of mineral oil, SIS, and polyethylene was produced by the method of Example 1. The resulting composition exhibited highly desirable temperature stability to about 160°F. The observed elasticity was considered acceptable for certain encapsulation jobs. Hot-melt encapsulation renders the blend acceptable for a wider range of applications.

Example 9

A blend consisting of 92.0-2.0-6.0 parts by weight respectively of mineral oil, SIS, and polyethylene was produced as in Example 1. The resulting composition on cooling to 70°F was acceptably inelastic. A sample of the blend was placed in a syringe and brought down to a temperature of 0°F. The material was easily extruded from the syringe, without undergoing oil separation or gel breakdown due to shearing. This material is preferred as a fill to be applied to splice closures by crewman working in the field during colder weather.

Example 10

A blend consisting of 92.5-1.5-6.0 parts by weight respectively of mineral oil, SIS, and polyethylene was produced as in Example 1. The resulting composition exhibited somewhat more softness than that of Example 9, connoting a reduced but still acceptable gel-producing pseudo-network. The product was easily pumped at 0°F. Temperature stability up to substantially 140°F is obtainable.

Example 11

A blend consisting of 90.0-2.0-8.0 parts by weight respectively of mineral oil, SIS, and polyethylene was produced as in Example 1. The resulting composition exhibited a more desirable degree of inelasticity than the products of Examples 9 and 10. Its pumpability at 0°F was demonstrated. Its temperature stability was acceptable up to substantially 150°-160°F.

Experiments were made with blends of mineral oil and polyethylene respectively without SIS. The resulting compositions significantly lacked gel stability. Moreover, the compositions exhibited drastic shear thinning (as, for example, when pumped or molded around a splice), with insignificant recovery following termination of the shear.

Experiments were also made with blends of mineral oil and 0.5-5.0 weight percent SIS, without polyethylene. The resulting gel at all points exhibited at least an undesirable degree of elasticity, and an undesirable narrow temperature stability range. The relative acceptability of gel-type encapsulants as a function in part of gel elasticity, is a factor not heretofore considered in any depth by designers of encapsulants for electrical splices. The factor is assessable in a number of ways, including: microscopic measurement of "drawback" of a given encapsulant from a given splice configuration; visual inspection of drawback in a given

case, and measurements on the materials themselves from which an extrapolation as to elasticity factor may be made.

Experiments involving the last-noted method were made by hot-casting of samples of the following blends in a syringe, and then noting the volume percent swell when each is extruded from the syringe.

Material			Volume Percent Swell
Mineral Oil	SIS	Polyethylene	
85.0	5.0	10.0	64
86.5	3.5	10.0	57
87.0	3.0	10.0	43
85.0	3.0	12.0	35

The data suggests the variability of elasticity with differing blends. It should be recognized, however, that overall acceptability of a given gel is also a function of its temperature stability properties.

Mineral oils suitable in the above formulations include Drakeol 35 from Pennsylvania Refining Co. and Pentol from Witco Chemical. In general, mineral oils are all similar in composition; but some contain impurities and unsaturated hydrocarbons, and vary in odor and color. Many of the unsaturated hydrocarbons are aromatic and these may cause stress cracking of polycarbonate, for example. For this reason the use of an unsaturated mineral oil is some applications of the invention would depend on its stress-cracking activity. White mineral oil is a preferred material, however, for its freedom from stress cracking.

An antioxidant stabilizer such as Irganox 1010 available from Ciba-Geigy, or Ionol from Shell Chemical of the hindered phenol type has been found desirable. The addition of about 0.1 weight percent of stabilizer to the compounds of Examples 1-11 is desirable.

Closure Structure

A closure which is generally suitable to receive the preferred compounds described above, is depicted in FIG. 1 as consisting of an upper half 10 and a lower half 11, advantageously sharing a common hinge 12. The fill material designated 13 is placed either in the factory, or where appropriate in the field, within each of the halves. The outer edges of the halves 10, 11 are formed as an extending lip designated 10a for the upper half and 11a for the lower half. To accommodate telephone cables to be spliced, the lips 10a, 11a are provided with a throat section formed by semicylindrical portions denoted 15.

A variation of the closure shown in FIG. 1 is depicted in FIG. 2 as having a dual diameter neck portion denoted 16, to accommodate, for example, cables 17, 18 and wires 19, 20.

The assembling of either of the closures shown in FIG. 1 and FIG. 2 is facilitated by use of the spring clips such as 21, 22, 23, and 24 which grasp and secure the outwardly extending edges of the upper and lower halves.

In order to meet the requirement of storage at temperatures at least up to 140°F without leakage for those closures filled in the factory, the compound must have substantially strong bonds which in turn may cause it to be harder or more solidified at normal working temper-

atures than is desired. To the extent the compound is elastic, it will not flow as readily into all the interstices about the splices when the closure is sealed about the splice joint. A method has been discovered of solving the foregoing problem, by stratifying the compound within the closure. The portion of the compound in intimate contact with the splices is made relatively soft and flexible and thus can be easily redistributed to fill all interstices thereabout. The remainder of the compound in the closure is made stiff and thus provides added strength to prevent leakage at the high storage temperatures.

One way of softening or deelasticizing the gels is to release the yield shear stress by preshearing of the material. This can be achieved by any mechanical working of the material. Experiments have also revealed that the original properties are recovered by a reheating of the sheared material in the region of approximately 190°F.

FIG. 3 is a cross-sectional view through the midsection of a closure 110 in an open configuration showing a stratified formation of the compound therein. A first layer or stratum 30a of compound 30 having a relatively rigid structure to provide stiff support at the high storage temperatures and to inhibit the movement of the conductor splices out to the walls of the cover, is formed in the bottom of cavities 118 and 120 of cover halves 112 and 114, respectively. A second relatively soft layer or stratum 30b of compound 30 is formed over layer 30a. The splices lie in the compound 30 so that when closure 110 is sealed about the splice joint, the relatively soft layer 30b is easily redistributed about the splices filling all interstices at the parting line of the closure 110. A sheet of separator material 140 lies over layer 30b and temporary separator blocks 142 or a separator frame can be placed thereon for shipping and to allow slight overfilling of the closure if desired. The separator is removed at installation.

The relative thicknesses of layers 30a and 30b depend upon such factors as the size of the specific closure being utilized and the number of splices to be placed therein and hence the quantity of relatively soft flexible compound required to fill all interstices. The maximum thickness of layer 30b is set by the mechanical properties of the sheared material. The thickness of 30a simply fills the rest of the cavity. When the thickness of the layer 30b is near the maximum allowable thickness, the throat section of the closure, such as designated 15 in FIG. 1, advantageously can be filled with the relatively stiffer material comprising layer 30a in order to help support the softer less viscous material in layer 30b when the closure is stored on end, i.e., in an upright position.

The formation of strata or layers in a compound having a uniform composition throughout, but having different yield shear stresses for the various strata, is accomplished by forming the strata by different mechanical processes. For example, if the composition is subjected to the previously described shearing pressures or forces such as those encountered when the material is extruded through a nozzle, the yield shear stress thereof is decreased because some of the bonds creating the gelled condition are ruptured. This phenomena is utilized in forming the strata, such as 30a and 30b. For example, stratum or layer 30a comprises a layer of compound 30 which is hot-poured into the closure while the compound 30 is in a liquid state. When layer

30a cools, it gels or solidifies to form an extensive internal bonding network which results in a relatively stiff strong layer. Layer 30b comprises a layer of compound 30 which is extruded through a nozzle at room temperature thereby shearing and rupturing a portion of the bonds and resulting in a relatively soft, that is, low yield shear stress layer which can be easily forced into interstices. The shearing pressure applied to the compound of layer 30b is determined by the nozzle size, rate of extrusion, etc., and will depend upon such factors as the composition being utilized and the relative degree of low yield shear stress required in layer 30b.

FIG. 4 illustrates a further variety of spliced closure constructed pursuant to this aspect of the invention. The closure denoted 60 consists of two halves 61, 62, advantageously joined by a common hinge 63, and including extended edge portions 64 on each half which abut when the closure is sealed. The edge portions 64 of the closure 60 are provided with semicylindrical throat portions 75, 76, 77, 78 to accommodate incoming and outgoing cables 80, 81, 82, and 83. The numerous splice mechanisms, denoted generally as 90 in FIG. 4 are merely symbolic of the type of splice connector that can be used in this closure. The splices 90 are shown as completed and ready to be embedded and encased in the fill 30.

The spirit of the invention is embraced in the scope of the claims to follow.

What is claimed is:

1. In combination: a plurality of insulated conductor splice points enveloped in a waterproof encapsulating agent comprising from 84.5 to 92.5 parts by weight of mineral oil, from 0.5 to 3.0 parts by weight styrene-isoprene-styrene block copolymer, and from 6.0 to 13.0 parts by weight of polyethylene having a weight average molecular weight above 2000.
2. A closure receiving and encapsulating therein a plurality of spliced insulated conductors, comprising:
 - a lower shell and an upper shell mating along extended flat edge portions and each shell having a recess,
 - a plurality of spliced electrical conductors disposed between said shells
 - the recess of each said shell containing an encapsulating agent enveloping said spliced electrical conductors, and comprising a blend of from 84.5 to 92.5 parts by weight of mineral oil, from 0.5 to 3.0 parts by weight of styrene-isoprene-styrene block copolymer, and from 6.0 to 13.25 parts by weight of a polyolefin.
3. A closure pursuant to claim 2, further comprising elongated clip means fastening said upper and said lower shells together along their respective extended edge portions.
4. A closure pursuant to claim 3, wherein the parts by weight of said mineral oil are in the range of 84.5 to 87.0, the parts by weight of said styrene-isoprene-styrene block copolymer are in the range 1.5 to 3.0, and the parts by weight of said polyolefin are in the range 10.0 to 13.25.
5. A closure pursuant to claim 4 wherein said agent contained in each said mating shell is stratified into a first layer contacting said electrical splices, and a second layer filling the remainder of said shell, said first layer adapted to be rendered relatively soft by subjecting the same to shearing motion and said second layer

being relatively rigid providing stiff support and protection for said first layer.

6. An article of manufacture useful as a closure for protecting a plurality of spliced insulated conductors of a telephone cable comprising:

a lower shell and an upper shell having flat outer edges formed as a generally rectilinear extending lip, said lower half being joined to said upper half along a common hinge formed by a connection of a lip edge of said first half and a corresponding lip edge of said second half,

an encapsulating agent substantially filling said lower half and said upper half and comprising:

a blend of from 84.5 to 92.5 parts by weight of min-

eral oil, from 0.5 to 3.0 parts by weight of styrene-isoprene-styrene block copolymer, and from 6.0 to 13.25 parts by weight of a polyolefin, the said agent being stratified into a first layer located substantially at the level of said edges for contacting said splice, and a second layer filling the remainder of said lower half and said upper half, said first layer adapted to be rendered relatively soft by subjecting the same to shearing motion, and said second layer being relatively rigid to supply stiff support and protection for said first layer, and a sheet of separator material placed over said first layer.

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