Dec. 10, 1963 S. J. SPURGEON ETAL 3,113,881

ELECTRICAL CONTACT SEALING COMPOSITION

Filed June 21, 1960

Fig. 1.

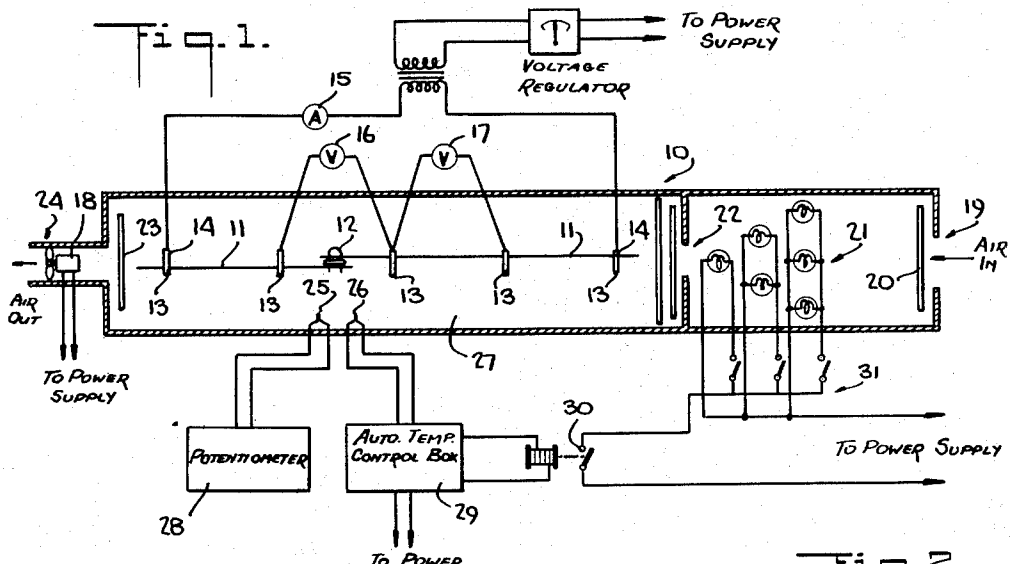


Fig. 2.

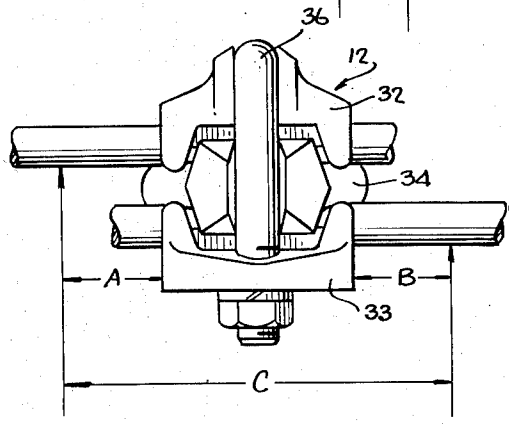


Fig. 3.

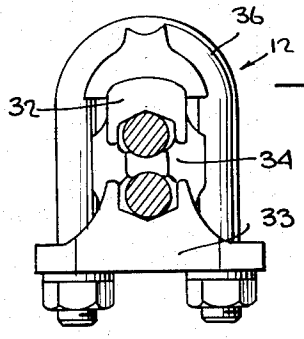
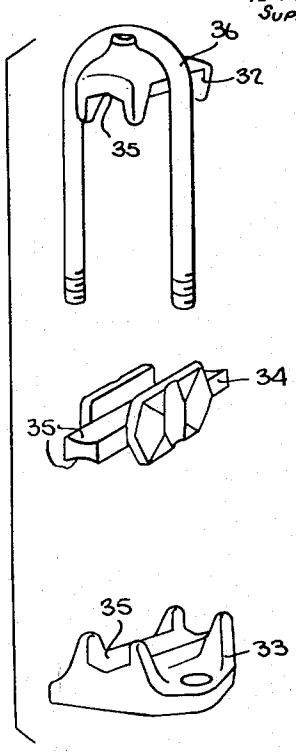


Fig. 4.



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3,113,881 ELECTRICAL CONTACT SEALING COMPOSITION

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Filed June 21, 1960, Ser. No. 37,670
8 Claims. (Cl. 106—287)

This invention relates to a method and composition of matter particularly adapted for use in connection with electrical connectors of the bolted or compression type or the like to effect improved electrical contact between the conductors and the connector, and to maintain such improved contact for an indefinite period of time.

Heretofore the need for a good contact sealant particularly adapated for use with electrical compression fittings of aluminum and its alloys has been manifest. For many years various means have been tried by those skilled in the art to provide better interconnection between the electrical conductors and the splicing connector. As is well known, aluminum oxidizes easily and rapidly when exposed to the atmosphere and the oxide film which thus forms on the surfaces of an aluminum electrical connector, as well as on the surface of the electrical conductor being used, particularly when it is also composed of aluminum, presents a barrier, i.e. resistance, to an electric current passed through the connector and interconnected conductors, thereby causing a power loss and diminution in economies. In the past it has been necessary to clean the oxide film carefully from the conductors and the clamping surfaces of the connector by various means such as brushing or other abrasive techniques, and in all instances the cleaned surfaces would become partially or even entirely re-oxidized by the time that assembly of the splice was completed.

In an effort to overcome this problem in the past, the connectors were designed with axial ridges or knife edges which, upon compression of the clamping surfaces of the connector onto the conductor, caused the connector to bite into the conductor and thus cause the oxide film to be cut away along a portion thereof. However, such efforts did not satisfactorily solve the problem and with the latter type of connector, as well as with other compression types, some sort of sealing compound was employed not only to aid in cleaning the contacting surfaces, but also to inhibit or prevent re-oxidation of such surfaces during subsequent use.

Such prior sealing compounds have been, for example, a silica-aerogel thickened grease containing metallic particles, and have relied on the abrasive character of the composition to perform a mechanical cleaning function. Such compositions in use, however, have proved to have many drawbacks and to be in general rather unsuccessful. Furthermore, these prior compounds have not been sufficiently stable either electrically or chemically, neither have they exhibited sufficient resistance to degradation at the high temperature encountered during use or to penetration by moisture.

It is an object of the present invention to provide an electrical contact sealing compound particularly adapted for use with aluminum electrical connectors which effectuates improved electrical contact between the conductors and the connector.

Another object is to provide such a composition which markedly lowers the electrical resistance of the electrical connection, increases the temperature which such connection will withstand without an increase in resistance due to oxidation, and increases the oxidation resistance of such connection with an increase in the useful life thereof.

A further object is to provide such a composition which is electrically, chemically and thermally stable, and exhibits a particular affinity for aluminum oxides, forming a chemically stable complex with such oxides and removing the same from metal surfaces with a minimum of mechanical abrasive force.

Still another object is to provide such a composition which prolongs the life of electrical connectors for power utility transmission and distribution systems.

Another object is to provide a contact sealant composition for use with aluminum electrical connectors, which wets and adheres to the metal of the connector and enables substantial removal of the aluminum oxide film therefrom during assembly of the conductors with the connector.

Briefly, the composition of the present invention comprises a major proportion of an oxidation and water-resistant high temperature dispersoid and a minor proportion of a chelating agent having particularly affinity for oxides and hydrated oxides of aluminum, such that there is the formation of stable coordination complexes between the chelating agent and the aluminum oxide species. One typical embodiment of the invention comprises a neutral methyl siloxane polymer base or vehicle e.g. a silicone grease comprised of dimethyl silicone fluid and a silica filler having the following characteristics:

Dropping Point, ° C.	Bleed, Percent [1]	Evaporation, Percent [1]	Useful Temp. Range, ° C.
None	1.0	1.5	−50 to 200

[1] Bleed and evaporation determined after 24 hours at 150° C. in a wire mesh cone. See vol. 12, Encyclopedia of Chemical Technology, page 405.

Added to such vehicle is 8-hydroxyquinoline. ("Neutral" as used herein in reference to said base or vehicle means that the base or vehicle, prior to admixture with the chelating agent, contains no constituent which will react with the connector or the conductor, or which will alter the physical properties of the siloxane.) Such composition can be used in the usual compression-type electrical connector as a contact sealant and is found to result in a metal-to-metal contact of very low resistivity which will be found to persist upon exposure to the severest atmospheric conditions for an indefinite period of time.

Other objects and features of the invention will become apparent in the following specification and claims, and in the drawings, in which:

FIG. 1 shows a simplified schematic diagram of the electrical circuit used for test purposes;

FIG. 2 shows a side elevation in perspective of an electrical connector used in tests;

FIG. 3 shows an end elevation of the connector of FIG. 2; and

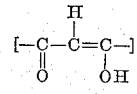
FIG. 4 shows an exploded perspective of the parts of the connector.

Compositions according to the present invention may contain a variety of constituents. In general, such composition comprises a vehicle and at least one additive. The vehicle is generally a base such as a thickened silicone fluid, i.e. a siloxane, but may also be a fluo-carbon type grease, or a hydrocarbon grease having a thickener which will not react with or be dissolved by water, and which will be otherwise chemically and electrically stable under the conditions of use. In other words, the vehicle is generically a non-aqueous dispersoid of a hydrocarbon or a substituted hydrocarbon grease composition. The silicone-type greases may be preferable in that they retain body with an increase in temperature rather than slump or flow, the latter sometimes being a characteristic of other types of greases.

In summary, the essential properties of the vehicle are that it should:

(1) Wet and adhere to the metal surfaces
(2) Be non-corrosive
(3) Be stable to air oxidation
(4) Be stable at the high temperatures encountered during use in an electrical system
(5) Be electrically stable, i.e. not subject to an electric current through it
(6) Not interfere with the formation of the chemical complex between the additive and the aluminum oxide film.

The additive in general can be a monohydroxy substituted quinoline such as 8-hydroxyquinoline, a polyhydroxy substituted benzene such as pyrogallol or catechol, diones such as 2,4-pentanedione, substituted 2,4-pentanediones, e.g. 1,1,1-trifluoro-4-(2-thenoyl)-2,4-butanedione, 1-phenyl-1,3 butanedione, or 1-(2 furyl)-1,3-butanedione, or ethylenediaminetetraacetic acid or 1,2 diamino cyclohexane tetraacetic acid. The dione compounds can be any of a rather broad class of compounds having as the functional group:



The substituents on the end carbon valences can be methyl, ethyl, thenoyl, phenyl, beta-naphthoyl, or fluor-substituted methyl, and the substituted groups may be the same or different on each dione molecule, except that only one such group may be fluorosubstituted methyl.

The essential properties of the additive are:

(1) It must be able to react or combine with the aluminum oxide film in the manner hereinafter described
(2) The oxide film-additive complex must be somewhat soluble in or somewhat dispersible in the vehicle such that during the squeezing of the connector or conductors, the vehicle which is thereby extruded will carry the additive-oxide film with it
(3) It must retard atmospheric re-entry toward the metal contact surfaces for extended periods of time.

While the phenomenon exhibited by the composition of the present invention is not entirely understood, it is believed that the above described additives react or combine with the aluminum oxide layer, breaking the continuity of the layer or film by the formation of a chelate-type of complex of the inner-salt type, which is characterized by non-ionic properties and solubility in organic-type solvents. Thus it is believed that a coordination complex of the chelate-type is formed between the aluminum oxide and, for example, the 8-hydroxyquinoline, which forms a stable molecule and permits removal of the oxide from the metal surfaces upon disturbance thereof by migration of the grease in response to clamping action of the connector.

According to the present theory the inert vehicle or carrier, e.g. siloxane, as a medium for the reaction, must possess a group or substituent such as an oxygen atom located between the silicon and the carbon in the siloxane molecule, so that such medium is able to perform a nucleophilic attack on the protons of the hydroxyl groups in the additive, e.g. 8-hydroxyquinoline, to such an extent that such protons are partially removed from the 8-hydroxyquinoline, thereby enabling the remaining electrons on the oxygens in the 8-hydroxyquinoline to be donated to the vacant orbitals of the aluminum ion in the aluminum oxide molecule. In the case of greases containing soaps, it is possible that the oxygen atoms in the soap constituent provide the group necessary for the above described nucleophilic attack.

According to the above theory the molecular energy of the coordination complex is at a lower level, i.e. a more chemically stable state, than the molecular energy of the uncoordinated additive molecule, and as a consequence the electrons of the oxygen have two rings to orbit around, i.e. one benzene ring of the 8-hydroxyquinoline, and the coordination ring, and thus the aluminum of the aluminum oxide molecule is held more firmly and results in relatively easy removal of the oxide film from the metal surface of the connector and of the conductors.

Furthermore, since the silicone molecule of the base has an irregular shape it tends to wrap up the aluminum oxide-additive coordination complex and facilitate the removal of the oxide film from the metal surfaces.

In order to evaluate various compositions according to the present invention, test apparatus was assembled as shown in the drawings FIGS. 1–4. Within enclosed housing **10**, a pair of A.W.G. No. 2 solid aluminum conductors **11**, joined by an electrical connector **12** (a type LC 70 B aluminum connector manufactured by Anderson Electric Corporation, Birmingham, Alabama) were mounted in spaced clamps **13**. Regulated A.C. voltage was applied to the conductors at end clamps **14** in order to provide a constant current of 131 amperes as indicated on ammeter **15**. Voltmeter **16** was connected across circuit length C which included the connector **12** and adjoining portions of the conductors as shown (see FIGS. 1 and 2), and measured V_x in the formula below. Voltmeter **17** was connected across a one foot length of one conductor to measure V_r in the formula.

Since the above conductors **11** have a current capacity rating measured at 40° C. (104° F.) the temperature within the housing was maintained at that level by an automatic system consisting of an electric fan **18** which drew air in from one end **19**, around baffle **20**, past a battery of incandescent bulbs **21**, then past baffles **22**, along the conductors **11**, around baffle **23** and thence to exit **24**. The controlled temperature was employed in an attempt to eliminate any variation in results caused by fluctuations in ambient temperature.

Iron-constantan thermocouples **25**, **26** were provided to measure the temperature within the test cell **27**, the former being connected to a potentiometer **28** to permit an instantaneous reading of the temperature, and the latter interconnected with the automatic temperature control box **29** which, through relay **30**, controlled such temperature by cutting in and out the electrical power for the incandescent lamps **21** selected by manually operated switches **31**. The manual selection of the appropriate lamps **21** was made in accordance with the prevailing ambient temperature at the time a particular test was being run.

The LC 70 B connector **12** has top member **32**, a bottom member **33**, and a spacer **34**, all having opposed complementary conductor receiving grooved contact surfaces **35** as shown. A U-bolt **36** interconnects the above elements of the connector and applies the compressive force necessary to clamp the conductors **11** in place against the contact surfaces **35**.

For each test the compounded and modified grease composition was applied during assembly to all contact surfaces **35** of the connector **12** as well as to the corresponding parts of the conductors **11**. The connector resistance was calculated according to the following relation:

$$R_c = R_r(V_x/V_r - 0.85417)$$

where

V_x=voltage drop in connector plus $2x$ inches of conductor
V_r=voltage drop across standard foot of conductor
R_r=resistance of standard foot
R_c=resistance connector

In the above formula R_r, the resistance of a standard foot of the conductor, was known. The readings of voltmeters **16** and **17** provided the additional information needed to calculate the resistance of the connector.

The above test apparatus was used for the following examples.

Example I

85 grams of a silicone grease comprised of dimethyl silicone fluid thickened with a silica filler and having the characteristics described above in column 2, were mixed with 15 grams of 8-hydroxyquinoline and applied to the connector and conductors as previously described. R_c was found to be 40–60 micro ohms and remained substantially constant for an extended period of time. Without the use of a contact sealant the resistivity was found to be about 100 micro ohms and higher, and it increased in time to even higher values.

Example II

85 grams of a silicone grease comprised of dimethyl silicone fluid thickened with a silica filler and having the characteristics described above in column 2, and 15 grams of pyrocatechol were mixed together and similarly found to provide a contact resistivity under test of 40 to 60 micro ohms.

Example III

85 grams of a silicone grease comprised of dimethyl silicone fluid thickened with a silica filler and having the characteristics described above in column 2, and 15 grams of ethylenediaminetetraacetic acid were mixed together and tested and gave a similar contact resistance of 40 to 60 micro ohms.

Example IV

85 grams of a silicone grease comprised of dimethyl silicone fluid thickened with a silica filler and having the characteristics described above in column 2, and 15 grams of 2,4-pentanedione were mixed together and tested and gave a similar contact resistance of 40 to 60 micro ohms.

Example V

85 grams of a silicone grease comprised of dimethyl silicone fluid thickened with a silica filler and having the characteristics described above in column 2, and 15 grams of 1,1,1-trifluoro - 4 - (2-thenoyl)-2,4-butanedione were mixed together and tested and gave a contact resistance of 40 to 60 micro ohms.

In the prior art it was known to use compounds such as pyrocatechol, pyrogallol, and similar materials in amounts ranging from 0.05 to 5% to stabilize organo-siloxanes against oxidation. (See McGregor Patent 2,389,804 issued November 27, 1954.) In order to achieve the purposes of the present invention, the chelating agent must be present in organo-siloxanes in amounts of about 10% to about 15% in addition to the small but effective stabilizing amount taught by the aforesaid prior art. Furthermore the present invention involves the use of other chelating agents undisclosed in the prior art which provide the novel results hereinabove shown.

The composition of the present invention provides a contact sealant having properties long sought in the art and having economical advantages promising wide commercial acceptance.

With the compositions of the present invention it is now possible to obtain markedly lower contact resistivities on electrical connectors and fittings of a wide variety of standard types, and the improved contact resistivity persists over the required useful life of the fittings.

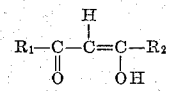
When employing the present contact sealant with aluminum alloy castings or conductors containing other constituents such as silicon or the like, it is possible that in removing the oxide film from the contact surface by the above described technique, random projections of silicon will be left on said surface and acts as undesirable spacers, preventing or tending to prevent maximum electrical contact between said surface and the conductors. In such cases this possibility should be kept in mind, and to obtain optimum results it may be desirable in such cases to use supplementary techniques already known in the art to insure the desired results.

It is to be understood that certain changes and additions can be made to the composition of the present invention by those skilled in the art without departing from the scope and spirit thereof.

This is a continuation-in-part of application Ser. No. 834,457, filed August 18, 1959, now abandoned.

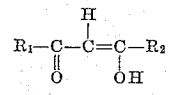
We claim:

1. An electrical contact sealant composition consisting essentially of a compound selected from the group consisting of polyhydroxy-substituted benzenes, monohydroxysubstituted quinolines, ethylenediaminetetraacetic acid, 1,2 diamino cyclohexane tetraacetic acid, and a compound having the structural formula:



wherein R_1 and R_2 each are substituents selected from the group consisting of methyl, ethyl, thenoyl, phenyl, beta-naphthoyl, or fluorosubstituted methyl, and a neutral, oxidation- and water-resistant grease being non-corrosive, adherent to metal surfaces and chemically and electrically stable and usable within a temperature range of from about −50 to about 200° C., said compound present in an amount of from about 10.05 to about 20% by weight, the balance of said composition being said grease.

2. An electrical contact sealant composition consisting essentially of a compound selected from the group consisting of polyhydroxysubstituted benzenes, monohydroxysubstituted quinolines, ethylenediaminetetraacetic acid, 1,2 diamino cyclohexane tetraacetic acid, and a compound having the structural formula:

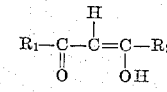


wherein R_1 and R_2 each are substituents selected from the group consisting of methyl, ethyl, thenoyl, phenyl, beta-naphthoyl, or fluorosubstituted methyl, and a silicone grease comprised of dimethyl silicone fluid thickened with a silica filler and having 1% bleed and 1.5% evaporation determined after 24 hours at 150° C. in a wire mesh cone and a useful temperature range of −50 to 200° C., said compound present in an amount of from about 10.05 to about 20% by weight, the balance of said composition being said grease.

3. An electrical contact sealant composition consisting essentially of a silicone grease comprised of dimethyl silicone fluid thickened with a silica filler and having 1% bleed and 1.5% evaporation determined after 24 hours at 150° C. in a wire mesh cone and a useful temperature range of −50 to 200° C., and from about 10.05 to about 20% by weight of 8-hydroxyquinoline.

4. An elecarical contact sealant composition consisting essentially of a silicone grease comprised of dimethyl silicone fluid thickened with a silica filler and having 1% bleed and 1.5% evaporation determined after 24 hours at 150° C. in a wire mesh cone and a useful temperature range of −50 to 200° C., and from about 10.05 to about 20% by weight of 2,4 pentanedione.

5. An electrical contact sealant composition consisting essentially of a silicone grease comprised of dimethyl silicone fluid thickened with a silica filler and having 1% bleed and 1.5% evaporation determined after 24 hours at 150° C. in a wire mesh cone, and a useful temperature range of −50 to 200° C., and from about 10.05 to about 20% by weight of a compound having the structural formula:



wherein R_1 and R_2 each are substituents selected from the group consisting of methyl, ethyl, thenoyl, phenyl, beta-naphthoyl and fluorosubstituted methyl.

6. An electrical contact sealant composition consisting essentially of a silicone grease comprised of dimethyl silicone fluid thickened with a silica filler and having 1% bleed and 1.5% evaporation determined after 24 hours at 150° C. in a wire mesh cone, and a useful temperature range of −50 to 200° C., and from about 10.05 to about 20% by weight of ethylenediaminetetraacetic acid.

7. An electrical contact sealant composition consisting essentially of a silicone grease comprised of dimethyl silicone fluid thickened with a silica filler and having 1% bleed and 1.5% evaporation determined after 24 hours at 150° C. in a wire mesh cone, and a useful temperature range of −50 to 200° C., and from about 10.05 to about 20% by weight of 1,1,1-trifluoro-4-(2-thenoyl)-2,4-butanedione.

8. An electrical contact sealant composition consisting essentially of a silicone grease comprised of dimethyl silicone fluid thickened with a silica filler and having 1% bleed and 1.5% evaporation determined after 24 hours at 150° C. in a wire mesh cone, and a useful temperature range of −50 to 200° C., and from about 10.05 to about 20% by weight of 1,2 diaminocyclohexanetetraacetic acid.

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