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3,013,907

COATING ALUMINUM WITH A SPERM WHALE OIL CORROSION RESISTANT, LUBRICATING COMPOSITION

Norman F. Baird, Bloomfield, and William J. Monohan, Preakness, N.J., assignors to Westinghouse Electric Corporation, East Pittsburgh, Pa., a corporation of Pennsylvania

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4 Claims. (Cl. 117-134)

The present invention relates to metallic parts, such as incandescent lamp bases and, more particularly, to a lubricating and corrosion resistant coating for such a base.

This application is a divisional application of application Serial No. 477,406, filed December 23, 1954, by the inventors of this application and now U.S. Patent No. 2,916,402 issued December 8, 1959.

With the advent of aluminum as an incandescent lamp base metal, a problem arose which threatened the continued use of aluminum in the industry. Within a few weeks after installation, an aluminum based lamp corroded in its brass or aluminum alloy socket to such an extent that it was difficult to remove the lamp therefrom. Within a few months the corrosion had reached the point where the lamp was frozen in the socket and its removal therefrom was practically impossible. In areas having high humidity, acid or similar ionic atmospheres such as salt mist, the incidence of corrosion increased and the corrosion was accelerated.

The corrosion of the aluminum lamp base is believed to be caused by the galvanic action between the aluminum base metal and, particularly, the metal of the socket in an electrolyte present in the atmosphere, such as for example carbonic acid (H_2CO_3), which acid is formed by the combination of water vapor and carbon dioxide in the air.

In addition to the corrosion problem encountered in its use in a lamp base, aluminum has the characteristic property of galling or resisting sliding on another metal surface, such as for example the metal threads on the aluminum base binding on the threads of the metal socket. This galling action produces a noise, characteristic of an unlubricated surface under a dynamic load.

Hence, it has been found desirable to apply a film to an aluminum lamp base which will serve as both a lubricant and as a corrosion resistant coating. Such a coating or film must not affect adherence of the solder to the metallic base when said solder is employed to secure the lead wires of the lamp to the base shell and to the bottom contact of the base.

In addition, adherence of indicia marking ink and basing cement to the base must in no way be affected by the use of the film. Again the coating must not produce a discoloration of the metal.

Since the base holding heads on lamp finishing machines must necessarily mask a portion of the base, while at the same time supporting a base, and since these heads do not necessarily rotate as they index from station, application of such a corrosion resistant coating on a finishing machine is difficult. The added complications in both equipment modifications and process modifications renders impractical the coating of a base at this point in the manufacturing cycle. Hence, it is desirable to coat the base prior to the basing operation.

The use of water as a bulk medium in the existing prior processing of the metal bases, makes it desirable for compatibility reasons to continue the use of water as the bulk medium in the application of the corrosion resistant coat-

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ing to the bases. A concentrate consisting of hydrogenated sperm whale oil (a white solid with a wax-like consistency at room temperature, soluble in toluene and hot trichlorethylene and having a melting point at 126° F.) as solute was dissolved in solvent toluene and mixed with a "Tergitol" wetting agent No. 7.

"Tergitol" wetting agent No. 7 is a trade name for a 25% water solution of sodium heptadecyl sulfate manufactured by Carbide and Carbon Chemicals Co., a division of the Union Carbide and Carbon Corporation. Physically blending this concentrate with water produced an emulsion, a liquid mixture in which the hydrogenated sperm oil is suspended in the water as minute globules covered with the surfactant or wetting agent, "Tergitol" No. 7.

Although this emulsion is permanently stable the toluene is explosively dangerous both in storage and during the application of the emulsion. The hydrogenated sperm whale oil, being a solid, left a discontinuous film on the metal surface. Discontinuities in the protective coating, not seen by the naked eye, are a source of galvanic corrosion under the conditions as enumerated above.

Hence it has been found advantageous according to the invention to provide an oil film on the metal surface which is and remains continuous in its physical state. This objective can be accomplished by formulating a novel emulsion which comprises a concentrate of sperm whale oil, and a wetting agent or surfactant, these together being mixed with water to form the desired emulsion of the invention. The surfactant may be derived from a hydrophobic material, isoctylphenol, combined with a hydrophilic substance, such as ethylene oxide, to produce polyoxyethylated isoctylphenol which gives the utmost stability in oil concentration ranges of from 1 to 15% (by volume) without the use of high speed mixing.

According to the invention we have found that the protective film on the metal surface during the first 24 hours after coating is easily soluble in toluene and may be easily re-emulsified with water and washed from the metal surface. Thus the emulsion of the invention may be used as a lubricant for a metal part and easily removed by water, leaving no residue thereon.

During this initial period basing cement, marking ink and solder will not adhere to the base. During this preliminary 24 hour period, while the protective film is in contact with air, the film chemically reacts with oxygen in the air to change its chemical nature. After 24 hours the film is no longer soluble in toluene and is water repellent. Moreover, after this 24 hour reaction period cement prefilling of the base, ink marking of the base and the soldering of the lead wires to the base may be effected without difficulty.

This metallic part immersed in the sperm oil emulsion of the invention is provided with a coating which has corrosion resistance and high lubricity. After the initial 24 hour curing period the coating permits the facile prefilling of the coated base, ink marking of the base shell and the soldering of the lead wires thereto.

In its general aspect the present invention has as its objective a metallic part provided with a protective coating having corrosion resistance and high lubricity.

A specific object of the invention is a metallic part having a protective coating which is water repellent and has permanent film stability after sufficient time has elapsed to permit a chemical reaction to take place between the initial protective film and oxygen.

A further object is a sperm whale oil emulsion for coating an incandescent metallic lamp base which permits the cement prefilling and the ink stamping of the coated base and soldering of the lead wires thereto.

An additional object is a sperm whale oil emulsion

comprising a sperm whale oil concentrate in the range of 1% to 15% by volume and the balance water.

A further object is a sperm whale oil concentrate for a sperm whale oil emulsion having sperm whale oil in the range from 96.4%–97.2% by volume and the balance a surfactant, such as polyoxyethylated isoctylphenol.

Another object is an alternative embodiment of a sperm whale oil concentrate having sperm whale oil in the range of 72.2 to 72.8% by volume and the balance a surfactant, such as polyoxyethylated nonylphenol, suitably dissolved in water.

A still further object is a further alternative embodiment of a catalyzed sperm whale oil concentrate having sperm whale oil and one or more catalysts from the group consisting of the naphthenates and linoleates of iron, cobalt, manganese, zinc and lead.

Other objects of the invention will become apparent to those skilled in the art to which it appertains as the description thereof proceeds both by direct recitation thereof and by implication from the context.

A metallic part, for purposes of illustration only a lamp base suitably aluminum, may desirably (according to the invention) be provided with a protective coating or film. A stable emulsion of a concentrate of sperm whale oil and a suitable surfactant and water is employed to coat the base, as for example by dipping, and to provide a protective coating thereon.

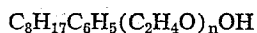
SPERM OIL

The sperm whale oil employed by applicants may suitably be bleached winter grade of light yellow color which is soluble in toluene $C_6H_5CH_3$ and hot trichloroethylene C_2Cl_3H . The oil should be moisture free and clear. The sperm oil may have a viscosity SSU at 100° F., 98.6 sec.; a specific gravity 0.9; flash point 505° F.; fire point 585° F.; acid No. 0.5 max.; saponification value 132–138; iodine value 82–92; cloud point 38° F. max.; and density 7.5 lb./gal.

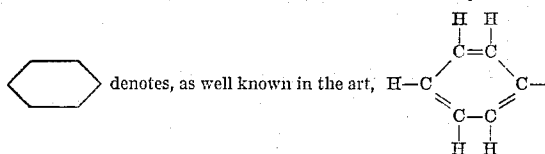
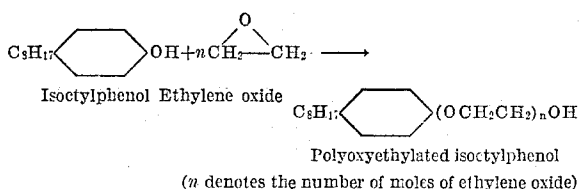
SURFACTANT

A surfactant or wetting agent employable in the emulsion of the invention is capable of mixing oil with water, or water with oil, as a result of its dual hydrophobic and hydrophilic natures. A hydrophobic substance usually has good solubility in oil and is either insoluble, or has low solubility, in water. On the other hand, a hydrophilic material has high water solubility and usually is insoluble in most oil based compounds. Usable surfactants may combine both these functions in varying degrees in one product.

The particular preferred surfactant employed by the applicants is derived from the same hydrophobic starting material, isoctylphenol $C_8H_{17}C_6H_5OH$ and differs only in the amount of hydrophilic substance, ethylene oxide C_2H_4O combined with isoctylphenol. The chemical structure of the surfactant employed by the applicants is that of a polyoxyethylated isoctylphenol



illustrated by the following reaction and final product formula:



According to the invention we prefer surfactants hav-

ing an *n* equal to 8, i.e. a ratio of 8 moles of ethylene oxide to 1 mole of isoctylphenol.

PREPARATION OF SPERM WHALE OIL CONCENTRATE

It will be understood that when the concentrate is being prepared the measuring utensils, stirring equipment and mixing vessels must be absolutely free of water. For example, to 9.463 liters of sperm oil in the mixing vessel, about .275 liter of surfactant may be added. The sperm whale oil and surfactant are stirred for about 5 minutes until the materials are thoroughly mixed and form 9.738 liters of sperm oil concentrate (about 96.9% sperm oil by volume).

Other equally desirable ratios of sperm whale oil to surfactant are shown in Table I as follows:

Table I

Volume of Surfactant (liters)	Volume of Sperm Oil (liters)	Total volume of Concentrate (liters)	Percent by volume of Sperm Whale Oil in Concentrate
.250	9.463	9.713	97.2
.275	9.463	9.738	96.9
.300	9.463	9.763	96.4

Thus we have found that a satisfactory sperm whale oil concentrate has a range of from 96.4% to 97.2% by volume of sperm whale oil and the balance surfactant, or 3.6%–2.8% by volume of surfactant.

PREPARATION OF SPERM WHALE OIL EMULSION

The final volume of the sperm whale oil concentrate required for the sperm whale oil emulsion may depend upon the volume of the dipping tank. For example, to a tank of suitable capacity is added 237 liters of city water. The 9.738 liters of concentrate are then added slowly and the mixture agitated, as with air, until the desired emulsion is achieved. The resultant 246 liters of stable emulsion (4% sperm oil concentrate by volume) will have a milk white color and foam to a slight degree.

To the water emulsion may be added .010%, by volume, of a masking compound deodorant. A high boiling ester oil masking compound employed in fish oils and fish soaps has been found satisfactory.

Table II below gives desirable ratios of sperm whale oil concentrate to water required to produce the sperm whale oil emulsion of the invention:

Table II

Volume of Sperm Oil Concentrate (liters)	Volume of Water (liters)	Total Volume of Sperm Oil Emulsion (liters)	Percent by volume of Concentrate in Sperm Oil Emulsion
2.46	243.54	246	1
9.738	246.62	246	4
19.6	226.4	246	8
27.0	219.0	246	11
36.8	209.2	246	15

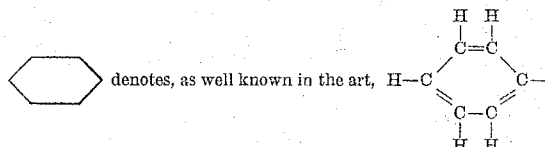
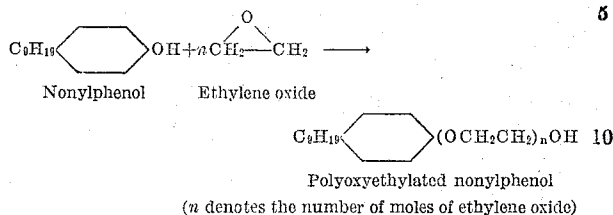
Thus we have found that a satisfactory sperm whale oil emulsion has a range of from 1% to 15% by volume of sperm whale oil concentrate and the balance water.

ALTERNATIVE EMBODIMENT OF THE SPERM WHALE OIL CONCENTRATE

Applicants have found that by employing an alternative surfactant or wetting agent, which is highly hydrophilic and practically insoluble in the oil, the coating on the metal part has good film strength and is water repellent immediately after application.

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The chemical structure of the alternative surfactant employed by applicants is that of a polyoxyethylated nonylphenol $C_9H_{19}C_6H_5(C_2H_4O)_nOH$ illustrated by the following reaction and final product formula:



According to the invention we prefer surfactants having an n equal to 10, i.e. a ratio of 10 moles of ethylene oxide C_2H_4O to 1 mole of nonylphenol $C_9H_{19}(C_6H_5)OH$.

An emulsion employing this type surfactant is prepared in a different manner. About 45 grams (38.4 cc.) of the solid surfactant, polyoxyethylated nonylphenol, may be added to about 3.785 liters of city water and the mixture (3.785 + .038 = 3.823 liters) heated to about 75° C. to dissolve the surfactant. About 9.463 liters of sperm whale oil is added to this solution and the mixture subjected to a high speed agitator operating at a speed of about 1500 r.p.m. to form 13.286 liters of sperm whale oil concentrate (about 72.5% sperm whale oil by volume).

Other equally desirable ratios of surfactant to water are shown in Table III as follows:

Table III

Volume of Surfactant (liters)	Volume of water (liters)	Total Volume of Surfactant (liters)	Percent by Volume of Surfactant
.034	3.785	3.819	.89
.038	3.785	3.823	.99
.042	3.785	3.827	1.05

Other equally desirable ratios of sperm whale oil to surfactant are shown in Table IV as follows:

Table IV

Volume Surfactant (liters)	Volume of Water (liters)	Volume of Sperm Whale oil (liters)	Total volume of Concentrate (liters)	Percent by Vol. of Surfactant	Percent by Vol. Sperm whale oil in Concentrate
.034	3.785	9.463	13.282	.256	72.2
.038	3.785	9.463	13.286	.286	72.5
.042	3.785	9.463	13.290	.316	72.8

Thus we have found that a satisfactory sperm whale oil concentrate has range of from 72.2% to 72.8% by volume of sperm whale oil and the balance surfactant.

In turn a satisfactory sperm whale oil emulsion having a percentage by volume of the above mentioned alternative sperm oil concentrate in the range of from 1% to 15% and the balance water may be formulated as shown in Table II.

ALTERNATIVE EMBODIMENT OF CATALYZED SPERM WHALE OIL CONCENTRATE

A further alternative embodiment of this invention is the employment of means for accelerating the reaction of oxygen with the emulsion residue or protective film which resides on the metal part as the result of the aforementioned treatment. This oily film is, for a period of at least 24 hours, of a wet, mobile nature; is easily remov-

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able by solvents, such as toluene $C_6H_5CH_3$ and may be re-emulsified away with water. Moreover, it exhibits the characteristic, rather unpleasant, odor of fish oil unless masked as previously noted.

We have found, according to the invention that the 24 hour oxidizing period may be shortened by two methods.

First, the treated or coated metal part may be heated in air to a temperature of about 200° C. (a temperature desirably lower than the flash point of the sperm whale oil or the surfactant) for 15 minutes.

Secondly, the concentrate may be fortified by the addition of a catalyst or accelerator which serves to quicken the oxidation and harden the emulsion residue on the metal parts at ordinary temperatures or to implement the attainment of this result at 200° C. in about 100 seconds.

The catalyst may be a metallo-organic compound such as an iron, cobalt, manganese, zinc, or lead compound of naphthenic or linoleic acid. Mixtures of two or more of these also may be used.

Examples of a satisfactory naphthenic base catalyst employable to fortify the sperm whale oil concentrate of the invention are as follows:

Nuodex 6% iron catalyst
Nuodex 6% manganese catalyst
Nuodex 6% cobalt catalyst
Nuodex 24% lead catalyst
Nuodex 3% cerium catalyst
Nuodex 6% vanadium catalyst

Nuodex is the trade name for catalysts manufactured by the Nuodex Products Co. of Elizabeth, New Jersey.

It will be understood that the use of a naphthenic base catalyst with the sperm whale oil concentrate of the invention is not limited to the above enumerated examples but may also include linoleic base catalysts of the same metals and mixtures thereof.

The catalysed film residing on the metal part thus achieves a quick permanence and immobility, immediately exhibits complete water repellence and resistance to galvanic corrosion and no longer possesses the unpleasant fish oil odor. These characteristics are of great processing and economic value in the application of the protective film and potentially and practically extend its use and the use of metal parts, such as aluminum lamp bases, which may be so coated and employed in more corrosive applications where normally only brass would be employed. This extension of the use of aluminum leads to the conservation of critical metals, such as copper and zinc.

A typical preparation of the fortified concentrate and emulsion comprises the mixing together of 9.463 liters sperm whale oil and 0.118 liter of the catalyst, for example, Nuodex 6% iron catalyst. The mixture is then preferably heated to about 150° C. To this mixture is then added .275 liter of the polyoxyethylated isooctylphenol surfactant $C_8H_{17}C_6H_5(C_2H_4O)_nOH$. The fortified concentrate, so prepared, is then added with constant stirring into a prescribed volume of water (as explained in the preferred embodiments of Table II, such as 237 liters of water) and the resultant emulsion is used to coat the metal parts. In this regard it is noted that the metal parts preferably heated by hot water or steam to a temperature of 100° C. or more, are then dipped into, or are otherwise coated with, the emulsion at room temperature.

METHOD OF DIPPING

The bases may be prepared for dipping in the sperm whale oil emulsion of the invention by immersion in hot water between 80-90° C. After draining the hot bases of excess water, they are immersed in the sperm whale oil emulsion of the invention which is desirably maintained at a temperature of about 70° C. The coated bases are then allowed to drain and are dried for a period of about 100 seconds in a suitable drier, such as a vibratory or rotary type, maintained at a sufficient temperature, such as 180° C.-200° C., to render the bases free of water.

EMULSION TESTING

The sperm whale oil emulsion of the invention may be tested by placing 500 cc. of the emulsion heated to 70° C. in a 1000 cc. graduated cylinder and mixing (with constant stirring) about 30 cc. of concentrated sulfuric acid therewith. After about four minutes have elapsed a sperm whale oil layer of not more than 20 cc. nor less than 15 cc. shall be present. If the oil layer is greater than 20 cc., water should be added to the emulsion to bring it to the desired limits. If the oil layer is less than 15 cc., sufficient sperm whale oil concentrate must be added to the emulsion.

INSPECTION

To qualitatively measure the oxidized coating on metal parts, such as bases, an inspection test has been devised whereby the effectiveness of the oil film in resisting galvanic corrosion is measured.

About 100 cc. of city water and 7 grams of mercuric chloride may be heated to 80° C. to effect a complete solution. When the mercuric chloride solution has been cooled to room temperature the test part, such as a coated base, may be completely immersed in said solution. The test base may be immediately removed therefrom and placed upon a level surface with the eyelet in the down position and the open end of the base up.

After the base has been allowed to stand for approximately 10 minutes, it should be examined. A sufficiently coated base will have only black spots thereon. Grey areas or gray spots on the base are evidence of corrosion or direct attack upon unprotected metal and indicates that the base was not sufficiently coated.

Although a preferred embodiment of the invention has been disclosed, it will be understood that modifications may be made within the spirit and scope of the invention.

We claim:

1. A corrosion resistant coating composition of high lubricity for an aluminum object comprising an emulsion of sperm whale oil concentrate and water, said sperm whale oil concentrate being in the range of from 1% to 15% by volume and comprising sperm whale oil in

the range of 72.2% to 72.8% by volume and the balance of a surfactant.

2. A corrosion resistant coating composition of high lubricity for an aluminum object comprising an emulsion of sperm whale oil concentrate and water, said sperm whale oil concentrate being in the range of from 1% to 15% by volume and comprising sperm whale oil in the range of 72.2 to 72.8% by volume and the balance a polyoxyethylated nonylphenol surfactant dissolved in water, said surfactant comprising 1 mole of hydrophobic nonylphenol and 10 moles of ethylene oxide.

3. A method of coating an aluminum part with a corrosion resistant film of high lubricity comprising mixing sperm whale oil in the range of from 96.4% to 97.2% by volume with a surfactant consisting of nonylphenol and ethylene oxide to form a sperm whale oil concentrate, adding water to said sperm whale oil concentrate to form a sperm whale oil emulsion having from 1% to 15% by volume of sperm whale oil concentrate and the balance water, and dipping said part in said sperm whale oil emulsion.

4. The method of coating an aluminum part with a corrosion resistant film of a high lubricity comprising mixing a surfactant consisting of 10 moles of ethylene oxide and one mole of nonylphenol, mixing from 96.4% to 97.2% by volume of sperm whale oil with said surfactant to form a sperm whale oil concentrate, adding water to said sperm whale oil concentrate until said concentrate is from 1% to 15% by volume of the mixture, heating said mixture to a temperature of 75° C. and agitating said mixture at high speeds to form a sperm whale oil emulsion and dipping said aluminum part in said sperm whale oil emulsion.

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