United States Patent [19]

Kemp

[11] **3,884,851**

[45] **May 20, 1975**

[54] **PRIMER FOR TREATING METAL SURFACES**

[75] Inventor: **Woodrow E. Kemp**, Pittsburgh, Pa.

[73] Assignee: **Koppers Company, Inc.**, Pittsburgh, Pa.

[22] Filed: **May 19, 1972**

[21] Appl. No.: **255,128**

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 64,613, Aug. 17, 1970, abandoned, and a continuation-in-part of Ser. No. 785,358, Dec. 19, 1968, abandoned.

[52] **U.S. Cl.**..... **260/3.3;** 117/132 R; 117/161 A; 117/161 P; 260/23.7 H; 260/28; 260/28.5 D; 260/30.6 R; 260/33.6 A; 260/33.8 UA; 260/45.8 A; 260/857 UN; 260/857 D

[51] **Int. Cl.**..... **C08c 13/00;** C08g 41/04

[58] **Field of Search**..... 260/3.3, 3.5, 23 R, 23 EP, 260/23.7 H, 857 U, 735, 736

[56] **References Cited**

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Primary Examiner—John C. Bleutge

Attorney, Agent, or Firm—Sherman H. Barber; Olin E. Williams; Oscar B. Brumback

[57] **ABSTRACT**

A primer composition for treating metal surfaces prior to applying coal tar enamel is provided. The cured primer coating is essentially the reaction product of chlorinated rubber and a polyamide resin. Plasticizers and pigments optionally may be included in the composition. The cured primer coating gives improved adhesion between the primed metal surfaces and a finishing coat of coal tar enamel. The primer composition, when plasticized with coal tar heavy oil is the only hereto known primer suitable for use with coal tar enamels made from high volatile containing coals.

10 Claims, No Drawings

PRIMER FOR TREATING METAL SURFACES

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a Continuation-in-Part of my co-pending application, Ser. No. 64,613, filed Aug. 17, 1970, and claims priority from my earlier application Ser. No. 785,358 filed Dec. 19, 1968 both now abandoned.

BACKGROUND OF THE INVENTION

Coal tar enamels are highly effective for the protection of the surfaces of metal and particularly ferrous metal surfaces, such as pipes and conduits. But satisfactory adherence of such enamels to such metal surfaces necessarily requires the interposition of some sort of primer coat. The use of the primer coat is necessary to wet the metal surface thoroughly, and to establish a good bond between the coal tar enamel and primed metal. The coal tar enamel is generally applied in a hot condition to the primed metal. Applying hot coal tar enamel to an unprimed metal surface results in an uneven wetting of the metal surface and a resultant poor bond between the metal surface and the coal tar enamel.

Yeager, U.S. Pat. No. 2,228,102 discloses a primer for metal surfaces which are to be subsequently coated with coal tar enamel. The primer is a solvent cutback coal tar pitch. This type of primer requires the use of high boiling solvents to dissolve the coal tar pitch. As a result, time consuming drying periods are required. Also, valuable storage space must be used to hold the primed metal surfaces for the long drying periods.

Shideler, U.S. Pat. No. 2,752,267 discloses a faster drying primer which consists of chlorinated rubber and heavy creosote both of which are dissolved in a volatile solvent. This primer, although being quick drying, is not suitable for use with all types of coal tar enamels.

The composition disclosed in Shideler, U.S. Pat. No. 2,752,267 is particularly unsuitable for use with coal tar enamels which are made of high volatile containing coals. This is a grave disadvantage because high volatile containing coals are being used increasingly throughout the world, particularly in the United States, where native coal reserves are being depleted. High volatile containing coals produce coal tar enamels with higher viscosities than comparable coal tar enamels made with lower volatile containing coals. These higher viscosity coal tar enamels upon application to a metal surface which has been primed with the chlorinated rubber-creosote oil primer of the type disclosed in the Shideler patent, do not sufficiently wet the primed surface and as a result do not form a strong bond with the primed metal surface.

The primer composition of this invention overcomes the disadvantages associated with the above mentioned primers of the prior art. The primer composition of this invention does not contain high boiling solvents and long drying times are not needed. Also, the primer composition of this invention forms a strong bond with all types of coal tar enamels, particularly with those made from high volatile containing coals.

SUMMARY OF THE INVENTION

In accordance with this invention, a primer composition for treating metal surfaces prior to the application of coal tar enamel comprises chlorinated rubber and a polyamide resin. The chlorinated rubber and the polyamide resin are dissolved in a volatile organic solvent having an evaporation rate that is between that of toluene and high flash naphtha. The chlorinated rubber and the polyamide resin are present in reactive proportions and react after application of the primer to a metal surface to form a hard infusible coating with accompanying volatilization of the solvent.

A plasticized modification of the primer composition can be made by including one-third to two-thirds part by weight, based on one part by weight of chlorinated rubber, of a suitable plasticizer such as a coal tar heavy oil in the formulation.

A storage-stable version of the primer composition can be made by including 1/100 – 1/20 parts by weight per one part by weight chlorinated rubber, of a volatile organic acid in the primer composition.

Pigments and additional plasticizers may optionally be added.

DETAILED DESCRIPTION

The primer composition of this invention is a solution of a chorinated rubber and a polyamide resin and optionally, certain stabilizers and modifiers, in aa volatile organic solvent. The chlorinated rubber and the polyamide resin when mixed are present in reactive proportions so that after the mixed components are applied to a metal surface, the chlorinated rubber and polyamide resin react to form a tough infusible coating which strongly adheres to the metal surface. The primer components after mixing can be made indefinitely stable at room temperature and somewhat higher temperatures by including a volatile organic acid inhibitor in the composition. The primer components after mixing can also be refrigerated to 0°C. to give a primer composition having an indefinite shelf life. Plasticizers and so-called "stabilizers" may optionally be included in the primer composition. A particularly effective plasticizer is coal tar heavy oil which gives a resultant primer coating which has outstanding adherences of coal tar enamels made from high volatile containing coals. Pigments may also be included to give color to the resultant primer composition.

The primer composition of this invention is applied to metal surfaces in a film of about 1 to 3 mils thickness. If no organic acid is present in the primer composition, the chlorinated rubber and polyamide resin will react to form a hard infusible coating which strongly adheres to the metal surface. The inert organic solvent will of course evaporate away following application of the primer to a metal surface. If an organic acid is present in the primer composition, however, the reactive components will not react completely to form the hard infusible coating unless sufficient heat is applied to the coating to evaporate all of the organic acid since the organic acid is not appreciably volatile at ambient temperatures. The heat to completely evaporate the organic acid is conveniently supplied by the coal tar enamel itself since conventionally the coal tar enamel is flowed or brushed over the metal surface at a temperature of about 250° to 270°C.

The chlorinated rubber used in the primer composition of this invention is made by the chlorination of either natural or synthetic rubber. Chlorinated rubber is commercially available. Chlorinated natural rubber is sold, for example, under the trademarks PARLON and ALLOPRENE. Commercial chlorinated rubber gener-

ally contains approximately 50 to 67% by weight total chlorine, has a viscosity of from 4 to 300 centiposes as determined on solutions of 20% by weight concentration in toluene at 25°C. with a viscometer based on the Bingham and Murry Plastometer, and is an odorless, tasteless, white powder. It is a stable material in a dry state at room temperature, but, at temperatures above 125°C., begins to decompose. The rate of decomposition increases with higher temperatures and at about 135°C. the chlorinated rubber begins to show noticeable chemical break-down.

The polyamide resins used in the composition of this invention are the condensation products of a polyalkylene-polyamine and unsaturated polymeric fatty acids. The polyalkylene-polyamines are illustrated by the general formula: $H(HNCR_2CR_2)nNH_2$, wherein R is hydrogen or lower alkyl radical containing less than 5 carbon atoms and *n* is an integer less than 6. Examples of polyalkylene-polyamines are ethylene diamine, diethylene triamine, triethylene tetraamine, tetraethylene pentamine, di(1,2-propane) triamine, 3,3-iminobispropylamine and the like. The polymeric fatty acids employed in preparing the polyamid resins are those resulting from the polymerization of drying or semi-drying oils, or their free acids or the simple aliphatic alcohol esters of these acids, particularly sources rich in linoleic acid. Suitable drying or semi-drying oils include soybean, linseed, tung, perilla, cottonseed, corn, sunflower, safflower, and dehydrated castor. Suitable fatty acids may also be obtained from tall oil, soapstock, and other similar materials. Particularly suitable polyamide resins are those sold commercially by General Mills, Inc. under the tradename VERSAMID. These polyamide resins are disclosed in U.S. Pat. Nos. 2,379,413; 2,839,549; and 3,002,941.

The relative amounts of chlorinated rubber and polyamide resin which may be used in this invention should be within a weight ratio range of 1 – 18 parts of chlorinated rubber to 1 part polyamide resin. When a greater proportion of chlorinated rubber is used the resultant coating is extremely brittle, cracks easily, and does not bond well with the subsequently applied coal tar enamel. When a smaller proportion of chlorinated rubber is used, the resultant coating is soft and tacky and does not bond well with the metal surface.

Plasticizers such as chlorinated bi-phenyls, chlorinated triphenyls, chlorinated poly-phenyls, chlorinated waxes, e.g., chlorowax 40(R) diphenyl phosphates and mixtures of these plasticizers may be added to the primer composition to improve the flexibility of the cured primer coating. One such plasticizer is sold commercially under the trademark AROCLOR. The use of a plasticizer is not essential in the primer composition for many uses because the polymeric fatty acid moiety of the polyamide resin itself, induces an internal plasticizing effect on the primer. The additional plasticizers when used are generally used in the amount of from one-tenth to one-fifth part by weight on one part by weight of chlorinated rubber.

If the coal tar enamel to be applied to the primed metal surface is of the type made from high volatile containing coal, coal tar heavy oil is preferably added to the primer composition. Coal tar heavy oil is the distillate that in the distillation of coal tar distills between 225°C. and 385°C. The coal tar heavy oil has a specific gravity of between 1.04 to 1.3 at 13°C.

High volatile containing coals are found predominantly in Australia and New Zealand. Such coals contain about 27 to 44% by weight volatile matter based on a dry mineral matter free basis. Coal tar enamels made from these coals are very viscous, having a high surface energy. These viscous coal tar enamels do not wet sufficiently conventional cured primer surfaces to form a strong primer-coal tar enamel bond. High viscosity coal tar enamels readily wet the cured primer coating composition of this invention when the primer composition is plasticized with the coal tar heavy oil. Accordingly, coal tar heavy oil plasticizer, when used for this purpose, is used in the range of from one-third to two-thirds part by weight based on one part by weight of chlorinated rubber.

As stated previously, the commercial chlorinated rubbers useful in the invention are stable at room temperature in a dry state. When dissolved in the inert organic solvents of the invention, however, the chlorinated rubber may decompose slightly to form HCl. The HCl is corrosive to metals. Thus, if it is contemplated to store the primer compositions of the invention in metal cans for any length of time, a stabilizer to absorb the HCl should be added to the primer compositions to ensure against corrosion of the cans. Compounds useful as stabilizers in the primer composition include compounds containing oxirane oxygen such as epichlorohydrin, propylene oxide and styrene oxide. Any compound, however, which is known to protect against the corrosive effects of decomposing chlorinated rubber and HCl may be used as a stabilizer. Stabilizers when used are used in amounts up to one-third part by weight based on one part by weight of chlorinated rubber and generally one-tenth part. Additionally, the stabilizers should be volatile at the temperatures at which coal tar enamel is applied and, therefore, evaporate from the metal surface.

Pigments may also be included in the primer formulation to give the resultant primer color. Examples of suitable pigments are iron oxide, talc, and lampblack. The pigments are used up to about one-fourth part by weight based on one part by weight of chlorinated rubber.

The solvent used in the primer composition should be inert to all ingredients. The only requirement as to the amount of solvent used is that it be present in an amount sufficient to dissolve the two essential ingredients, i.e., the chlorinated rubber and polyamide resin. It is also desirable to have enough solvent present to make the primer composition brushable and sprayable. Usually one to three parts by weight of solvent per one part by weight of the chlorinated rubber is used. The solvent evaporates readily after the primer coating has been applied to the metal surface being primed (generally in a film thickness of 0.3 to 3 mils). The solvent should have an evaporation rate between the evaporation rate of toluene and high flash naphtha. Examples of suitable solvents besides toluene and high flash naphtha are diacetone alcohol, butyl alcohol, benzene, xylene, solvent naphtha, ethyl benzene, chlorinated solvents such as trichloroethylene and mixtures of these solvents. A solvent which evaporates too fast results in a primer coating which is powdery and which does not adhere well to the metal surface being primed, and a solvent which evaporates too slowly requires too much time for primer application, making the economics of the primer coating operation impractical.

The chlorinated rubber and the polyamide resin of the primer composition of this invention react quickly with one another at room temperature to form a cross-linked polymer and the reaction is accelerated with an increase in temperature. In a closed container a typical primer composition gels within about 24 minutes at room temperature and within about 13 to 20 minutes at 70° to 75°C. It has been found that this gelling of the primer composition of this invention may be retarded by keeping the composition under refrigeration, for example below 0°C. Refrigerating the composition, however, may not be practical.

It is possible to operate with the primer composition at room temperature by formulating the composition as a two-can system in which the chlorinated rubber is in one can and the polyamide resin is in a separate can. In such systems chlorinated rubber and stabilizer and plasticizers, if used, are mixed with a suitable amount of solvent and placed in one can and the polyamide resin and pigment are mixed with a suitable amount of solvent and placed in another can. When it is desired to apply the primer, the contents of the two cans, which are in the desired proportions, are thoroughly mixed at the job site and immediately either sprayed, flowed, or brushed onto the metal surface to be primed.

It has also been found that the composition can be made in a one-can system and that the composition will have an indefinite shelf life in a closed can if about one one-hundredth to one-twentieth part by weight, based on one part of weight of chlorinated rubber, of a volatile organic acid is included in the primer composition. The amount of organic acid used is that necessary to tie up the polyamide resin. The acid prevents room temprature gelling of the primer composition by reacting with the free amine groups of the polyamide to form the salt of the organic acid thus preventing reaction of the polyamide resin with the chlorinated rubber. The volatile organic acid should be soluble in the primer mixture and should be sufficiently volatile to evaporate quickly when the coal tar enamel is subsequently applied to the primed metal surface. Examples of suitable volatile organic acids are formic acid, acetic acid, chloroacetic acid, propionic and butyric acids. Using less than the recommended lower limit of acid results in eventual room temperature gelling. Using greater amounts than the recommended lower limit of organic acid may be advantageous in circumstances where the primer is to be stored for an indefinite period of time. The acid is slightly volatile and a certain amount should be expected to escape the primer composition with long storage periods. Using greater than the recommended upper limit of acid serves no advantage.

Conventional means may be used to apply the primer composition of this invention to the metal surface. Usually the primer composition is applied to the metal surface to be coated by spraying, flowing, or brushing onto the surface. Complete evaporation of the solvent is essential if the primer is to adhere well to the substrate. A coating thickness of 0.3 to 3 mils is recommended. Devolatilization may not be complete at room temperature in a thick film and adhesion of the primer to the substrate would be inadequate and blistering of the subsequently applied coal tar enamel finishing coat may occur. Thin films of primer coatings may not provide sufficient primer protection and as the subsequently applied coal tar enamel coating may not bond well. A primer coat, not containing the organic acid, of the recommended thickness, will devolatilize and form an infusible hard coating within 24 hours at room temperature. If the substrate is heated (e.g. to 50° – 90°C.) devolatilization and reaction will occur quicker, usually within 12 hours.

The organic acid, if present in the primer composition, will not completely evaporate from the primer coat even after prolonged drying at ambient temperatures. It has been determined, however, that the coal tar enamel can be applied to a primer coat containing appreciable amounts of the organic acid without noticeable deterioration of the adherence of the primer to the substrate. The heat of the hot coal tar apparently evaporates any organic acid remaining in the primer coat.

The coal tar enamel is applied to the primer coating at temperatures between 200° and 270°C. The enamel forms a coating layer of a harder, tougher character than the primer which bonds strongly to the primer coating.

The invention is further illustrated by the following examples of suitable primers prepared according to the invention wherein the parts referred to are on a weight basis.

EXAMPLE I

This example shows a formulation for a two-can primer system. Into a mixing tank is charged 600 parts of xylol, 40 parts of epichlorohydrin and 157 parts of chlorinated rubber (PARLON) having about 67% total chlorine. The mixture is stirred at 75°C. until the chlorinated rubber dissolves. Then, 22 parts of a chlorinated bi-phenyl plasticizer (AROCLOR 1254) is added and mixing is continued for 5 to 10 minutes. The mixture is placed in a can and the can sealed as one can of the two-can system.

To another mixing tank is charged 20 parts of a polyamide resin (VERSAMID 140) and 30 parts of xylol. Mixing is conducted for 10 minutes to 15 minutes. Then, 30 parts of No. 10 lampblack, as the pigment, is slowly charged to the mixture. Mixing is continued for 1 to 2 hours to evenly disperse the lampblack throughout the mixture. This mixture is then placed in a can and the can sealed as the second can of this two-can system.

When it is desired to apply the primer composition, the two cans at room temperature are combined, the combination stirred for 10 to 15 minutes to insure homogeneity, and then the composition is painted onto the metal surface to be primed.

EXAMPLE II

To provide a primer composition that has utility for priming surfaces that are to be final coated with high viscosity coal tar enamel, a two-can system which contains the additional plasticizer, coal tar heavy oil, is made by charging 1200 parts of xylol, 80 parts of epichlorohydrin, and 314 parts of chlorinated rubber (PARLON) having 67% total chlorine to a mixing tank where the ingredients are mixed at 80°C. until the rubber dissolves. Then 180 parts of coal tar heavy oil having a viscosity of 1.2 at 13°C. and having a distillation range of 230°C. to 355°C. is charged to the mixture and stirring is continued for an additional 5 to 10 minutes. Thereafter, 44 parts of chlorinated bi-phenyl plasticizer (AROCLOR 1254) is added and the stirring is contin-

ued for an additional 5 to 10 minutes. The mixture is placed in a container and the container is sealed.

To another mixing tank is charged 40 parts of a polyamide resin (VERSAMID 100) and 60 parts of xylol. Stirring is commenced and continued for 10 to 15 minutes. Then, 53 parts of NO. 10 lampblack is charged slowly and mixing continued for an additional 2 to 3 hours. This mixture is then placed in a container and the container is sealed. When it is desired to apply this primer composition, the contents of the two containers at room temperature are combined and mixed well and then the resulting composition is painted or sprayed onto the metal surface to be primed.

EXAMPLE III

To make a storage stable composition that may be placed in one container, two vessels are used. For the one vessel, 300 parts of xylol, 20 parts of epichlorohydrin, and 80 parts of chlorinated rubber (PARLON) having 67% total chlorine are charged to a mixing tank. Stirring is commenced and continued at 70°C. until the chlorinated rubber dissolves. The 11 parts of chlorinated bi-phenyl plasticizer (AROCLOR 1254) is added and mixing is continued for an additional 5 to 10 minutes.

For the other vessel, a mixing tank is charged with 10 parts of a polyamide resin (VERSAMID 115) and 15 parts of xylol. Mixing is continued for 10 to 15 minutes. Then 15 parts of No. 10 lampblack is slowly charged to the mixture. Mixing is continued for 1 to 2 hours to evenly disperse the lampblack throughout the mixture. The contents of these two vessels are cooled to 0°C. and are then combined at this temperature. The composition resulting from this combination is rendered uniform by stirring for a period of 10 to 15 minutes. The mixture is placed in a container and the container sealed. The primer composition in the container has an indefinite shelf life if the container is maintained under refrigeration, i.e., at a temperature of 0°C.

EXAMPLE IV

To make a stable one-can primer system that has an indefinite shelf life at room temperature, 300 parts of xylol, 20 parts of epichlorohydrin, 80 parts of chlorinated rubber (PARLON) having 67% total chlorine are charged to a mixing tank. Stirring is commenced and continued at 70°C. until the chlorinated rubber dissolves. Then 11 parts of chlorinated bi-phenyl plasticizer (AROCLOR 1254) is added, and mixing is continued for an additional 5 to 10 minutes. Thereafter, 1.8 parts of glacial acetic acid are added and mixing is continued for an additional 10 to 15 minutes. A solution of 10 parts of the liquid polyamide resin (VERSAMID 140) and 30 parts xylol are added and mixing is continued for an additional 10 to 15 minutes. 10 parts of No. 10 lampblack are slowly mixed in and mixing continued for an additional 1 to 2 hours. The mixture is cooled to room temperature, placed in a container and the container is sealed. The primer composition has an indefinite shelf life.

EXAMPLE V

This example shows the process for making a room temperature stable primer which has been additionally plasticized with coal tar heavy oil. To a mixing tank is charged with mixing 600 parts xylol, 45 parts of epichlorohydrin, and 157 parts of chlorinated rubber (PARLON) having 67% total chlorine. Mixing is continued at 70°C. until the chlorinated rubber dissolves. Then 90 parts of coal tar heavy oil having a viscosity of 1.2 at 13°C. and a distillation range of 225°C. to 385°C. and 22 parts of chlorinated bi-phenyl plasticizer (AROCLOR 1254) are charged with mixing. Mixing is continued for an additional 5 to 10 minutes. Then 3.5 parts of formic acid is charged with mixing. Mixing is continued for an additional 10 to 15 minutes. Then, a solution of 20 parts polyamide resin (VERSAMID 140) and 30 parts xylol are charged to the original mixture with mixing. Mixing is continued for an additional 10 to 15 minutes. The 30 parts of No. 10 lampblack is slowly charged and thoroughly mixed into the composition. The mixture is cooled, placed in a container and the container is sealed. The composition in the container has an indefinite shelf life at room temperature.

EXAMPLE VI

This example shows another room temperature stable primer and a process for making it. To a mixing tank are charged, with mixing, 575 parts xylol, 7 parts epichlorohydrin and 274 parts of chlorinated rubber (ALLOPRENE 20). Mixing is continued until the solids dissolve. To another mixing tank are added, with mixing 485 parts xylol, 60 parts diacetone alcohol, 20 parts butyl alcohol, 35 parts of a liquid polyamide resin (VERSAMID 115), 8 parts acetic acid, 29 parts of diphenyl octyl phosphate (sold under the tradename SANTICIZER 141), 157 parts of coal tar heavy oil and 70 parts of lampblack. Mixing is continued for a time sufficient to thoroughly mix the ingredients. The contents of the two mixing tanks are then added to one mixing tank, thoroughly mixed and then placed in a sealed container. The composition in the container has an indefinite shelf life.

While the invention has been described in conjunction with the foregoing examples it will be understood that it is not intended to limit the invention to those examples. On the contrary, it is intended to cover all alternatives, modifications and equivalents that may be included within the spirit and scope of the invention as defined by the appended claims.

What is claimed is:

1. A storage stable coating composition for the priming of a metal surface, said coating composition consisting essentially of, by weight:

a. 1 part chlorinated natural rubber having about 50 to 67% by weight total chlorine;

b. from ⅛ to 1 part of a polyamide resin that is the condensation product of a polyalkylene — polyamine and an unsaturated fatty acid;

c. from one one-hundredth to one-twentieth part of a volatile organic acid and chloroacetic acid; and

d. from 1 to 3 parts of an inert volatile solvent that has an evaporation rate between the evaporation rate of toluene and high flash naphtha and that dissolves the chlorinated rubber, and said polyamide resin being present in said solvent in reactive proportions but stabilized by the presence of said organic acid so that the composition displays an indefinite shelf life in a sealed container but after application to a metal surface a crosslinking reaction occurs between the chlorinated rubber and polyamide resin to form an insoluble infusible coating.

2. The composition of claim 1 wherein said composition includes up to one-fifth part of a plasticizer for improving the flexibility of the cured primer coating.

3. The composition of claim 2 wherein said plasticizer is a chlorinated polyphenyl, a chlorinated wax a diphenyl phosphate or a coal tar heavy oil.

4. The composition of claim 1 wherein said composition includes an inorganic pigment.

5. The composition of claim 1 wherein said composition includes a stabilizer selected from the group consisting of epichlorohydrin and propylene oxide to protect against decomposition of the chlorinated rubber.

6. A coating composition for the priming of a metal surface, said coating composition consisting essentially of, by weight:

a. 1 part chlorinated natural rubber having about 50 to 67% by weight total chlorine;

b. from 1/18 to 1 part of a polyamide resin that is the condensation product of a polyalkylene-polyamine and an unsaturated fatty acid; and

c. from 1 to 3 parts of an inert volatile solvent that has an evaporation rate between the evaporation rate of toluene and high flash naphtha and that dissolves both said chlorinated rubber and polyamide resin.

7. The composition of claim 6 wherein said composition includes up to one-fifth part of a plasticizer for improving the flexibility of the cured primer coating.

8. The composition of claim 7 wherein said plasticizer is a chlorinated polyphenyl, a chlorinated wax a diphenyl phosphate or a coal tar heavy oil.

9. The composition of claim 6 wherein said composition includes an inorganic pigment.

10. The composition of claim 6 wherein said composition includes a stabilizer selected from the group consisting of epichlorohydrin and propylene oxide to protect against decomposition of the chlorinated rubber.

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