United States Patent

Nishihara

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[54] **PROCESS FOR THE FORMATION OF A SUPER-BRIGHT SOLDER COATING**

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Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 600,044, Dec. 8, 1966, abandoned.

[52] **U.S. Cl.** **204/43,** 204/DIG. 2
[51] **Int. Cl.** **C23b 5/38,** C23b 5/46
[58] **Field of Search** .....204/43, 44, 53, 54 R; 106/1; 117/130 E

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[57] **ABSTRACT**

A process for electrodepositing solder on a basis material from an aqueous, acidic bath comprising stannous and plumbous compounds, o-toluidine or a 2-alkyl anil as a brightening agent and a non-ionic surface-active agent as a dispersant, to obtain a super-bright solder coating on the basis material.

3 Claims, No Drawings

PROCESS FOR THE FORMATION OF A SUPER-BRIGHT SOLDER COATING

This application is a continuation in part of application Ser. No. 600,044, filed Dec. 8, 1966 and now abandoned.

This invention relates to a process for electro-depositing solder (alloy of tin and lead) on a basis material from a bath containing a compound of divalent tin and a compound of divalent lead together with a brightening agent, and a non-ionic surface-active agent.

By use of the process of this invention, there will be gained the following advantages:

1. a solder coating formed by electrodepositing the solder on a basis material from such a bath has a super-bright surface which is not less smooth than that of a coating formed by a conventional solder melt-plating process, such tin and lead contents and such thickness as intended before the electrodeposition, and a satisfactory size distribution of the particles in the coating, the size distribution influencing the anticorrosive effect of the coating on the basis material surface on which is is formed,

2. any stains and spots are not created on the surface of the coating during and after the plating operation,

3. the coated basis material withdrawn from the bath can be easily subjected to subsequent treatments such as washing with water,

4. especially the electrodeposits composing the coating are oriented during the electrodeposition from the bath,

5. a lower operating temperature may be used in the process,

6. a wide variety of basis materials may be used in the process, and

7. the coating will not be degraded with the lapse of time. And therefore the process will find many uses in the industrial world.

On the other hand, the composition of a bath which has normally been used in a conventional non-bright solder electroplating process and from which is obtained a coating of alloy consisting of 60 percent Sn and 40 percent, is approximately as follows:

Total tin	60 g./l.
Divalent tin	55 g./l.
Lead	25 g./l.
Free borofluoric acid	40 g./l.
Free boric acid	25 g./l.
Glue	5 g./l.

It is very difficult to control an electroplating when a bath of such composition as above is used in the electroplating because the ratio of a tin compound to a lead one and the content of glue will have delicate effects on the electroplating.

In general, in an electroplating using such a bath as above there usually arises a problem of variations (1) in composition of electrodeposit (coating of solder), (2) in distribution of the deposited particles and (3) in thickness of the deposit (coating). These three variations are correlated to one another in the plating; and therefore a bath having such a conventional composition will have disadvantages, when used in the plating, that a solder coating which has the desired contents of tin and lead, thickness and uniform distribution of solder particles throughout the coating (the distribution governing the anticorrosiveness of the coating) is remarkably difficult to form, that some fairly more troublesome operations are required for the after treatment of the coating fresh from the bath, and that, what is more important, the electrodeposit forming the coating is not oriented at all in its crystallization state whereby the surface of the coating is rough, a portion of the plating solution and other liquids such as washing water used after the plating readily enters into the interstices between the particles of the coating and the removal of the liquids from the interstices is not easy and stains and spots are apt to create in or attach to the coating easily during the plating operation and subsequent treatments. The conventional solder electroplating process, thus, has been used only for specific purposes in spite of many uses being expected if said disadvantages should be eliminated.

On the other hand, a conventional solder melt-plating (plating by dipping a basis material in a bath of molten solder) process can give a solder coating the surface of which is smooth and bright, and therefore it has been usually use in place of the conventional solder electroplating process.

The solder melt-plating process, however, has disadvantages that it cannot be applied to the plating of a basis material vulnerable to heat with solder because it requires a high temperature of about 230° – 280° C., it cannot give easily a solder coating the thickness of which is as predetermined, it can form nothing but a solder coating the thickness of which is only between about 3 and about 10μ depending partly upon the temperature of a bath used, it generally forms on a basis material a coating the thickness at every part of which is not uniform, for example, very large at one part while very small at another part of the coating whereby a large number of small particles to be plated cannot be satisfactorily plated at the same time, and it forms on a basis material a solder coating which is apt to change in composition with the lapse of time because of the oxidation of the solder coating due to being heated during plating operation. And the aforesaid disadvantages have set severe limits to the uses of the solder melt-plating process.

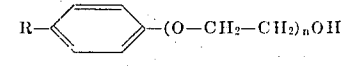
An object of this invention is to provide a novel solder electroplating process from which such disadvantages of the conventional solder electroplating and solder melt-plating processes have been eliminated. The novel process of this invention is characterized in that a plating bath used in this process is prepared by adding to a solution of a stannous compound and plumbous compound, any brightening additive selected from a 50 percent solution of o-toluidine in n-butyl alcohol, a 40 percent solution of a 2-alkyl anil in isopropyl alcohol and a 40 percent solution of this anil compound in n-butyl alcohol, and any dispersing additive selected from a polyethylene glycol-derived alkylphenol-type non-ionic surface-active agent, a polyethylene glycol-derived ether-type one and a mixture of the polyethylene glycol-derived alkylphenol-type non-ionic surface-active agent and a polyethylene glycol-derived alkylamide-type one, to obtain a super-bright coating by the electrodeposition of solder from the bath.

Another object of this invention is to provide a plating bath which may be used in the process of this invention.

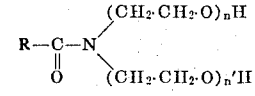
Examples of the stannous and plumbous compounds are stannous and plumbous borofluorides (or fluoroborates), respectively.

The alkyl of the 2-alkyl anil compounds which may be used in the practice of this invention, is methyl, ethyl or propyl.

As is known, said three surface-active agents are represented, in their order of description, by the following formulas:



wherein R is an alkyl group having carbon atoms of eight to nine and n is an integer of 8 to 15. R — (O — CH_2 — CH_2)$_n$O H wherein R is an alkyl group having carbon atoms of 12 to 18 and n is as defined above.



wherein R is an alkyl

group having carbon atoms of 12 to 18 and n and n' are an integer of 0 to 20.

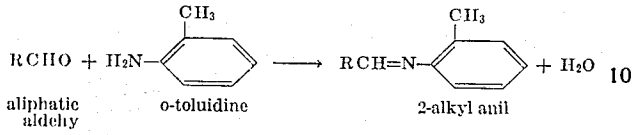
The previously-mentioned 2-alkyl anils can be obtained by reacting an aliphatic aldehyde with o-toluidine, based the well-known Schiff reaction, in the presence of an excess of alkali catalyst at a temperature of 10° – 25° C. for 190 – 360 hours. The reaction is illustrated by the following reaction formulas:

Theory of synthesis:

$$RCH{=}O + R'NH_2 \xrightarrow[\text{Schiff reaction}]{\text{Alkali catalyst}} RCH{=}NR' + H_2O$$

In the above reaction formula R and R' are alkyl and aryl, respectively.

The reaction used in this invention is:



wherein R is methyl, ethyl or propyl.

This invention will be better understood by the following examples in which copper test pieces (10 cm. × 10. cm × 0.3 mm.) were used as a cathode.

The general composition of a bath which may be used in the practice of this invention, is as follows:

45% aqueous solution of stannous borofluoride	140–180 ml./l. (40–50 g./l., calculated as divalent tin)
45% aqueous solution of plumbous borofluoride	20–40 ml./l. (8–16 g./l., calculated as divalent lead)
Brightening agent	20–50 g./l.
Formalin (formaldehyde 35%)	10–30 ml./l.
Surface-active agent	2–5 g./l.
Free HBF_4	50–120 g./.

If divalent tin is present in a bath in an amount of less than 40 g./l., a solder to be obtained will have no brightness; while the presence of divalent tin in the bath in an amount of more than 50 g./l. will produce a solder coating which is too hard and liable to crack.

The presence of less than 8 g./l. of divalent lead will decrease the solderability of a solder coating to be obtained, and that of more than 16 g./l. of divalent lead will produce a blackish solder coating.

The use of the brightening agent in an amount of less than 20 g./l. will give no brightness to a solder coating to be obtained, while that of the agent in an amount of more than 50 g./l. will give no increased brightness thereby making this uneconomical.

If formalin (formaldehyde 35 percent) is used in an amount of less than 10 ml./l. a solder coating being produced will have a dull brightness, while the use of more than 30 ml./l. of such formalin will no longer serve to increase the brightness of the coating.

The use of less than 2 g./l. of a surface-active agent will accelerate the decomposition of the brightening agent thereby decreasing the bright surface portion of a coating to be obtained, while that of more than 5 g./l. thereof will hinder the effect of the brightening agent.

And, the use of less than 50 g./l. of free HBF_4 will decrease the solubility of an anode used in the bath, and that of more than 90 g./l. will tend to allow the metals to excessively increase in concentration in the bath.

The solder coatings obtained according to this invention usually have at least 80 mirror plane brightness area as measured by Hull cell test.

EXAMPLE 1

(Electrodeposition of solder comprising 90% Sn and 10 percent Pb)

Composition of the bath used:

45% aqueous solution of stannous borofluoride (50 g./l., calculated as divalent tin)	180 ml./l.
45% aqueous solution of plumbous borofluoride (8 g./l., calculated as divalent lead)	20 ml./l.
Free HBF_4	110 g./l.
40% solution of 2-methyl anil in isopropyl alcohol	20 ml./l.
Polyoxyethylene alkyl aryl ether	2 ml./l.
Formalin (Formaldehyde conc., 35%)	10 ml./l.
Distilled water	balance

Operational conditions:

Cathode (substrate to be plated)	
Cathode current density	2 A/dm²
Temperature	20±5° C.
Anode	Tin 90% - Lead 10% Alloy
Agitation	The agitation was effected by rocking gently the cathode, which was basis material to be plated, during plating.

The coatings obtained by the electrodeposition from said bath and under said conditions were super-bright alloy of 90 percent Sn and 10 percent Pb as intended.

The same results were obtained when said procedure was repeated except that said solution of brightening agent and surface-active agent were substituted by a 50 percent solution of o-toluidine in butyl alcohol and polyoxyethylene oleyl ether, respectively.

EXAMPLE 2

(Electrodeposition of solder comprising 90 percent Sn and 10 percent Pb)

The procedure of Example 1 was repeated, but substituting as the brightening agent the 2-methyl anil by 2-ethyl anil. The same results as in Example 1 were obtained.

EXAMPLE 3

(Electrodeposition of solder comprising 80 percent Sn and 20 percent Pb)

The composition of the bath used in this Example was as follows:

45% aqueous solution of stannous borofluoride (45 g./l., calculated as divalent tin)	160 ml./l.
45 percent aqueous solution of plumbous borofluoride (12 g./l., calculated as divalent lead)	30 ml./l.
Free HBF_4	90 g./l.
40% solution of 2-methyl anil in n-butyl alcohol	20 ml./l.
Polyoxyethylene oleyl ether	2 ml./l.
Formalin (formaldehyde 35%)	10 ml./l.
Distilled water	balance

The electrodeposition was effected under the same conditions as in Example 1 except for the proportions of the stannous and plumbous compounds and the composition of the anode used in the bath.

EXAMPLE 4

(Electrodeposition of solder comprising 80 percent Sn and 20 percent Pb)

The same procedure of Example 3 was followed, but using as the brightening agent 2-ethyl anil instead of the 2-methyl anil. The solder coating obtained was the same as that obtained in Example 3.

EXAMPLE 5

(Electrodeposition of solder comprising 70 percent Sn and 30 percent Pb)

Composition of the bath used:

45% aqueous solution of stannous borofluoride (40 g./l., calculated as divalent tin)	140 ml./l.
45% aqueous solution of plumbous borofluoride (16 g./l., calculated as divalent lead)	40 ml./l.
Free HBF_4	90 g./l.
50% solution of o-toluidine	

in butyl alcohol	20 ml./l.
Polyoxyethylene alkyl aryl ether	2 ml./l.
Formalin (formaldehyde 35%)	10 ml./l.
Distilled water	balance

The electrodeposition was carried out under the same conditions as in Example 1 except for the proportions of the stannous and plumbous compounds and the composition of the anode used in the bath.

EXAMPLE 6

(Electrodeposition of solder comprising 70 percent Sn and 30 percent Pb)

The same procedure of Example 5 was followed, but substituting the solution of o-toluidine by a 40 percent solution of 2-propyl anil in isopropyl alcohol in the same amount.

The same results as those in Example 5 were obtained.

As is seen from the foregoing, the baths employed in said six Examples varied from one another particularly in amount of the stannous and plumbous sources and in kind of a brightening agent. They gave the desired solder coatings, respectively.

EXAMPLE 7

(Electrodeposition of 90 percent Sn – 10 percent Pb solder)

Composition of the bath used:

45% aqueous solution of stannous borofluoride (40 g./l. calculated as divalent tin)	140 ml./l.
45% aqueous solution of plumbous borofluoride (15 g./l., calculated as divalent lead)	35 ml./l.
Free HBF_4	110 g./l.
50% solution of o-toluidine in n-butyl alcohol	40 ml./l.
Formalin (formaldehyde 35%)	20 ml./l.
Polyoxyethylene alkyl aryl ether	4 ml./l.
Distilled water	balance

Operational conditions:

Cathode current density	1 A/dm²
Temperature	20±2° C.
Anode	90% Sn - 10% Pb alloy
Agitation	The cathode was gently rocked during plating.

The electrodeposition was effected from the above bath under the above operational conditions, thereby obtaining the desired super-bright solder coating comprising 90 percent Sn and 10 percent Pb.

EXAMPLE 8

Electrodeposition of 90 percent Sn – 10 Pb solder)

The procedure of Example 7 was repeated, but substituting the o-toluidine solution by a 40 solution of 2-methyl anil in isopropyl alcohol in the same amount by volume.

The same results as in Example 7 were obtained.

EXAMPLE 9

(Electrodeposition of 90 percent Sn – 10 percent Pb solder)

The same procedure of Example 7 was followed, but replacing the solution of o-toluidine by a 40 percent solution of 2-ethyl anil in n-butyl alcohol in the same amount.

The same results as in Example 7 were obtained.

EXAMPLE 10

(Electrodeposition of 80 percent Sn – 20 percent Pb solder)

The same procedure of Example 7 was followed, but using 90 g./l. of free HBF_4 and 15 ml./l. of formalin and using a cathode current density of 2 A./dm², 30 ml./l. of the same brightening agent and 3 ml./l. of the same surfactant.

The solder coating thus obtained was super-bright and had the expected composition of 80 percent Sn and 20 percent Pb.

EXAMPLE 11

(Electrodeposition of 80 percent Sn – 20 percent Pb solder)

The procedure of Example 10 was repeated, but substituting the solution of the brightening agent in isopropyl alcohol for that in n-butyl alcohol.

EXAMPLE 12

(Electrodeposition of 80 percent Sn – 20 percent Pb solder)

The same procedure of Example 10 was followed, but substituting the solution of o-toluidine by a 40 percent solution of 2-propyl anil in the same amount.

The same results as in Example 10 were obtained.

EXAMPLE 13

(Electrodeposition of 70 percent Sn – 30 percent Pb alloy)

The procedure of Example 7 was repeated but using 90 g./l. of free HBF_4 and 10 ml./l. of formalin and using a cathode current density of 3 A./dm², 20 ml. of the same brightener solution and 2 ml./l. of a mixture of polyoxyethylene alkyl aryl ether and polyoxyethylene alkyl amide in a ratio by volume of 3 : 1.

EXAMPLE 14

(Electrodeposition of 60 percent Sn – 40 percent Pb solder)

The procedure of Example 7 was followed, but using 70 g./l. of free HBF_4 and 10 ml./l. of formalin and using a cathode current density of 2 A./dm², 20 ml./l. of the same brightener solution and 2 ml./l. of a mixture of polyoxyethylene alkyl aryl ether ad polyoxyethylene alkyl amide in a ratio by volume of 1:1.

EXAMPLE 15

(Electrodeposition of 60 percent Sn – 40 percent Pb solder)

The procedure of Example 14 was repeated, but replacing the solution of o-toluidine by a 40 percent solution of 2-ethyl anil in isopropyl alcohol in the same amount.

The results obtained were the same as those obtained in Example 14.

As is apparent from Examples 7 to 15, the electrodeposition in these Examples was effected to see if a solder coating having the desired composition of tin and lead was obtained by varying a cathode current density used and varying the kinds and amounts of the additives used (brightening agent, surface-active agent, free HBF_4 and formalin) while keeping identical the remaining operational conditions and the compositions of the principal constituents (tin and lead) of the baths between these Examples; and the solder coatings thus obtained were the desired ones as expected.

The ratio (percent) of Sn to Pb content in a solder coating to be obtained varies with a temperature used. For instance, in some cases, the use of higher than 20° C. will increase the content of Sn, while that of lower than 20° C. will decrease it. It was thus preferable that the temperature should be kept at 20° C. ± 2° C. during the electrodeposition of Examples 4 to 9.

In connection with the surface-active agent, there was obtained a solder coating comprising 60 percent of tin and 40 percent of lead in Examples 14 and 15, using a mixture of polyoxyethylene alkyl aryl ether and polyoxyethylene alkyl amide in a ratio by volume of 1:1. If a solder coating is desired to contain more than 60 percent of tin (consequently less than 40 percent of lead) when formed by using the same procedure of any one of these Examples except for a mixing ratio between these two surfactants, the ratio will be more than 1:1, and vice versa.

The surface of solder coatings obtained by the process of this invention is much brighter than that of those obtained by the conventional processes, and the solder particles present in the former surface are more minute than those present in the latter.

In addition, using a bath according to this invention, any coating the composition of which is as desired can be formed by varying the bath in contents of a stannous and a plumbous

compound and/or by varying the current in density; and my experiences have shown that the contents of tin and lead in a solder coating formed according to this invention are accurate to within about 1 – 3 percent of those predetermined. The brightness of the coating is not necessarily enhanced in proportion to the increase of the amount of a brightening agent used in the bath, and, more particularly, the brightness will be enhanced till the concentration of the agent reaches a certain high level while it will no longer be enhanced after the concentration has exceeded the level. The use of the agent in unduly large amounts will thus cause greater consumption of the agent thereby constituting poor economy.

A bath according to this invention contains an organic brightening agent and non-ionic surface-active agent as well as a stannous and a plumbous compound, the two metallic compounds being contained in the bath in such amounts that a solder coating composed of tin and lead in the desired ratio may be formed on a basis material by the electrodeposition thereon of the tin and lead from the bath. The combined use of the brightening agent and surface-active agent allows the formation of stable micells thereof in the bath which is a strongly acidic solution and the micells function anionically whereby the concentrations of the two agents are the highest in the neighborhood of the anode during electrolysis, and on the other than the concentrations are higher at interfaces including the surfaces of the bath and cathode therein than at other portions of the bath when the surface-active agent is present in the bath. In this case the surface-active agent functions as a carrier for the brightening agent thereby to carry electrically the latter agent in the form of a compound like a complex salt with stannous and plumbous ions, to the cathode. The brightening agent thus carried to and near the cathode becomes too unstable to be kept in said form and then liberated in the bath when contacted with a solution of a high value of pH produced by electrolysis on the surface of the cathode; and, because the brightening agent which is an organic additive is hydrophobic, this agent is pushed towards the cathode from every part of the bath solution, attached to the surface of alloy of tin and lead deposited from the bath and then adsorbed in the alloy.

The adsorption of the brightening agent effected according to said mechanism mainly serves to restrict the electrodeposition from the bath thereby to obtain a solder coating composed of electrodeposits of satisfactorily oriented microcrystallinity. The thus-obtained coating has super-brightness at the surface which has never been achieved by the few conventional processes for the formation of a bright or a semi-bright coating by plating. In addition, the brightening agent can be prevented from wasteful consumption such as by decomposition because the brightening and the surface-active agent in the bath according to this invention from micells therein, and the latter agent plays more effectively the same role as that of the glue used in the conventional solder plating; and therefore there can be obtained, by electrodeposition from the bath, any coating the thickness and composition of which are respectively as predetermined.

In the practice of this invention, what suitable amounts of the brightening and the surface-active agent should be present in a bath can be readily determined by using Hull cell method and therefore these agents can be easily kept under control during plating operation. A process of this invention, thus, is superior to the conventional ones in easiness of controlling the concentrations of the agents in the bath, and in operational manners and economy which will be mentioned later. Because of its advantages as mentioned above, the process of this invention can be used for forming on a basis material a super-bright solder coating composed of tin and lead in any desired ratio, whether the basis material is to be stationarily (as in the case of usual stationary plating) or movingly (as in the case of barrel plating) plated in a bath.

In addition, a solder plate obtained by the process of this invention is constituted of electrodeposited metallic particles which are very microcrystalline and well oriented, and, therefore, the plate can subsequently be readily and completely dried thereby to prevent it from internally creating stains and from being externally adhered to by dirts during the handling of it.

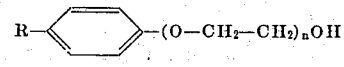
A solder plate according to this invention is remarkably improved in antirust property and brightness as compared with that composed of coarse particles according to a conventional solder plating process, and tin and lead from a bath according to this invention can also be deposited in greater thicknesses without such dendritic electrodeposits as seen in the conventional solder plate even if they are electrodeposited to a thickness of more than 2,000 microns on a basis material.

A process of this invention has further advantages that this process can be carried out at a room temperature while a solder melt-plating process necessarily carried out at a very higher temperature, basis materials which are being plated at the same time in the same bath do not adhere to one another during plating operation, a solder coating can be formed in any thickness as desired, a plating operation can be performed with a remarkably improved efficiency because barrel plating can also be carried out using a bath according to this invention, the composition of the plating bath can be controlled in any way as desired and the surface of the solder coating is less degraded than that of the conventional one.

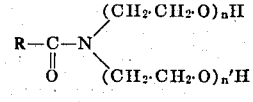
As seen from the foregoing advantages, a process of this invention is a novel and superior one which can be used for forming not only anticorrosive coating but also preliminary solder coating on various basis materials for use in many fields of industry, especially the field of electronic industry, for preparing containers for foodstuffs and for manufacturing machines and appliances, and the process will make many contributions to the various fields of industry.

What is claimed is:

1. A process for electroplating onto a basis material a bright solder of a 70 percent tin and 30 percent lead alloy which comprises electrodepositing the solder on the basis material from an aqueous acidic bath comprising () stannous borofluoride and plumbous borofluoride, (2) 90 g./l. of free HBF_4, (3) 10 ml/l of formalin, (4) 20 ml/l of a 50 percent solution of O-toluidine in n-butyl alcohol, as a brightener, and (5) 2 ml/l of a mixture of

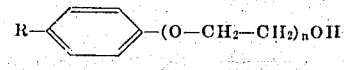


wherein R is an alkyl group having 8 or 9 carbon atoms and n is an integer of 8 to 15 and

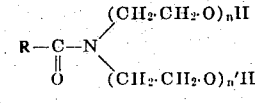


wherein R is an alkyl group having 12 to 18 carbon atoms and n and n^1 are an interger of 0 to 20 in the ratio of 3:1, with a current density of 3 A/dm^2.

2. A process for electroplating onto a basis material with a bright solder of a 60 percent tin and 40 percent lead alloy which comprises electrodepositing the solder on the basis material from an aqueous acidic bath comprising (1) stannous borofluoride and plumbous borofluoride, (2) 70 g./l. of free HBF_4, (3) 10 ml./l. of formalin, (4) 20 ml/l of a 50 percent solution of O-toluidine in n-butyl alcohol as a brightener, and (5) 2 ml./l. of a mixture of

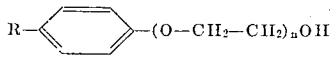


wherein R is an alkyl group having eight or nine carbon atoms and *n* is an integer of 8 to 15 and

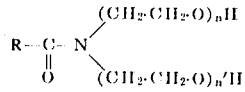


wherein R is an alkyl group having 12 to 18 carbon atoms and *n* and n^1 are an integer of 0 to 20 in the ratio of 1:1, with a current density of 2 A./dm².

3. A process for electroplating onto a basis material with a bright solder of a 60 percent tin and 40 percent lead alloy which comprises electrodepositing the solder on the basis material from an aqueous acidic bath comprising (1) stannous borofluoride and plumbous borofluoride, (2) 70 g./l. of free HBF_4, (3) 10 ml./l. of formalin, (4) 20 ml./l. of a 40 percent solution of 2-ethylanil in isopropyl alcohol, and (5) 2 ml./l. of a mixture of



wherein R is an alkyl group having eight or nine carbon atoms and *n* is an integer of 8 to 15 and



wherein R is an alkyl group having 12 to 18 carbon atoms and n and n^1 are an integer of 0 to 20 in the ratio of 1:1, with a current density of 2 A/dm².

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