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FLUXES FOR SOLDERING AND METAL COATING

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13 Claims. (Cl. 148—23)

The instant invention relates to improved fluxes for the joining and for the coating of metals and to methods for their use. More particularly, it relates to fluxes having more desirable characteristics and which are easier and more economical to use than those heretofore available.

In soldering and in metal coating processes, it is the general practice to employ a fluxing agent to remove films from and to clean the surfaces of the metals to be joined or coated. To produce a good result in soldering or metal coating, a true alloy must be formed between the metals. The presence of impurities and/or oxide coating on the surface of the metal interferes with the alloying action and results in a contaminated joint or metal coat. Fluxes are used to clean the surface of interfering impurities and oxides and to promote formation of a smooth and strong joint or coating.

At present, zinc chloride either in solid form or in a carrier such as an aqueous solution is in wide use as a flux for soldering and metal coating, but its use has several serious disadvantages. It deposits fused residues which cause corrosion unless removed. Also the fumes from zinc chloride fluxes have a very corrosive action and are dangerous.

Fluxes containing hydrohalogen salts of organic bases, such as those of hydrazine and its derivatives, alone or in combination with one or more salts of ammonia, an amine, a tetra alkylammonium halide, or an aliphatic carboxylic acid amide and the like have also been used. Although most of these type fluxes are less corrosive than zinc chloride, their fluxing activity is generally not as great as zinc chloride.

A satisfactory flux for joining and for coating metals should have the following characteristics: it is capable of cleaning the metal, removing oxides, and preventing oxidation of the metals; it should spread easily; it should be capable of being displaced from the metal by the solder or coating metal and should reduce the surface tension of the molten solder or coating metal and the metal to be soldered or coated; it should leave no corrosive residues; and its use should involve a minimum of unpleasantness and of health hazards.

It is an object of the instant invention to provide fluxes for the joining and for the coating of metals which have characteristics superior to those of the prior art.

It is a further object of the instant invention to provide improved fluxes for soldering, tinning, galvanizing, and lead coating which are highly active, highly versatile, and substantially non-corrosive, and processes for their use.

It is a further object of the instant invention to provide commercially feasible processes for producing superior quality soldering, tinning, galvanizing, and metal coating in general.

These and other objects of the instant invention will become more apparent from the following description.

We have found that the foregoing objects are obtained by use of a flux composition comprising essentially as a

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first component at least one hydrohalide salt of at least one amine from the class consisting of alkyl amines, and alkyl diamines, wherein the alkyl group contains between one and six carbon atoms per group, and ethylene polyamines having between two and five amino groups per molecule, and wherein said hydrohalide is from the group consisting of the hydrochlorides and the hydrobromides; and as a second component at least one ammonia derivative from the group consisting of ammonium chloride, ammonium bromide, and urea.

In practicing the instant invention, the alkylamine can be a mono-, di-, or trialkylamine. Typical alkyl amines which can be used include: mono- and dimethylamine; mono-, di, and triethylamine, mono-, di-, and tripropylamine; isopropylamine; mono-, di-, and tributylamine; and mono-, di-, and triamylamine. Typical of the alkyl diamines which can be used is N^1-isopropyl-2-methyl-1,2-propanediamine.

Ethylene polyamines which can be used include ethylene diamine, and polyethylene polyamines, such as diethylene triamine, triethylene tetramine, and tetraethylene pentamine.

Generally the instant compositions contain between about 50% and about 99% by weight, preferably between about 80% and about 99% by weight, of amine hydrohalide or combination of hydrohalides. In preparing the instant compositions, any one of the above amines or combination of two or more of these amines can be used. If desired, guanidine can be substituted for up to about 10% of the amine.

In preparing the instant flux compositions the hydrochloride and/or hydrobromide salt of the amine can be used, or the amine and hydrochloric or hydrobromic acid can be mixed with the other components. In the case of the polyamines, the amine polyhydrochloride and polyhydrobromide salts can be used as well as the amine monohydrochloride and monohydrobromide. When the instant combination contains more than one amine, any combination of the above hydrohalide salts of the various amines can be used. The amines or the amine hydrohalide salts can be added in substantially pure form, or as a solution or emulsion. For example, the amine or amine hydrohalide salt can be added with water; dissolved in an alcohol, such as isopropyl alcohol, methyl alcohol, ethyl alcohol, or other solvent, with or without a portion of water. For example, some amine hydrohalides such as triamylamine hydrochloride and hydrobromide are of limited solubility in water. In preparing the instant fluxes the hydrohalide can be dissolved in alcohol, such as isopropyl alcohol, or a solution containing about equal parts of alcohol and water. If desired, a conventional emulsifier can be used to aid in dissolving of the amine hydrohalide.

The ammonia derivative of the instant composition is generally present in amount between about 0.5% and about 49%, preferably between about 0.5% and about 30% by weight. Ammonium chloride, ammonium bromide, or urea can be employed singly, in a pair or all three of these ammonia derivatives may be employed, either in equal or unequal proportions by weight. In the preferred embodiment urea is used.

In the preferred embodiment of the instant invention the compositions also contain between about 0.2% and 2.0% by weight of wetting agent or combination of several wetting agents. The effect of wetting agents is well known, and amounts of wetting agents outside of this range may be employed if desired. Any one or combination of the many commercially available conventional non-ionic wetting agents which are not affected by the hydrogen ion concentration and conventional cationic wetting agents may be employed.

Specific non-ionic and cationic wetting agents which can be employed are obvious to those skilled in the art. Typical of the many well known cationic wetting agents which can be employed are the alkyl trimethyl ammonium chlorides, wherein the alkyl group is derived from fatty acids, such as soya fatty acid and coconut fatty acid. Typical examples of this class of conventional wetting agents are sold under the tradenames "Arquad S" and "Arquad C" by Armour and Company. Another type of the many commercially available wetting agents are the polyethylene glycol alkylphenyl ethers. "Nonic 300" by Sharples Chemicals, Incorporated, is a typical example of this class of wetting agents.

When the instant compositions comprising essentially the hydrohalide of the amine, the ammonia derivative, and the wetting agent are used as a flux, it is generally mixed with a carrier such as water; a solvent such as isopropyl alcohol, ethyl alcohol, methyl alcohol, and mixtures of alcohol and water; acetone; or a plastifier, such as petrolatum, rosin, and other substances conventionally employed for this purpose. The various fluxes can be used ranging from paste form to a diluted form of up to about one hundred and forty parts carrier per part of flux. Generally water is the preferred carrier.

These aqueous compositions may be substantially neutral, slightly alkaline or may contain an amount of free acid up to about 10% by weight, for example as determined by titration with methyl orange or other suitable indicator. The acidity, as well as the degree of dilution of the flux, depends upon the particular type jobs for which the flux is to be used. The factors involved in the determination of the degree of dilution used, and whether a neutral flux or a flux having a small percentage of free acid, are well known to those skilled in this art. Generally, for most work an aqueous fluxing composition which is substantially neutral or which has a small percentage of free acid is desired.

The instant flux compositions are highly versatile, and each or a combination of the various fluxes can be used for a wide variety of jobs in the fields of joining and coating of metals. These fluxes can be used for any of the conventional soldering processes such as soft soldering, in the same manner as are the presently known fluxes. That is, in use, the flux, the solder, and the metal or alloy to be treated is heated for example to between 120° F. and 900° F. to activate the flux, fuse the solder, and make the joint. After cooling, the soldered joint is smooth and strong and will not corrode.

The instant fluxes can also be used in all forms of soldering such as dip soldering, hand soldering, sweat soldering, torch soldering, convection soldering, baked soldering, mechanical soldering, and so on.

The instant fluxes can also be used in conventional galvanizing, tinning, and lead coating processes for coating a metal object with metal to obtain a desired type finish, which involves dipping the object into the flux dip and then into the molten metal. The instant fluxes can also be mixed with water and used in the conventional manner as a pre-flux dip. The instant fluxes can also be used in the conventional manner with a binder or absorption material to be filled into a hollow core of solder.

The instant flux compositions are useful in connection with the metals commonly used in soldering, tinning, galvanizing, and similar processes, such as copper, brass, bronze, zinc, tin, galvanized surfaces, iron, steel, and alloys of these. The fluxes are used where a wide variety of metal finishes are desired. For example, the following types of finishes can be obtained: in soldering processes, a bright finish of solder is obtained; in galvanizing processes, by selection of the flux, either spangel or satin finishes are obtained; in tinning and in lead coating processes, bright finishes are obtained.

In order to more fully illustrate the character of the instant invention, but without intention to be limited thereto, the following specific examples are given.

Example I

A composition was prepared by mixing 1569 grams ethylamine hydrochloride, 171 grams butylamine hydrochloride, 16 grams urea, and 6 grams of a cationic wetting agent.

For use, this mixture was combined with water in amount to obtain 133½ ounces of liquid flux. When an acid flux was desired, for example having about 3.7% free hydrochloric acid (using methyl orange indicator), the composition was mixed with 237 grams hydrochloric acid and sufficient water to obtain a total volume of 133½ ounces.

A liquid flux similar to the above flux was prepared by mixing 1200 grams of about 72.2% ethylamine, 120 grams of 95% butylamine, 2340 cc. of 20° Bé. hydrochloric acid, 16 grams of urea, 6 cc. of a non-ionic wetting agent, and 25 cc. water. This yielded 133½ ounces of liquid flux having 3.72% free acid as determined by titration using methyl orange indicator.

The above two fluxes were used in processes involving joining or coating of the following metals: stainless steel, galvanized iron, terneplate (tin-lead alloy), copper, brass, tinplate, and many other alloys. When the flux was used with stainless steel, it was not necessary to first clean the steel for sweat joints.

Depending upon preference and factors well known to those skilled in the art, the fluxes can be used in various degrees of dilution. For example, they can be used as a dip flux for copper and brass when one part flux is diluted to about 20 parts with water; for brass (new) radiators when one part is diluted to about 35 parts. For sweat soldering of terneplate, copper and brass, 1 part flux to about 20 parts water can be employed. About 1 part flux diluted with up to 40 parts water can be used for straight soldering of tinplate.

Example II

A flux was prepared by mixing 2531 grams ethylamine hydrobromide, 259 grams butylamine hydrobromide, 15 grams ammonium chloride, and 15 grams of a non-ionic wetting agent. This was diluted with water to obtain one gallon of liquid flux. If desired, hydrochloric acid can be added to obtain a flux containing free acid.

This flux can also be used with petrolatum as a carrier and an emulsifier to aid in mixing. For example, for each part by weight of the above flux, 8 parts of melted petrolatum and 0.1 part monoethanolamine oleate was added. The resulting mixture was agitated until cooled to about atmospheric temperature. Ammonium oleate, or any other ammonium soap or an amine soap of a fatty acid, can be used as the emulsifier. This flux was used in processes for joining or coating of copper, brass, tinplate, galvanized iron, and black iron.

A similar flux was prepared and had the following composition by weight:

	Percent
Ethylamine	24.8
Butylamine	2.5
Hydrobromic acid (47–48%)	62.9
Hydrochloric acid (31–32%)	9.2
Wetting agent (non-ionic)	0.3
Ammonium chloride	0.3
	100.0

The free acidity of this flux was about 0.9% computed as hydrogen chloride. One gallon of these fluxes can be diluted to between about 30 gallons and about 120 gallons, depending upon the purpose and method used. In these concentrations, no tendency to discolor or injure the work was encountered.

Example III

Another flux was prepared by mixing 286 grams ethylene diamine dihydrochloride (20 grams urea, and 4 grams of a non-ionic wetting agent.

A similar flux can also be prepared from ethylene diamine monohydrochloride or hydrobromide, rather than the dihydrochloride.

A similar flux was prepared having the following composition by weight.

	Percent
Ethylene diamine	13.9
Hydrochloric acid (20° Bé.)	48.6
Wetting agent (non-ionic)	0.4
Urea	1.8
Water	35.3
	100.0

This flux had a free acidity of about 3.7%, and one part flux is generally diluted with between about 25 and about 50 parts water for use.

When these fluxes are prepared with less acid, the resulting flux is not used in as high dilution as the above fluxes. If desired, about ¼ ounce of additional wetting agent can be added to the fluxes when the soldering job is especially greasy in type.

Example IV

A flux was prepared by combining 333 grams ethylene diamine dihydrobromide, 20 grams urea, 10 grams ammonium chloride, 10 grams ammonium bromide, and 4 grams of non-ionic wetting agent.

This mixture was dissolved in water to obtain 22 ounces of full strength flux.

This flux was diluted 30 times its volume for flux coating of copper and brass for soldering.

This flux also worked well with galvanized iron, tinplate, iron, and tereneplate either by sweat soldering or using a soldering iron. When acidified to about 4% free acid, it was also satisfactory for soldering stainless steel.

Example V

Triamylamine was neutralized with a slight excess of hydrobromic acid and allowed to stand until two layers of the resulting liquid formed. The oily layer was separated; and for each part of oily layer, 3½ parts petrolatum was added. The resulting mixture was heated until the petrolatum melted. It was then stirred and cooled.

The resulting soldering paste was used with copper, brass, tinplate, galvanized iron, black iron, and other alloys.

Example VI

A flux was prepared by the same method described in Example V except the oily layer was mixed with isopropyl alcohol instead of petrolatum. The resulting flux can be used in a wide variety of soldering and metal coating processes.

The foregoing examples show that a wide variety of improved flux compositions can be prepared in accordance with the instant invention. These compositions are highly versatile and can be applied to various metals and alloys by dipping, spraying, wiping, or any of the conventional methods for utilizing flux compositions in soldering, tinning, galvanizing, and metal coating generally. The instant compositions and processes involving their use are commercially feasible. The flux compositions are easy to work with, have excellent spreading action, and are substantially non-corrosive.

Having thus fully described and illustrated the character of the instant invention, what we desire protected by Letters Patent is:

1. A composition, which upon dilution is suitable for use as a flux for the joining and for the coating of metals and consisting essentially of, as a first component between about 80% and about 99% by weight of at least one hydrohalide salt of at least one alkyl amine from the class consisting of polyalkyl amines wherein the alkyl groups contain between one and six carbon atoms per group, and ethylene polyamines having between two and five amino groups per molecule, and wherein said hydrohalide is from the group consisting of the hydrochlorides and the hydrobromides; and as a second component, at least one ammonia derivative of the group consisting of urea, ammonium chloride, and ammonium bromide.

2. A composition suitable for use as a flux for soldering and for metal coating, consisting essentially of as the first component, between about 98% and about 99% by weight of at least one hydrohalide salt of at least one polyalkyl amine in which the alkyl groups contain between one and six carbon atoms, and wherein said hydrohalide is from the group consisting of hydrochloride and hydrobromide; and as the second component, between about 0.5% and about 1.0% by weight of at least one ammonia derivative from the group consisting of urea, amonium chloride, and ammonium bromide; and a small amount of a non-ionic wetting agent.

3. The composition of claim 2 wherein the amine salt is said hydrohalide of diethylamine.

4. The composition of claim 2 wherein the amine salt is said hydrohalide of triethylamine.

5. The composition of claim 2 wherein the amine salt is said hydrohalide of dibutylamine.

6. The composition of claim 2 wherein the amine salt is said hydrohalide of tributylamine.

7. A composition suitable for use as a flux for soldering, for galvanizing, lead coating, and for tinning, consisting essentially of between about 88% and about 93% by weight of at least one hydrohalide salt of at least one ethylene polyamine having between two and five amino groups per molecule, said hydrohalide from the group consisting of hydrochlorides and hydrobromides; between about 6.0% and about 11% by weight of at least one ammonia derivative from the group consisting of urea, ammonium chloride, and ammonium bromide; and a small amount of cationic wetting agent.

8. The composition of claim 7 wherein the amine is ethylene diamine.

9. In processes for soldering and metal coating wherein a carrier containing a fluxing composition is used in order to wet, clean, and flux said metal, the step comprising applying to said metal a fluxing composition consisting essentially of as the first component between about 80% and about 99% by weight of at least one hydrohalide salt of at least one amine from the class consisting of polyalkyl amines wherein the alkyl groups contain between one and six carbon atoms per group, and ethylene polyamines having between two and five amino groups per molecule, and wherein said hydrohalide is from the group consisting of the hydrochlorides and the hydrobromides; as a second component, at least one ammonia derivative of the group consisting of urea, ammonium chloride, and ammonium bromide.

10. In processes for soldering and for metal coating involving cleaning, wetting and fluxing of said metal with a fluxing composition, prior to contact of the cleaned, fluxed metals with another metal, the improvement comprising use of a fluxing composition, in water as the carrier, said composition consisting essentially of between about 80% and about 99% by weight of at least one hydrohalide salt of at least one amine selected from the group consisting of dimethylamine, diethylamine, triethylamine, dipropylamine, tripropylamine, dibutylamine, tributylamine, triamylamine, and N′-isopropyl-2-methyl-1, 2-propanediamine, and said hydrohalide being of the group mono- and poly hydrochlorides, and hydrobromides; at least about one-half percent by weight of urea; and between about 0.2% and about 2.0% by weight of at least one wetting agent selected from the group consisting of non-ionic and cationic wetting agents.

11. In processes for soldering and for metal coating

involving cleaning, wetting and fluxing of said metal with a fluxing composition, prior to contact of the cleaned, fluxed metal with another metal, the improvement comprising use of a fluxing composition, in water as the carrier, said composition consisting essentially of between about 80% and about 99% by weight of at least one hydrohalide salt of at least one amine selected from the group consisting of ethylene diamine, diethylene triamine, triethylene tetramine, and tetraethylene pentamine, and said hydrohalide being of the group mono- and poly-hydrochlorides and hydrobromides; at least about one-half percent by weight of urea; and between about 0.2% and about 2.0% by weight of at least one wetting agent selected from the group consisting of cationic and non-ionic wetting agents.

12. In processes for joining and coating of metals involving dipping of said metal in an aqueous pre-flux dip, the improvement comprising use of an aqueous pre-flux dip containing a flux composition consisting essentially of as the first component between about 80% and about 99% by weight at least one hydrohalide salt of at least one amine from the class consisting of polyalkyl amines wherein the alkyl groups contain between one and six carbon atoms per group, and ethylene polyamines having between two and five amino groups per molecule, and wherein said hydrohalide is from the group consisting of the hydrochlorides and the hydrobromides; as a second component, at least one ammonia derivative of the group consisting of urea, ammonium chloride, and ammonium bromide.

13. A composition suitable for use as a flux with copper and copper-containing metals, which does not corrode or discolor the metal and which does not leave harmful residues thereon, which consists essentially of as a first component at least about 92% by weight of at least one hydrohalide salt of at least one amine from the class consisting of: polyalkyl amines wherein the alkyl groups contain between 1 and 6 carbon atoms per group, and ethylene polyamines having between 2 and 5 amino groups per molecule, and wherein said hydrohalide is from the group consisting of hydrochlorides and hydrobromides; as a second component at least about one-half percent by weight of an ammonium compound from the group consisting of urea, ammonium chloride, and ammonium bromide; and as a third component between about one-fifth percent and about two percent by weight of at least one wetting agent.

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CERTIFICATE OF CORRECTION

Patent No. 2,880,126 March 31, 1959

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It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 38, for "combination" read -- composition --; line 63, for "0.2% and" read -- 0.2% and about --; column 5, line 1, for "dihydrochloride (20 grams" read -- dihydrochloride, 20 grams --; column 6, line 67, for "N′" read -- N^1 --; column 7, line 11, for "poly-hydrochlorides" read -- poly- hydrochlorides --.

Signed and sealed this 18th day of August 1959.

(SEAL)
Attest:

KARL H. AXLINE
Attesting Officer

ROBERT C. WATSON
Commissioner of Patents