United States Patent Office 2,931,713
Patented Apr. 5, 1960

2,931,713

METHOD OF AND MATERIAL FOR ETCHING ALUMINUM

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No Drawing. Application September 27, 1957
Serial No. 686,579

6 Claims. (Cl. 41—42)

This invention relates to the art of etching aluminum and alloys thereof in which aluminum is the principal or predominant ingredient and in the following description and claims the word "aluminum" is used to denote both the pure material and such alloys.

It has been known in the art that aluminum can be etched by treating it with a bath containing tartaric acid and a suitable alkali such as sodium hydroxide. However, certain difficulties have been encountered with the materials and techniques heretofore employed such for example as a certain sluggishness in the bath activity when it is first put into use as well as a rather short bath life. In the work which we have done relating to the present invention we discovered that these difficulties were very largely traceable to the calcium content normally present in most tap water which calcium, of course, reacts with tartaric acid to form calcium tartrate. This retards the etchant action of the bath especially at the start which additionally tends to prevent the tartaric acid from effectively serving as a sequestering agent.

With the foregoing brief reference to prior practice in mind, it can be said that the principal object of our invention is to provide a method of as well as materials for etching aluminum which avoids or greatly minimizes the objections mentioned.

A concomitant object of the invention is to provide a method of and materials for etching aluminum which resist the formation of sludge and the heavy incrustation of hard scale on the walls of containers, etc., normally associated with sludge-like deposits.

Another object of the invention is to provide an alkali solution for etching aluminum which tends to function at a uniform rate throughout the life of the bath even from the time that the bath is first prepared and put into use.

It is also an object of our invention to provide solutions for etching aluminum which can be employed at temperatures ranging from average living or room temperatures all the way up to boiling temperature.

The invention is based upon the discovery that all of the objects and advantages recited above may be attained by employing an aqueous bath which contains, as its essential active ingredients, an alkali from the class consisting of sodium and potassium hydroxide, tartaric acid and an alkali orthophosphate from the class consisting of mono-, di- and tri-ammonium, sodium and potassium salts in certain well-defined proportions. Baths of the kind described avoid sluggishness at start, function satisfactorily and at a uniform rate throughout their life and have a greatly reduced tendency to form sludge and also that whatever sludge is formed is of a nature which does not tend to deposit hard, undesirable scale on the metallic surfaces of the equipment used in the process.

The composition of the new etching admixture which we employ in preparing and replenishing our etching bath contains from 80 to 96% alkali, from 3 to 10% tartaric acid and from 0.025 to 2.5% phosphate ion (as PO_4) all calculated on a dry weight basis. Preferably

we employ an etchant admixture which contains from 90 to 95% alkali, from 4 to 6% tartaric acid and from 0.5 to 1% phosphate ion (as PO_4) and it is possible to form extremely useful compositions with a variety of ratios of the ingredients specified within the limits given. In this connection it should be noted that the use of less than 3% tartaric acid results in copious sludging within the bath and also that formulations containing more than 10% tartaric acid offer no particular advantages and are not economically practical. Furthermore, admixtures containing less than 0.025% phosphate ion, in our experience, have exhibited erratic behavior and non-uniform etching properties in baths made therefrom. We have also found that use of up to 2.5% of phosphate ion produced entirely satisfactory etching rates and results.

By way of example only the following formulas have proved to be excellent compositions for use with our invention.

Formula 1

	Percent
NaOH	80.00
Tartaric acid	10.00
$(NH_4)_2HPO_4$	10.00

Formula 2

KOH	96.00
Tartaric acid	3.00
$K_3PO_4.12H_2O$	1.00

Formula 3

NaOH	93.00
Tartaric acid	4.50
$Na_3PO_4.12H_2O$	2.50

In order to form the actual etching bath or solution it is merely necessary to dissolve some of the etchant composition in water, and we have found that a concentration of the etchant composition of from 1 to 12 ozs. per gallon has proved to be very acceptable in producing finely etched aluminum surfaces. As preferred practice, we use a concentration of from 4 to 6 ozs. per gallon of the etchant formulation.

As is familiar in the art the etching solution may be applied to the aluminum surfaces by flowing or spraying the solution over the work or the parts to be treated may be dipped into the bath of the etching chemicals.

During the etching procedure the baths may be employed at temperatures ranging anywhere from average living or room temperatures all the way up to boiling, but we have found that with the preferred 4 to 6 ozs. per gallon etchant formulation the optimum operating bath temperature is from 160 to 180° F. Lower temperatures require longer bath cycles to obtain the desired degree of etching whereas, higher temperatures produce more rapid action and are accompanied by considerable frothing and vigorous evolutions of hydrogen gas.

In preparing our solutions the concentrations of raw ingredients should be maintained within the following limits in order to produce baths which yield the most satisfactory results:

(1) The alkali concentration must be in a range of 6.0 to 86.4 grams per liter, and preferably, from 27.0 to 42.8 grams per liter.

(2) The tartaric acid concentration must be in a range of 0.23 to 9.0 grams per liter, and preferably, from 1.2 to 2.7 grams per liter.

(3) The phosphate ion concentration must be in a range of 0.001875 to 2.25 grams per liter, and preferably, from 0.0075 to 1.125 grams per liter.

The following are given by way of example only and represent suitable solutions or baths for use in carrying out our improved method.

Formula 4

	Ounces
NaOH	3.73
Tartaric acid	0.16
K_2HPO_4	0.11
Water, to make 1 gallon.	

Formula 5

KOH	5.60
Tartaric acid	0.24
$Na_3PO_4.12H_2O$	0.16
Water, to make 1 gallon.	

Formula 6

NaOH	1.92
Tartaric acid	0.06
$(NH_4)_2HPO_4$	0.02
Water, to make 1 gallon.	

Formula 7

NaOH	9.6
Tartaric acid	1.2
$Na_3PO_4.12H_2O$	1.2
Water, to make 1 gallon.	

It should be realized, of course, that in order to form satisfactory etchant baths, it is not necessary to utilize the exact compositions listed in the several formulations given above, but those which have been listed have proven to be very satisfactory in service.

In order to maintain our etching solutions in proper operating condition during the processing of a succession of parts or surfaces, all that it is necessary to do is to add to the bath an amount of the desired etchant composition as may be indicated as being necessary by periodic titration analyses of samples of the bath. Continued renewal or fortification of the bath by additions so determined is entirely practical and satisfactory under normal operating conditions.

Our improved baths do not sludge to any excessive extent and can be decanted free of any precipitated granular sludge that may be formed whereupon they may be continued in use with entire satisfaction. Furthermore, our baths will operate at a uniform rate from the very beginning of the time when they are placed in operation and can be used over a wide range of temperatures, extending from ordinary or average living temperature up to boiling.

We claim:

1. A composition for use in aqueous solution as an etchant bath for aluminum, said composition consisting essentially of:

	Percent by Weight	
	Minimum	Maximum
Alkali from the class consisting of NaOH and KOH	80	96
Tartaric Acid	3	10
Phosphate (calculated as PO_4) from the class of alkali orthophosphates consisting of mono, di and tri-NH_4, Na and K salts	0.025	2.5

2. A composition for use in aqueous solution as an etchant bath for aluminum, said composition consisting essentially of:

	Percent by Weight	
	Minimum	Maximum
Alkali from the class consisting of NaOH and KOH	90	95
Tartaric Acid	4	6
Phosphate (calculated as PO_4) from the class of alkali orthophosphates consisting of mono, di and tri-NH_4, Na and K salts	0.5	1

3. A bath for etching aluminum consisting essentially of an aqueous solution of from 6.0 to 86.4 grams per liter of an alkali from the class consisting of NaOH and KOH, from 0.23 to 9.0 grams per liter of tartaric acid, and from 0.001875 to 2.25 grams per liter of phosphate ion calculated as PO_4 from the class of alkali orthophosphates consisting of mono, di and tri-NH_4, Na and K salts.

4. A bath for etching aluminum consisting essentially of an aqueous solution of from 27.0 to 42.8 grams per liter of an alkali from the class consisting of NaOH or KOH, from 1.2 to 2.7 grams per liter of tartaric acid, and from 0.0075 to 1.125 grams per liter of phosphate ion calculated as PO_4 from the class of alkali orthophosphates consisting of mono, di and tri-NH_4, Na and K salts.

5. A process of etching aluminum which comprises subjecting the aluminum to the action of the bath of claim 3 until the desired degree of etching is obtained.

6. A process of etching aluminum which comprises subjecting the aluminum to the action of the bath of claim 4 until the desired degree of etching is obtained.

References Cited in the file of this patent

UNITED STATES PATENTS

2,615,846	Dvorkovitz et al.	Oct. 28, 1952
2,650,875	Dvorkovitz et al.	Sept. 1, 1953
2,653,860	Meyer	Sept. 29, 1953

OTHER REFERENCES

Versenes: Tech. Bul. No. 2, Bersworth Chem. Co., Framingham, Mass., cop. Mar. 1, 1952, sec. 1, page 9.