

1

2,796,334

ETCHING ALUMINUM

Donald L. Robinson, New Kensington, Pa., assignor to Aluminum Company of America, Pittsburgh, Pa., a corporation of Pennsylvania

No Drawing. Application February 23, 1955,
Serial No. 490,130

2 Claims. (Cl. 41—42)

This invention relates to a process for treating aluminum by etching, and particularly to chemical etching of high purity and super purity aluminum foil for use in electrolytic capacitors or condensers. The etching treatment serves to roughen and thus increase the effective surface area of the aluminum treated. Aluminum surfaces so treated permit of forming high capacity condensers.

The invention is particularly applicable to the preparation of capacitors made of very pure aluminum, but it is not confined to the treatment of any particular grade of aluminum, or aluminum in any particular form. Aluminum is usually considered of capacitor grade, and is often spoken of as "high purity," when its purity is about 99.6 to 99.8 percent or better. Attention has recently been given to using aluminum of "super purity" for capacitors, by which is meant aluminum of about 99.95 to 99.99 percent purity or better. Both the so-called high purity and super purity aluminum present difficult problems when it comes to devising satisfactory methods of etching to increase effective surface area for capacitor service.

Electrolytic etching treatments for high purity aluminum have been proposed heretofore and with them etch factors of 4 or 5 to one have been obtained. By "etch factor" is meant the ratio of effective surface area after etching to surface area prior to etching, and this is usually determined by comparing the capacity of an etched and formed capacitor with the capacity of an unetched and similarly formed capacitor of the same grade of metal. This invention concerns a simplified, non-electrolytic or chemical, etching process capable of producing etch factors, even on so-called high and super purity aluminum, much greater than 4 or 5 to 1.

Heretofore, the conventional method of etching aluminum for capacitors has been to employ "dilute" hydrochloric acid solutions for chemical etching. The most "dilute" hydrochloric acid solutions previously described for this purpose, and with which I am familiar, are solutions of 5 to 10 percent hydrochloric acid by weight, and the usual reference is to solutions of about 9 to 11 percent or more. Using a solution of 11 percent by weight of hydrochloric acid, I have been able to obtain etch factors on super purity aluminum of only a little over 1, unless the solution was used electrolytically, in which case etch factors of 4 or 5 are obtainable, as previously stated.

The addition of large amounts, e. g. 2 percent up to saturation limits, of etching catalysts, to hydrochloric acid etching solutions for aluminum capacitors also has been proposed by some workers in this art. In such cases, reference is usually made to hydrochloric acid concentrations of at least 5 percent or more, just as discussed above. However, less than 5 percent hydrochloric acid has sometimes been mentioned, when large amounts of catalysts are employed. A disclosure of a concentration of 3 percent of hydrochloric acid in such case is the lowest with which I am familiar, and such disclosure is coupled with the admonition that the hydrochloric acid

2

concentration should not be less than 5 percent if high etch factors are to be obtained.

In contrast to what has been disclosed heretofore, my invention concerns a process for etching aluminum, to increase its effective surface area, which comprises chemically etching the aluminum by immersion in the novel etching solution now to be described. I employ an aqueous solution containing only about 0.5 to 2 percent by weight of hydrochloric acid, together with about 1 to 10 percent of sodium chloride and only about 0.06 to 0.2 percent of dissolved bivalent copper. I prefer to employ a solution consisting essentially of about 1.3 percent by weight hydrochloric acid, 5 percent sodium chloride, 0.13 percent dissolved bivalent copper introduced as copper nitrate, and water. The copper content of the solution employed may be provided by introducing other bivalent copper salts, such as copper chloride or copper sulfate. However, copper nitrate is preferred as it also introduces the nitrate radical which is beneficial. Copper chloride alone may be used and it introduces no negative radical not already in the solution, but the solution is not quite as effective when copper chloride is employed as it is when copper nitrate is employed, even when an equivalent amount of nitric acid or the like is added to introduce the nitrate radical. Other copper compounds may be used, but they are less desirable because they add other unneeded radicals.

The solution employed must be maintained at a temperature of about 70 to 100° C., preferably about 80° C., and the immersion period must be about ¾ to 2 minutes, preferably about 1¼ minutes. Concentrations and operating conditions outside those stated quickly fall off in effectiveness. I have discovered that, by employing the process exactly as described, etch factors of at least 6 or 7 may be obtained on aluminum foil of capacitor grade, and etch factors of at least 8 or 9 may be obtained on aluminum foil of 99.95 to 99.99 percent purity.

For best results, I recommend pre-cleaning the aluminum surface in an alkaline solution, such as an aqueous solution of 2 or 3 percent sodium hydroxide, at about 93° C. for 20 or 30 seconds, followed by water rinsing and drying. A convenient way of making up the etching solution is to employ, per liter of solution:

Commercial hydrochloric acid of 37% strength (1.19 sp. gr.)	cc.	30
Sodium chloride	g.	50
Copper nitrate (Cu(NO ₃) ₂ ·3H ₂ O)	g.	5
Water		Balance

After etching, it is desirable to rinse in cold water, in a cold 50% nitric acid solution to remove copper from the aluminum surface, and finally in cold distilled water. The etched aluminum surface may be "formed" by a conventional filming treatment in a boric acid electrolyte to produce an electrolytic capacitor or condenser. Other forming electrolytes and various conditions of treatment may, of course, be employed. Capacitance of the formed aluminum can be conveniently determined in the forming electrolyte, using a capacitance test bridge, and etch factors can then be calculated by reference to suitable standards.

I claim:

1. A process for treating aluminum, to increase its effective surface area, which comprises chemically etching the aluminum by immersion for about ¾ to 2 minutes in a solution maintained at about 70 to 100° C. and consisting essentially of about 0.5 to 2 percent by weight hydrochloric acid, about 1 to 10 percent sodium chloride, about 0.06 to 0.2 percent dissolved bivalent copper, and water.

2. A process for treating aluminum foil for electrolytic condensers, to increase its effective surface area, which comprises chemically etching the aluminum foil by immersion for about 1¼ minutes in a solution maintained at about 80° C. and consisting essentially of about 1.3 percent by weight hydrochloric acid, about 5 percent sodium chloride, about 0.13 percent dissolved bivalent copper introduced as copper nitrate, and water.

References Cited in the file of this patent

UNITED STATES PATENTS

1,418,253	Gleockler	May 30, 1922
1,681,499	Sauer	Aug. 21, 1928
2,336,846	Clark	Dec. 14, 1943
2,446,060	Pray et al.	July 27, 1948
2,614,913	Reindl et al.	Oct. 21, 1952