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ANTIMONY PLATING

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This invention relates to an improved electrolytic solution for the electrodeposition of antimony.

It is well known to electroplate antimony upon a conducting surface. However, largely because of the tendency of antimony to form a non-soluble salt in dilute acidic solutions, it has proved difficult to provide an aqueous electrolyte for plating antimony economically and rapidly. Most of the electrolytes previously known do not operate satisfactorily at speeds above about 5 to 10 amperes per square foot of average cathode area. Some of the previously known solutions have an extremely corrosive effect upon a large number of metals so that it is often difficult to start plating before the solution has etched the surface to be plated. When a smooth and bright deposit is desired, such an etch is undesirable.

An object of the invention is to provide an improved antimony electroplating solution.

Another object of the invention is to provide an improved antimony plating solution that is capable of plating at relatively high speed.

Another object of the invention is to provide an improved antimony plating solution employing antimony trichloride.

Another object of the invention is to provide an improved antimony plating solution capable of plating a smooth and relatively bright deposit at room temperature.

These and other objects will be more readily apparent and the invention more easily understood by reference to the following detailed description.

The present invention provides a novel and improved antimony plating solution that comprises antimony trichloride and is capable of plating at room temperature at speeds of up to about 25 amperes per square foot of average cathode surface.

In general, solutions according to the invention comprise antimony trichloride, sulfuric acid, hydrofluoric acid and water in somewhat variable proportions, and may be operated at room temperature or up to about 50° C.

EXAMPLE

A preferred embodiment of the invention comprises a solution of the following composition:

Antimony trichloride	300 grams/liter
Sulfuric acid (conc.)	150 cc./liter
Hydrofluoric acid (52% sol.)	450 cc./liter
Water	Balance

Since the antimony trichloride does not readily dissolve in the acids, it is necessary to make up the solution adding water in barely sufficient amount to cause decomposition of the antimony trichloride. The product of this decomposition is a white emulsion-like form of antimony, which it is thought may be antimony oxychloride, and which immediately goes into solution in the acids. The amount of water used is relatively critical. If too much water is employed in the solution, it is likely to cause an emulsion to form which would interfere with

good electroplating results. Too small an amount of water results in the dissolving of insufficient antimony to produce good plating. To prevent an accumulation of excess water in the solution, it is desirable to dry all articles before immersing them in the solution. This may easily be done by rinsing in acetone or in any other inert water-miscible, volatile solvent.

Electroplating in the solution described may be carried out advantageously at average cathode current densities of about 10 to 25 amperes per square foot at room temperature. A direct electric current source variable from about 1 to 6 volts is employed. The current density may be increased somewhat by operating the solution at a somewhat higher temperature. However, it is preferred to operate at a temperature below about 50° C. Some increase in current density may also be satisfactorily achieved by suitably agitating the solution.

No special pre-treatment of a surface is required before plating antimony upon it from this solution. The surface to be plated should be electrically conductive and chemically clean according to the usual standards for most other generally known electroplating processes. The surface should also be dry, and should be immersed into the plating bath reasonably soon after being dried in order to prevent the formation of an oxide film that might impede the plating or affect the adherence of the plated film to the surface.

Carbon and platinum anodes are suitable for use with this solution. Antimony anodes also provide satisfactory results although care must be taken to insure sufficient surface area of such antimony anodes to prevent polarization and to maintain high anode efficiency.

The solution should be made using ingredients of the highest readily obtainable purity, "commercially pure" materials being satisfactory, and in order to insure continuous satisfactory operation, the solution should be kept reasonably clean by means of periodic or continuous filtration.

The composition of a plating bath according to the invention may be varied considerably between the general limits given below:

Antimony trichloride	200–400 grams/l.
Sulfuric acid	130–190 cc./l.
Hydrofluoric acid (52% sol.)	400–600 cc./l.
Water	Balance

Variations in the composition sometimes require a change in the rate of plating. It is believed that the preferred composition as described in the example given above provides the most advantageous plating solution and allows satisfactory operation at about the highest speed of any of the different compositions within the scope of the invention.

Antimony films deposited by the solutions of the invention are reasonably smooth and bright and have a relatively fine grain structure. The high quality of films produced by the solutions of the invention is especially evident as the film thickness is increased, films over .001" thick being smooth, bright and uniform.

In certain instances, it is desirable to make a cathodic electric connection to the article to be plated before immersing it in the solution. This appears to be desirable when plating certain metals such as, for instance, copper and iron that are subject to rapid attack by the acids in the solution, and when it is desired also to provide a plated surface having maximum smoothness. However, in plating upon many metals, or when plating on metals subject to attack in cases where maximum brightness and smoothness of the plated surface is not required, this is not necessary and the plating can be carried out according to usual methods.

There has thus been described an improved antimony

plating solution which is capable of electro-depositing smooth and relatively bright antimony films at relatively high speed.

What is claimed is:

1. An electrolytic solution comprising the following proportions:

Antimony trichloride	200–400 grams/liter
Sulfuric acid	130–190 cc./liter
Hydrofluoric acid (52%)	400–600 cc./liter
Water	Balance

2. An electrolytic solution comprising the following proportions:

Antimony trichloride	300 grams/liter
Sulfuric acid	150 cc./liter
Hydrofluoric acid (52%)	450 cc./liter
Water	Balance

3. In a process for electroplating antimony, the step which comprises electrodepositing antimony from an aqueous solution comprising:

Antimony trichloride	200–400 grams/liter
Sulfuric acid	130–190 cc./liter
Hydrofluoric acid (52%)	400–600 cc./liter

4. In a process for electroplating antimony, the step which comprises electrodepositing antimony from an aqueous solution comprising:

Antimony trichloride	300 grams/liter
Sulfuric acid	150 cc./liter
Hydrofluoric acid (52%)	450 cc./liter

5. The process step according to claim 3 in which said electrodepositing is carried out at below about 50° C. temperature, and at an average electric current density of about 10–25 amperes per square foot.

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