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[54] **METHOD OF ELECTROLESS PLATING**

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[51] **Int. Cl.** **B44d 1/18**

[58] **Field of Search** ..... 117/212, 130 E, 130, 47 A; 204/29–30

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[57] **ABSTRACT**

A thin, discontinuous film of a catalyst is deposited on a surface to be plated. The surface is rinsed with a solution which includes a wetting agent for the surface, and is then electrolessly plated with a metal.

6 Claims, No Drawings

METHOD OF ELECTROLESS PLATING

BACKGROUND OF THE INVENTION

When a metal such as nickel is electrolessly deposited on a surface which is not normally a catalyst for such deposition, the surface must first be activated by treating it with a catalyst. Although a number of metals catalyze the electroless deposition of nickel, the one most often used is palladium. A very thin, discontinuous film of palladium particles is deposited as a catalyzing medium by reducing a solution of a palladium salt which is in contact with the surface to be plated. If the palladium film is non-uniform, a non-uniform layer of nickel results.

When the palladium film is first deposited under conventional, carefully controlled conditions, it is usually substantially uniform. But, during the conventional step of rinsing with deionized water between deposition of the palladium and the subsequent nickel plating, non-uniformity of the palladium is often introduced, and this then causes difficulties with the nickel plating operation. There are often areas where nickel does not deposit and others where the nickel is too thin or too thick.

During the rinsing step, the palladium-coated surface usually becomes dewetted. When the coated surface is lifted out of the rinsing bath, droplets of water form on the surface and these tend to pull the palladium into clumps. Also, when the coated surface is immersed in the nickel plating bath, the palladium particles are further moved around on the surface. Too much nickel may be deposited on the areas where the palladium is "clumped" and too little or none where the palladium is missing.

The present invention is based on the discovery that, if a wetting agent of a particular type is included in the rinsing solution which is applied between the palladium deposition step and the nickel plating step, the original palladium deposit is not disturbed during the rinsing operation and uniformity of the nickel plating is considerably enhanced. The wetting agent must be one which does not interfere with the nickel plating operation.

DESCRIPTION OF PREFERRED EMBODIMENT

An example of the improved process of the present invention, applied to the nickel plating of silicon semiconductor devices, is as follows.

The objects being plated are crystal slices of semiconducting silicon in which a multiplicity of devices such as transistors are being fabricated. Monolithic type integrated circuits can also be made in this way. Each slice has a layer of silicon dioxide covering one surface except where ohmic contacts are to be made to electrode areas. Openings through the silicon dioxide over the electrode areas have previously been made using conventional photoresist, exposure, and developing techniques followed by etching away the unwanted areas of silicon dioxide, The remaining hardened photoresist has then been removed.

In preparing the slices to be nickel plated on the electrode contact areas, they are etched lightly with a solution such as the following:

Glacial acetic acid	950 ml/liter
48% HF	50 ml/liter
5% by wt. $PdCl_2$	2 ml/liter

The amount of $PdCl_2$ solution may be varied between about 0.5 and 10 ml/liter. If too much $PdCl_2$ is present, the deposit of palladium metal tends to become non-uniform and this leads to non-uniformity in the nickel deposit.

The amount of 48% HF can be varied between about 20 ml and 150 ml. The glacial acetic acid and the hydrofluoric acid improve the wetting of the silicon surface by the palladium solution and thus permit more uniform deposition of palladium at low concentration. This requires less palladium.

The slices are immersed in the above catalyst solution for 20 seconds at 25°C. The time can be varied between about 5 seconds and 60 seconds. The solution is reduced by the silicon and palladium deposits mostly on the silicon and not on the silicon dioxide. However, some palladium does occasionally deposit on the oxide.

Next, the slices are rinsed with a solution which contains an agent which wets silicon. An example of this solution is:

2 propanol	200 ml
deionized water	800 ml

The slices are immersed in this solution for 25 seconds at 25°C. The propanol is used as a wetting agent. Other wetting agents, such as other lower molecular weight alcohols (i.e., up to C_7) which do not interfere with the subsequent nickel deposition, may be substituted. Higher molecular weight alcohols can also be used. The presence of the wetting agent prevents water droplets from forming and thus it prevents the rather loosely held palladium from being moved around on the surface. If one of the lower molecular weight members of this series is used, (e.g., propanol or isopropanol) the amount used should be at least about 10% by volume of the rinsing solution. As the molecular weight rises, the amount of the alcohol used can be correspondingly less.

After treatment with the rinse solution, the slices are immersed in a nickel plating bath which may have a composition such as the following, per liter of solution. The solvent used is deionized water.

Potassium glycolate	78 g
Nickel sulfate	36 g
Ammonium acetate	10 g
Sodium hypophosphite	15 g
Sulfuric acid	to pH 4.8 – 4.9

The slices are immersed in this solution for 3 minutes at 80°C. The glycolate and the acetate are chelating agents. Other chelating agents may be used. The ammonium acetate is included to complex any palladium salt that may have been dragged over from the palladium catalyst solution despite thorough rinsing. The glycolate complexes the nickel. Any soluble nickel salt may be used and the concentration is not critical. Any conventional reducing agent for nickel may be used in place of the hypophosphite. Other examples are amine boranes and hydrazine. The pH range may be either 3.8 – 5.1 or 7.5 – 10.5.

If the surface being plated is not one which is capable of reducing the catalyst ion, a coating of a sensitizing agent, such as tin, must first be deposited. This must be

done, for example, when the substrate being plated is glass or a plastic.

The method applies to any metal capable of being electrolessly deposited. Cobalt and copper are examples of metals other than nickel.

I claim:

1. In a method of electrolessly depositing a metal catalyzed by a film of palladium, on a substrate surface, comprising:

depositing a thin, discontinuous film of palladium on said surface,

rinsing said surface, and

electrolessly depositing a metal on said surface, the improvement comprising

rinsing said surface, after depositing said film of palladium, in an aqueous solution containing a wetting agent that does not interfere with the subsequent electroless deposition of said metal on said surface.

2. A method according to claim 1 in which said metal is nickel.

3. A method according to claim 2 in which said wetting agent is a low molecular weight alcohol.

4. A method according to claim 3 in which said alcohol is propanol.

5. A method according to claim 4 in which said propanol is present in an amount of at least about 10% by volume of the rinsing solution.

6. A method according to claim 1 in which said substrate is silicon.

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