

EXAMPLE

The best mode of practicing the invention is exemplified by the following procedure in which one liter of precipitation solution yielded 42 grams of silver powder.

To produce a first solution, 15 mg of a gelatin colloid was dissolved in 790 ml of distilled water. The solution was acidified to a pH of approximately 0. by the addition of 1 mol (134 ml) of fluoroboric acid (HBF₄), 48% mass fraction. Then, 0.433 mol (73.5 g) of silver nitrate (AgNO₃) was dissolved in the solution, followed by the addition of 0.03 mol of anionic surfactant and 335 mg of aluminum oxide (Al₂O₃—γ) having a particle size of 0.05 μm. A second solution comprising 0.210 mol (76 ml) of tin fluoroborate concentrate (800 g/l Sn(BF₄)₂) was then added to the first solution at ambient temperature (19–21° C.).

Colloidal silver was observed to form in approximately 3 seconds, and coarsening of the silver powder continued for approximately 20 minutes. After the powder was allowed to settle in solution for approximately 10 minutes, the liquid was decanted from the powder and the powder was washed in distilled water. The silver powder was then air dried and sieved through 200 mesh resulting in powder agglomerates having a size of approximately 25 to 75 μm, and comprising individual particles having a size of from 0.2 to 2.0 μm.

The present invention, therefore, provides a metallic silver powder which is chemically precipitated through a two solution technique. The precipitated powder agglomerate is primarily (almost 80% by weight) less than 25 μm in diameter, and the individual powder particles which compose the agglomerate range in size from 0.2 to 2.0 μm. In addition to the favorable size distribution, silver particles precipitated in the presence of gelatin can be used with a minimal amount of sieving so that little work hardening is imparted to the particles. The powder can be annealed at a temperature of up to 750° C. for two hours in air with minimal sintering. The acid-assisted hand consolidation of powder produced according to the present technique is capable of producing silver compacts which are nearly 80% dense. Advantageously, a hand consolidated silver compact which comprises the powder of the present invention equals or exceeds the transverse rupture strength, shear strength, creep, toughness, corrosion resistance, microleakage, and wear properties of conventional silver amalgam.

While only certain preferred embodiments of this invention have been shown and described by way of illustration, many modifications will occur to those skilled in the art. It is, therefore, desired that it be understood that it is intended herein to cover all such modifications that fall within the true spirit and scope of this invention.

What is claimed is:

1. A method for the chemical precipitation of metallic silver powder via a two solution technique, comprising the steps of:

- forming a first solution by dissolving in distilled water an amount of a colloid sufficient to promote formation of particles of a desired size;
- acidifying said first solution by adding an inorganic or organic acid;
- dissolving in said first solution a silver salt;
- adding to said first solution an amount of alumina sufficient to inhibit clumping of particles during precipitation and to retard sintering of particles;
- adding to said first solution an amount of an anionic surfactant sufficient to disperse said alumina during precipitation and to inhibit clumping of particles during precipitation;

forming a second solution of a tin salt;

adding said second solution to said first solution to form a precipitation solution in which the ion concentration ratio of silver to tin is approximately 2.0 at a temperature appropriate to effect the chemical precipitation of silver;

agitating said precipitation solution for a period of time sufficient to precipitate silver powder agglomerates in which from 30% to 80% of the agglomerates have an agglomerate diameter of less than 25 μm, and in which said agglomerates comprise individual powder particles having a diameter of from 0.2 to 2.0 μm; and

optionally heat treating said silver powder particles.

2. A method for the chemical precipitation of metallic silver powder according to claim 1, wherein said acid is selected from the group consisting of fluoroboric acid, acetic acid, perchloric acid, phosphoric acid, sulfamic acid, and sulfuric acid.

3. A method for the chemical precipitation of metallic silver powder according to claim 1, wherein said silver salt is selected from the group consisting of silver nitrate, silver oxide, silver fluoroborate, silver fluoride, silver perchlorate, silver sulfamate, and silver sulfate.

4. A method for the chemical precipitation of metallic silver powder according to claim 1, wherein said tin salt is selected from the group consisting of tin fluoroborate, tin fluoride, and tin sulfate.

5. A method for the chemical precipitation of metallic silver powder according to claim 1, wherein said colloid is a gelatin.

6. A method for the chemical precipitation of metallic silver powder according to claim 1, wherein said anionic surfactant is 2-ethylhexyl sulfate.

7. A method for the chemical precipitation of metallic silver powder according to claim 1, wherein said first solution has a pH of approximately 0.8 after the addition of said acid.

8. A method for the chemical precipitation of metallic silver powder according to claim 1, wherein said precipitation solution has a temperature of approximately 19–21° C.

9. A method for the chemical precipitation of metallic silver powder according to claim 1, wherein said first solution has a silver salt concentration of approximately 0.4 mol/l.

10. A method for the chemical precipitation of metallic silver powder according to claim 1, wherein said second solution has a tin salt concentration of approximately 0.2 mol/l.

11. A method for the chemical precipitation of metallic silver powder via a two solution technique, comprising the steps of:

- forming a first solution by dissolving in distilled water an amount of a colloid sufficient to promote formation of particles of a desired size;
- acidifying said first solution by adding an inorganic or organic acid;
- dissolving in said first solution a silver salt;
- adding to said first solution an amount of alumina sufficient to inhibit clumping of particles during precipitation and to retard sintering of particles;