METHOD FOR CHEMICAL PRECIPITATION OF METALLIC SILVER POWDER VIA A TWO SOLUTION TECHNIQUE

The invention described herein may be manufactured, used, and licensed by the U.S. Government for governmental purposes without the payment of any royalties thereon.

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates generally to a method for the chemical precipitation of metallic silver powder. The invention relates more specifically to a method which employs a two solution technique to produce powder suitable for use as a mercury-free, metallic restorative in dental applications.

2. Discussion of the Prior Art

Conventional dental amalgams have enjoyed widespread and long-standing popularity as a result of their ability to undergo the transformation from a soft, putty-like state into a strong cohesive solid at ambient temperature, under low pressure, and within a short time duration. According to a 1993 Department of Health and Human Service report, 100 million dental restorative procedures which employed amalgam were performed in 1990. In 1991, the U.S. Bureau of Mines reported that approximately 44 metric tons (97,000 lbs) of mercury, which amounts to approximately 88 metric tons (184,000 lbs) of amalgam, was used annually by the dental profession. Assuming a current price of approximately $1,650/kg of amalgam, there is a potential annual dental industry market value of approximately $150 million.

Concern has arisen, however, over possible long-term health hazards which may be associated with the use of amalgams in dental applications. In the event the use of mercury-containing restoratives is curtailed, a mercury-free metallic restorative could capture a portion or all of the amalgam market.

Although many conventional metallic materials display mechanical properties, corrosion resistance, and biocompatibility not unlike or even better than those associated with dental amalgams, none of these materials combines these properties with the ability to undergo a transformation from a soft, putty-like state into a strong cohesive solid at ambient temperature, under low pressure, and within a short period of time. Therefore, any search for a metallic alternative to amalgams must address the problem of consolidating an easily deformable, very plastic material into a strong solid under the strict temperature, pressure, and time limitations imposed by common dental practice.

A technique for the consolidation of silver powders based on acid-assisted cold welding and intermetallic formation is described in U.S. Pat. No. 5,711,866. After treating the surface of the powder with a dilute acid to remove the naturally occurring oxide layer, the individual silver powders are cold-welded under low pressure to form a cohesive solid. Subsequently, a slurry consisting of the wet mixture of the surface treated powder particles is placed and consolidated in a prepared dental cavity. The liquid film surrounding each particle serves both to maintain a clean surface, and to constrain the micron-size particles, so that they present no inhalation danger to the patient. The powders are consolidated into a solid mass using instruments normally employed in dental practice. With silver powders having a size range of from 0.2 μm to 2.0 μm and the appropriate thermal anneal procedures, acid-assisted hand consolidation, using normal dental tools, is capable of producing silver compacts with a density of greater than 75%. Such hand consolidated silver equals or exceeds the transverse rupture strength, shear strength, creep, toughness, corrosion resistance and microleakage properties of conventional silver amalgam.

U.S. Pat. No. 3,997,328 discloses particles in the form of what is variously referred to as microcut material, lathe-cut material, platelets, or filings so that the particles are generally of non-smooth, irregular shape. The reference indicates that conventional microcutting, lathe-cutting, or filing techniques can be employed satisfactorily to obtain the irregularly shaped particles.

U.S. Pat. No. 5,318,746 discloses that metallic powders have two basic forms, either minute lathe-cut filings, or atomized, spherical particles. The lathe-cut filings are subsequently milled and sifted to produce the desired particle size. The length of particles in a commercial lathe-cut alloy might range from 60–120 μm long, 10–70 μm wide and from 10–35 μm thick. The reference discloses that spherical particles, produced by means of an atomizing process whereby a spray of tiny drops is allowed to solidify in an inert gaseous (e.g., argon) or liquid (e.g., water) environment, have a maximum size between 40 and 50 μm. The preferred size of particles to be employed in the process disclosed by the reference is from about 0.5 μm to about 50 μm.

A general need exists to provide the dental profession with a mercury-free metallic restorative as an alternative to conventional dental amalgams. The restorative must be capable of being hand consolidated while retaining critical mechanical properties, and of being placed in a dental cavity in the same amount of time as that required to place an amalgam. To make possible techniques such as the aforementioned acid-assisted consolidation, a more specific need exists for a method of preparing a silver powder of the required particle size.

SUMMARY OF THE INVENTION

It is an object of the present invention to provide a method for the chemical precipitation of metallic silver powder. It is a further object of the present invention to provide a method which produces silver powder having a particle size that facilitates its hand consolidation for use in dental restoratives.

Accordingly, the present invention advantageously relates to a method which employs a two solution technique to produce a silver powder having the desired particle size distribution. The method comprises forming a mixture of a solution of a tin salt and a solution of a silver salt in the presence of an inorganic or organic acid, alumina, an anionic surfactant, and a colloid to form a precipitation solution at a temperature and pH suitable to effect the chemical precipitation of silver. 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 an optional subsequent process step, the precipitated particles can be heat treated in air at a temperature ranging from 450 to 750° C.

A hand consolidated silver restorative prepared from 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.

BRIEF DESCRIPTION OF THE DRAWINGS

Other objects, features, and advantages of the present invention will become more fully apparent from the follow-