

erates being greater than 250  $\mu\text{m}$  in size, and 30 wt. % of the agglomerates being less than 25  $\mu\text{m}$ .

Since the larger particles negatively impact the transverse rupture strength and density of hand consolidated compacts, the solution chemistry was altered in order to promote the precipitation of smaller agglomerates. As depicted in Table 1, Solution B has the same composition as Solution A except for the inclusion of 15 ppm of a gelatin colloid. As illustrated by FIG. 3B, the addition of the gelatin dramatically alters the size distribution in favor of the smaller agglomerates which are less than 25  $\mu\text{m}$  in size. As a result of the presence of the gelatin, nearly 80 wt. % of the agglomerates is less than 25  $\mu\text{m}$  in size. It is believed that the addition of gelatin favors the nucleation of new particles and inhibits the formation of large particles, presumably by adsorbing on the surface and inhibiting growth and/or consolidation. 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.

In a subsequent process step, the silver powder produced according to the invention can be annealed prior to consolidation, thus dramatically improving the transverse rupture strength of the compact. This result has been attributed to a reduction in the yield strength of the silver powder prior to consolidation. Differential thermal analysis reveals that the thermal anneal in air oxidizes contaminants on the silver surface. The removal of the contaminant allows a uniform, protective oxide to form upon cooling. Since silver oxide is unstable at temperatures above 300° C., however, the annealing step can promote sintering of the silver powder at the elevated temperatures once the protective oxide is removed.

To determine if the precipitated powder from the present invention differed from commercially available silver powder when heat treated, a comparison was made with atomized 18  $\mu\text{m}$  size powder and a commercial 1 to 3  $\mu\text{m}$  size powder. The powders were then heat treated at 750° C. for 2 hours in air. Annealing at this temperature rendered the commercial 18  $\mu\text{m}$  and 1 to 3  $\mu\text{m}$  powders useless due to sintering into a semi-solid mat.

Sintering is not a problem, however, for powder of the present invention which has been precipitated using the Solution B chemistry summarized in Table 1. More than 95% of the Solution B precipitated powder passed through a 200 mesh sieve. In FIG. 4, where both density and transverse rupture strength of hand consolidated compacts are plotted as a function of annealing temperature, the benefit of the annealing step on the transverse rupture strength is clearly evident. On average, annealed powder results in at least a 25% increase in transverse rupture strength. The density of the consolidated material also changes significantly due to the annealing step. The density is clearly influenced by two factors: the yield strength of the silver and the extent of powder sintering. The unannealed samples show relative high density but low strength. The powders are uniform and not agglomerated, giving rise to fairly easy packing. The silver surface, however, is still contaminated so the degree of cold welding is reduced. Annealing the powder does result in some sintering and agglomeration (as verified by BET) and the density of these samples drops dramatically. As the annealing temperature is increased, the density steadily increases, presumably due to the reduced yield strength of the silver. A preferred temperature range for the thermal anneal is from 450 to 750° C., with a more preferred range of from 650 to 750° C., since the 750° C. anneal results in compacts having the highest density and rupture strength.

The silver powder obtained from the Solution B chemistry of Table 1 has all of the properties required for hand consolidation under clinical conditions. Nearly 80% of the powder is less than 25  $\mu\text{m}$  in size, and this particle size distribution is not significantly altered by the high temperature anneal. This was achieved by adding alumina, a surfactant, and a colloid to the basic precipitation chemistry. A three-factorial experimental design was used to investigate the impact that each of these components and combination of components have on powder sieving, powder handling during consolidation, and properties (density and transverse rupture strength) of hand consolidated silver compacts. The results are illustrated in Table 2. The component elements were ranked in order for i) ease of sieving; ii) consolidation density; and iii) transverse rupture strength.

TABLE 2

Addition component Evaluation from Experimental Design (Surfactant - 0.03 ml/l; Alumina - 334 mg/l; Colloid - 15 ppm).			
Rank	Ease of Sieving	Density	TRS
1	All	Surfactant Only (7.83 $\pm$ .03)	No additions (122.9 $\pm$ 14.7)
2	Colloid Plus Al <sub>2</sub> O <sub>3</sub>	Colloid Plus Al <sub>2</sub> O <sub>3</sub> (7.70 $\pm$ .07)	Surfactant Only (121.2 $\pm$ 5.5)
3	Colloid Plus Surfactant	Al <sub>2</sub> O <sub>3</sub> only (7.67 $\pm$ .14)	Colloid Plus Al <sub>2</sub> O <sub>3</sub> (117.0 $\pm$ 11.2)
4	Colloid Only	All (7.62 $\pm$ .21)	All (115.3 $\pm$ 11.5)
5	Al <sub>2</sub> O <sub>3</sub> Plus Surfactant	No additions (7.59 $\pm$ .15)	Al <sub>2</sub> O <sub>3</sub> Only (113.6 $\pm$ 3.9)
6	Al <sub>2</sub> O <sub>3</sub> Only	Colloid Only (7.56 $\pm$ .09)	Colloid Only (106.3 $\pm$ 8.4)
7	Surfactant Only	Colloid Plus Surfactant (7.54 $\pm$ .11)	Colloid Plus Surfactant (105.3 $\pm$ 12.8)
8	No additions	Al <sub>2</sub> O <sub>3</sub> Plus Surfactant (7.14 $\pm$ .03)	Al <sub>2</sub> O <sub>3</sub> Plus Surfactant (94.9 $\pm$ 3.1)

The ease of sieving coincides with the addition of the colloid to the precipitation solution, with the solution containing all of the additions rated best. Little sieving effort is required to obtain 200 mesh powder where 93% by weight of the dried as-precipitated powder passes through 200 mesh. For ease of sieving, the top four ranked additions to the precipitation solution contained the colloid. The addition of the colloid promotes powder uniformity during precipitation and significantly minimizes the need for sieving.

The highest density was achieved when only the surfactant was added to the precipitation solution and the highest transverse rupture strength value was achieved with no additions. In both cases, more extensive sieving is required just to achieve 200 mesh powder than was noted for powder precipitated from a solution containing the colloid and alumina. More extensive sieving is defined as sieving which requires a longer period of time and applied energy to break up the agglomerated clumps from the dried precipitate. The precipitated powder from the Solution B chemistry in Table 1, designated as "All," was ranked fourth in both density and transverse rupture strength. The lowest ranked component combination, alumina plus surfactant, was from Solution A. No dramatic statistical significance can be attributed to any one component or combination of components except possibly the lowest ranked combination, Solution A. This may be attributable to either the concentration level used for each component in the evaluation, or to the possible overshadowing of the effects that an individual component or combination of components may have by the technique sensitivity of the hand consolidation.