Vapor-Phase Synthesis of Fine Ag Powder and its Application to the Production of Fine Grained Sintered Ag-C Compacts†

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Abstract

An experimental investigation was carried out on the production of silver powder based on a vapor phase synthesis process. The main objective of the work was to investigate the feasibility of producing fine metal particles in a lab scale basis, which would lead to a fine grained microstructure of compacts produced from a mixture of these powders with graphite. Using a thermal plasma high temperature reactor, fine silver particles were produced at a rate of 500 g/h, with the thermal plasma power rated at 15 kW. The powders produced were characterized for particle size and size distribution by laser scattering technique, and particle size and morphology using scanning electron microscopy. Sintering experiments of compacted bodies obtained from a mixture of the powders produced and graphite powder demonstrated the feasibility of producing a material presenting a homogeneous microstructure suitable to the fabrication of high performance electrical switch contacts. Metallographic preparations of Ag/C compacts were used for the microstructure analysis using optical microscopy. The experimental apparatus involved the use of a transferred arc plasma evaporator coupled with a tubular cooling section in which the hot gases carrying the metal vapor are quenched to produce fine particles. A study was carried out to provide theoretical support to the experimental investigation. Applied to the tubular gas quenching section, a 2-D model was used to determine the temperature, velocity and species concentration fields. Fluid-dynamics was combined with a model for the nucleation and growth of particles based on the moments of particle size distribution.

1. Introduction

The many methods employed in the preparation of metal and ceramics fine particles include gas-phase processes, laser ablation, sputtering techniques, and chemical methods. In the work of Kruis et al.¹ a comparison is made between two of these methods: gas-phase and liquid based process. A number of advantages of the first are pointed out, including higher purity products, the potential to create complex chemical structures, better process and product control, economics, and less chemical segregation. Moreover, because the gas-phase method usually leads to continuous processing, it is better suited to large scale production.

The evaporation-condensation technique is one of the gas-phase methods used to produce fine particles. In order to achieve considerable evaporation rates, this method requires high temperatures and the use
of intense energy sources. In particular, the evaporation-condensation of materials using thermal plasmas has been largely studied. A wide variety of different such plasma processes have been used to synthesize ultra-fine particles. Many different types of plasma generators including non-transferred arcs, transferred arcs, high frequency induction plasmas, and combinations of these have been applied in combination with both homogeneous and heterogeneous precursors. Usually very fine particles are produced, which may include pure metals, alloys, ceramics and composite materials.

This paper reports a study on transferred-arc syntheses of silver (Ag) powder. Commercially available Ag powder is classified as microcrystalline, spheroidal, or lamellar, according to its particle shape. All three types, which differ in method of manufacture, have specific areas of use. The most important method used for the production of microcrystalline Ag powder is precipitation by reducing agents. These powders were used almost entirely in sintering technology for a long time. Today, they are also used in stoving preparations (e.g., metallization of ceramics) and in pastes used in the manufacture of heated rear windows of motor vehicles. Spheroidal Ag powder is produced by atomization of molten silver by compressed air, inert gas, water jet, or a rapidly rotating knife. These powders are mainly used in sintering technology. Lamellar particles are obtained by ball milling microcrystalline silver powder and are mainly used as an additive to paints and adhesives to which they impart electrical conductivity as a result of the good particle-to-particle electrical contact.

The investigation has the main objective to produce Ag powder finer than the powders commercially available, particularly those produced by chemical precipitation intended for sintering of electrical contacts. To this respect we look at the production of sintering compacts with a refined microstructure, which can not be achieved with the commercially available powder.

In case of the present application, the main advantage of transferred-arc thermal plasma reactors is the unlimited temperature driving force for evaporation. Temperatures of more than 10000 K can easily be sustained either by striking the arc directly to the anode work piece, or by feeding raw material in the form of powders directly into the arc. Because transferred-arcs can be operated over a broad range of gas flow rates, including very low flow rates, operating costs can be kept low. Also, control over the concentration of the evaporated species in the exhaust gas (and consequently over particle size) can be provided simply by adjusting the desired flow rate in face of a given evaporation rate. An additional advantage of transferred-arc systems is that the plasma evaporator may be scaled up over a broad range of powers simply by increasing the arc current (100–5000 A).

A modeling study concerning the nucleation and growth of fine particles from the transition of gas-to-condensed phase, adapted from a previous work involving both AlN and pure Al synthesis, was carried out with the objective to provide theoretical support to the experimental investigation.

2. Experimental

A schematic diagram of the experimental apparatus is shown in Figure 1. The reactor mainly comprised an evaporation and a cooling chamber, according to the transferred-arc concept previously reported. Inside the first chamber (270 mm i.d. × 300 mm high) Ag was evaporated from a molten bath (3–5 kg) contained in a graphite crucible that worked as the anode of a DC transferred-arc plasma. The plasma and cooling chamber were connected through a 40 mm i.d. × 175 mm long graphite tube. In all experiments argon (Ar) gas was used as plasma and Ag vapor carrier gas. Typical operating conditions are presented in Table I. Ar was fed in the arc chamber through the cathode assembly at 120 lpm flow rate. Ar or nitrogen (N2) were used as the cooling gas in three different assemblies of the cooling chamber, as shown in Figure 2. In Assembly #1, Ar was radially injected at a flow rate Q_A = 80 lpm through a 54 mm i.d. ring comprising 24 equally spaced holes of 1 mm

![Fig. 1 Schematic diagram of the plasma reactor applied for the vapor phase synthesis of Ag fine powder.](image-url)
The dimensions of the cooling chamber were 54 mm i.d. \times 240 mm long. Using the same cooling chamber dimensions and radially fed Ar as in Assembly #1, in Assembly #2 N\textsubscript{2} was additionally fed at the outlet port of the cooling chamber, at a flow rate Q\textsubscript{B}=60 lpm through an annular slit 2 mm wide. In Assembly #3 the dimensions of the cooling chamber were increased to 220 mm i.d. \times 520 mm long. In this case only N\textsubscript{2} was radially injected at a flow rate Q\textsubscript{A}=160 lpm through a 80 mm i.d. ring comprising 18 equally spaced holes of 1.5 mm diameter each. The ring was placed on the center line of the cooling chamber, at a distance of 40 mm from its entrance.

Two experiments were carried out with each of the cooling chamber assemblies described above. Only the results for experiments #1, #3 and #5 are reported. Metal particles which formed in the cooling chamber were collected in a bag filter installed after a heat exchanger. The exhaustion line also included a vacuum pump used to keep constant pressure (close to 100 kPa) inside the evaporation chamber in spite of the head loss observed through the bag filter. Internally, the plasma chamber was heavily insulated in order to minimize heat losses. The external walls of reactor were water cooled to guarantee dimensional stability.

In all experiments, steady state condition at a plasma chamber off gas temperature of 1500 K was achieved after approximately 1 hr operation, with the DC transferred-arc plasma rated at 15 kW (60 V \times 250 A). Using an optical pyrometer, bath temperatures of the order of 2400 K were measured right after shutting down the arc. At the given conditions, Ag powder was produced at a 500 g/h rate. An evaluation of the quenching conditions in terms of the average after-quenching-temperature for each of the experiments considered is shown in Table II.

The Ag powders produced were characterized for particle size distribution using laser scattering analysis and for particle size and morphology using scanning electron microscopy (SEM). In order to evaluate the degree of homogeneity of the microstructure...
resulting from the preparation of sintered compacts leaving electrical-contact characteristics, 1 kg of powder was produced from which mixtures with both graphite powder (Fisher mean diameter of 0.001 mm) and carbon black (Degussa Printex G) in a 95%Ag/5%C mass ratio were prepared. Typically, such compacts are prepared from the dry mixture of commercial Ag powder with graphite powder. Because this practice did not provide good results for the fine Ag powder we produced, its mixing with C was carried out using a suspension of the constituent powders in isopropyl alcohol. Compacts (15 mm dia. x 5 mm height) were prepared and sintered at 800°C in H2/N2 atmosphere. Their microstructure was analyzed from metallographic preparations observed in an optical microscope. For the sake of comparison, the same microstructure analysis was also carried out with an Ag/C insert commercialized as electrical contact.

3. Numerical model

The cooling region is approximated to a tubular axi-symmetric section according to the geometry and dimensions shown in Figure 3. Hot gas containing Ag vapor enters a circular port of diameter 40 mm at a flow rate Q1. Room temperature Ar gas is fed through a circumferential gap 0.1 mm wide at 175 mm far from the entrance port at a flow rate Q2. The total length of the quenching section is taken as 435 mm.

The many conservation equations describing both fluid flow and phase transition phenomena are solved in two steps: (i) first, the fields for velocity, temperature, and chemical species concentration are calculated; (ii) following, the particle nucleation and growth process arising from the mixing of a hot gas flux carrying Ag(v) and a radial jet of a cold Ar gas mixture is determined. The following conservation equations are solved in the first step, assuming laminar axi-symmetric flow:

Continuity:
\[ \nabla \cdot (\rho \mathbf{u}) = 0 \]  

Moment conservation:
\[ \nabla \cdot (\rho \mathbf{uu}) = \nabla \cdot (\mu \mathbf{v}) - \nabla p \]  

Chemical species conservation:
\[ \nabla \cdot (\rho \mathbf{wu}) = \nabla \cdot (\rho D_{ij} \nabla w_i) + S_i \]  

Energy conservation:
\[ \nabla \cdot (\rho \mathbf{wh}) = \nabla \cdot \left( \frac{k}{c_p} \nabla h \right) + \nabla \cdot \left[ \sum_i h_i \left( \frac{k_i}{c_{pi}} - \frac{k}{c_p} \right) \nabla w_i \right] + S_h \]  

The particle nucleation and growth problem is solved using the method of moments of the PSD. It is assumed that: PSD can be represented by a single log-normal distribution at any location of the modeled region; particles are spherical and transported by convection, Brownian diffusion, and thermophoresis; and particles can grow by condensation and heterogeneous reaction and coagulation. The kth-moment conservation is written as:
\[ \frac{\partial M_k}{\partial t} + \nabla \cdot (\rho \mathbf{u} M_k) = \nabla \cdot (D_{ij} \nabla M_k) + v_c \sum_i b_{ik} \sqrt{v_c} \int_0^\infty v^k \frac{\partial (G_i n_i)}{\partial v} dv + (B - D)_k \]  

The birth and death term (B – D) includes a nucleation equation that considers the effect of heterogeneous reaction in the AlN system:
\[ J = \frac{n(v_c) \beta s_i}{3S_{\Delta \theta}} \sqrt{\pi \theta / \beta} \exp \left[ -\frac{\theta^3}{27 (\ln S_{\Delta \theta})^2} \right] \]  

These two sets of conservation equations are written in the form of algebraic equations and solved using the SIMPLER computational method as developed by Patankar.

4. Results and discussion

4.1. Ag powder synthesis

Figure 4 shows the particle size distribution (PSD) of powders produced with the three different cooling
chamber assemblies. The transition which is observed between PSDs with many peaks (Exps. #1 and #3) and the practically log-normal PSD of the powder produced in Exp. #5 results from the different cooling conditions (cooling chamber geometry and gas flow rate) which were applied to the plasma chamber off-gas stream. As described before, each cooling chamber geometry was combined with a different set of cooling gas composition and flow rate. The different arrangements shown in Figure 2 were progressively implemented aiming to increase cooling intensity and to prevent particle deposition on the cooling chamber walls that occurred with higher intensity in case of assemblies #1 and #3. SEM micrographs of typical agglomerates occurrences in Exp. #3 and Exp. #5 are shown in Figure 5. The large agglomerates observed in Figure 5(a) were also observed for the powder produced in Exp.#1. A more detailed analysis of primary particles shown in the SEM micrographs indicates that they are about the same size (100 ~ 200 nm).

Considering a plasma gas flow rate Q1=20 lpm at 2000 K entering the simplified quenching section shown in Figure 3, the Ag gas-to-condensed phase transition was analyzed using numerical simulation. At first a comparison was carried out between the nucleation and growth of Al, which was the subject of a previous study⁸, and Ag at a same set of operating parameters: radial quenching Q2=7 lpm of Ar at room temperature and pressure (298 K, 100 kPa). The corresponding temperature field is shown in Figure 6.

Assuming a metal vapor concentration at the entrance of the cooling section typically observed in Al evaporation/condensation experiments of the same sort⁹, conducted with a plasma chamber off-gas temperature of 2000 K, a metal vapor mole fraction 0.002 was considered in the phase transition simulation of both Al and Ag. The results for the nucleation rate field are presented in Figure 7. It is observed that for the above conditions the more intense Al nucleation (at a rate of $10^{11}$ 1/m³.s and above) occurs near the wall, before the region of stronger cooling and along the radial jet. The Ag nucleation on the other hand occurs only in the stronger cooling region along the radial jet and in a broader volume.

The vapor pressure and surface free energy of Al and Ag as a function of temperature are graphically...
**Fig. 6**  Temperature distribution inside the quenching section simulated for a tubular quenching section of 40 mm dia., Ar gas flow rate $Q_1=20$ lpm at 2000 K, and quenching gas injection $Q_2=7$ lpm.

**Fig. 7**  Distribution of the nucleation rates simulated for Al and Ag fine particle synthesis in a tubular quenching section of 40 mm dia., Ar gas flow rate $Q_1=20$ lpm at 2000 K, quenching gas injection $Q_2=7$ lpm, and metal vapor molar fraction of 0.002.
shown in Figures 8 and 9, respectively. The higher vapor pressures of Ag contributes to its easier evaporation when compared with Al. Accordingly, experimental data gathered along the present research point out to Ag vapor concentrations which are about one order of magnitude higher than could be obtained for Al evaporation experiments carried out at a higher temperature level (1500 K in case of Ag vs. 2000 K in case of Al). With respect to the surface free energy, it is observed that its change associated with the gas-to-condensed phase transition of Ag is greater than the corresponding change observed for Al. The Gibbs free energy change for such phase transition being given by:

$$\frac{\Delta G}{kT} = -(i-1)\ln S + i^{2/3} \theta$$  \hspace{1cm} (7)

This fact explains the results shown in Figure 7 according to which, for the metal vapor concentration considered, Ag vapor travels inside the cooling chamber longer than Al before critical conditions are achieved and particles nucleation is promoted. Considering these experimental and theoretical results, a new simulation of the Ag nucleation was carried out whose analysis is presented next.

The temperature distribution obtained for the simulation of $Q1=100$ lpm at 1500 K and $Q2=60$ lpm (Ar at room temperature) is shown in Figure 10. Compared with the graph of Figure 6, a sharper temperature gradient is observed in the present condition, with the influence of the radial jet reaching the center line of the tubular cooling chamber, very close to the cold gas injection axial coordinate. Two different Ag(v) concentrations were considered for the particle nucleation and growth simulation: $x_{Ag_{in}}=0.002$ and 0.02. The results presented in Figure 11 show that, analogous to the condition depicted in Figure 7 for the Al nucleation, in case of the lower concentration situation some of the Ag particle nucleation occurs near the wall, before and along the quenching jet. On the other hand, the simulation with the higher concentration results in the predominant occurrence of nucleation at the very entrance of the quenching section. Accordingly, the distribution of the Ag(v) concentration presented in Figure 12(b) for $x_{Ag_{in}}=0.02$
Fig. 10  Temperature distribution inside the quenching section simulated for a tubular quenching section of 40 mm dia., Ar gas flow rate Q1=100 lpm at 1500 K, and quenching gas injection Q2=60 lpm.

Fig. 11  Distribution of the nucleation rates simulated for Ag fine particle synthesis in a tubular quenching section of 40 mm dia., Ar gas flow rate Q1=100 lpm at 1500 K, quenching gas injection Q2=60 lpm, and metal vapor molar fractions of 0.002 and 0.02.
shows that most of the metal vapor is consumed before the jet zone; at a same location after the quenching jet, the iso-contour lines depict vapor concentrations which are lower than the $x_{\text{Ag in}}/L_{1155}=0.002$ case. As a consequence of the lower Ag(v) concentrations, the less intense particle nucleation that occurs in the jet dominated region when $x_{\text{Ag in}}=0.02$ results in a minor modification of average particle size, as shown in the distribution of particle diameter ($d_{\text{BET}}$) evaluated in terms of the specific surface area of powder shown in Figure 13 for both $x_{\text{Ag in}}$ considered. The use of the method of moments of PSD considerably simplifies the computational treatment of the problem. Nevertheless, because the method assumes that the PSD can be represented by a single log-normal PSD anywhere in the modeling region, multi-peaks PSD results as suggested from the two nucleation regions exhibited in the plot of Figure 11(b) can not be exhibited in the computational work. The single peak PSD predicted by the model at the outlet of the cooling section for the higher vapor concentration condition is shown in Figure 14. In spite of the many simplifications, the fact that most of the Ag vapor condenses right at the entrance of the cooling section (when simulation is carried out with a $x_{\text{Ag in}}$ that corresponds to the Ag vapor concentration experimentally determined) is in good agreement with the experimental results according to which the primary particle size does not depend on the quenching conditions and cooling chamber geometry. Both experimental and modeling results suggest that the gas-to-condensed phase transition giving rise to the formation of primary particles started in the duct that connects the evaporation and cooling chambers.
before the hot gas stream could be quenched by the cold gas jets. Therefore, the definition of the primary particle size, which in principle can be controlled by quenching intensity, remained little affected by the amount and composition of the cold gas injected at the entrance of the cooling section and its geometry. Nevertheless, as can be observed from the micrographs shown in Figure 5 changing the cooling variables was effective in preventing a stronger particle agglomeration.

4.2. Ag/C compacts

Because of its well defined PSD and absence of large agglomerates, the powder produced on Exp. #5 conditions was chosen to carry 95%Ag/5%C (Ag/5%C) mixture sintering experiments. For the sake of comparison, the PSDs of this powder and of a commercially available Ag powder are shown in Figure 15.
The morphology of particle agglomerates of both commercial and plasma synthesized powders are shown in the SEM micrographs of Figure 16(a) and Figure 16(b), respectively. Commercial powder was produced by chemical precipitation. Both PSD and SEM analysis show that average agglomerate size of plasma synthesized powder is about one order of magnitude smaller than the commercial powder; primary particles on the other hand are in higher ratio (between one and two orders of magnitude).

A comparison between PSDs of graphite and carbon black powder which were used in the sintering preparation is shown in Figure 17. Particle morphology of these powders are shown in the SEM micrographs of Figure 18. The graphite particles have a flake like morphology, while the carbon black although agglomerated have a spherical primary particle morphology.

Figure 19 shows the micrographs of potassium dichromate attacked metallographic preparations of the Ag/5C commercial electrical contact insert and a sintered compact produced with the plasma synthesized Ag powder. In both cases the graphite powder shown in Figure 18(a) was used as the C bearing material. Although the compact prepared with commercial Ag powder exhibits distinct large grains with better defined grain boundary, no relevant difference in terms of C dispersion in the Ag matrix can be noticed in both preparations. On the other hand, a considerable homogeneity improvement has been achieved with the preparation obtained with the powder of Exp. #5 mixed with the carbon black powder, whose micrograph is shown in Figure 20.
5. Conclusion

Fine Ag powders were produced at a 500 kg/h rate using a transferred-arc thermal plasma synthesis reactor basically formed by evaporation and condensation separate chambers, with the plasma power rated at 15 kW. It is observed that the powders produced are formed by agglomerates which may vary in size depending upon cooling conditions and cooling chamber geometry, but are basically constituted of primary particles within about the same size range (100–200 nm). The expected influence of cooling conditions on primary particle size was not observed. Modeling analysis of this result suggests that gas-to-condensed phase transition possibly occurs inside the evaporation chamber and along the transition duct that exists between the two chambers. The primary...
The particle size of the plasma synthesized powder is considerably smaller than the one observed in the commercially available powders. Agglomerate size is about one order of magnitude smaller in case of plasma synthesized powder. Sintered compacts of 95%Ag/5%C mass ratio where prepared using the plasma Ag powder and both graphite and carbon black powders. The grain boundary of compacts prepared with commercial Ag powder (commercial electrical contact insert) are better defined, the C dispersion in the Ag matrix observed in both commercial and plasma synthesized Ag powder preparations using graphite powder is about the same. A considerably finer microstructure was observed for the plasma Ag powder when it was mixed with carbon black.

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Nomenclature

- \(B\) = birth term in the moment conservation equation
- \(C_p\) = specific heat (J/kg.K)
- \(D\) = death term in the moment conservation equation
- \(D_{i,Ar}\) = mass diffusivity (m²/s) of species \(i\) in argon;
- \(D_p\) = particle diffusivity (m²/s)
- \(G\) = surface growth rate (m³/s); Gibbs free energy
- \(h\) = enthalpy (J/kg)
- \(i\) = chemical species index (Ar, or Ag); nr. of monomers in a cluster in Eq. 7
- \(J\) = nucleation rate (1/m³.s)
- \(k\) = thermal conductivity (W/m.K)
- \(M_k\) = \(k^{th}\)-moment of particle size distribution (k=0, 1 ou 2)
- \(n\) = particle number density (1/m³)
- \(p\) = pressure (N/m²)
- \(t\) = time (s)
- \(T\) = temperature (K)
- \(r\) = radius (m)
- \(s_1\) = monomer surface area (m²)
- \(S_k\) = energy conservation equation source term
- \(S_i\) = chemical species conservation equation source term
- \(SSA\) = specific surface area (g/m²)
- \(u\) = velocity (m/s)
- \(u_{th}\) = thermoforetic velocity (m/s)
- \(w\) = mass fraction
- \(v\) = particle volume (m³)
- \(v_i\) = monomer volume (m³)
- \(v_{cr}\) = critical particle volume (m³)
- \(x_{Al}\) = Al mole fraction
- \(x_{Ag}\) = Ag mole fraction
- \(x_{Ag, in}\) = Ag mole fraction at the entrance of the cooling region
- \(z\) = longitudinal coordinate (m)

Greek letters

- \(\beta\) = impingement (or condensation) rate (1/m²/s)
- \(\theta\) = dimensionless surface energy
- \(\mu\) = viscosity (kg/m.s)
- \(\rho\) = gas density (kg/m³)

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