

# POLYMER CONDUCTIVE PASTE FORMULATION BY MODIFIED Ag<sub>2</sub>O PARTICLES

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## ABSTRACT

*This paper addresses the polymer conductive paste formulation based on modified silver oxide (Ag<sub>2</sub>O) particles. This paste is distinguished by the self-reduction of Ag<sub>2</sub>O particles to conductive Ag through a thermal process. It is suitable for the fabrication of conductive flexible structures. In addition to detailing the paste's recipe, the paper provides a comparison and assesses the effect of modifying Ag<sub>2</sub>O particles through 5 hours of milling, comparing them with large-grain particles in their original state, focusing on screen printing technology. The investigation delves into the impact of milling on particle size and distribution using as well as to verify the purity of the homogeneously created powder. Samples are manufactured using an Ag<sub>2</sub>O-based paste, screen-printed on a flexible PET Mylar® A substrate, 50 µm thick. The printed patterns are cured at 120°C to 160°C for 10 minutes while monitoring the effect of vacuum and number of printed layers on sheet resistance. Sheet resistance measurements are conducted using a 4-point probe test method. The results suggest that wet planetary ball milling is a suitable technique for modifying Ag<sub>2</sub>O particles, rendering them suitable to produce polymer conductive paste. In addition, milling produces only silver oxide fine powder particles, according to XRD patterns.*

**Keywords:** planetary ball milling, Ag<sub>2</sub>O powder particles, mechano-chemical milling principle, polymer Ag<sub>2</sub>O paste formulation

## 1. INTRODUCTION

Metallic particles, such as Au [1], Ag [2], Cu [3], and their oxides, play a crucial role as functional materials in the formulation of conductive pastes for the creation of electrical conductive layers. The development and production of new polymer conductive pastes processed at low temperatures pose a one of the challenges in the field of flexible electronics [4]. Among the mentioned metal particles, fine silver particles have garnered considerable attention due to their numerous advantages, including high chemical stability, reasonable cost, excellent electrical conductivity, and thermal conductivity [2]. Recent trends in creating and modifying conductive inks and pastes involve the addition of particles of the same metal (but with a different shape) [5], particles of other metals [6], or oxides of that metal [7].

Recent studies have shown that silver oxide (Ag<sub>2</sub>O) particles can be reduced to Ag nanoparticles using specific solvents at low sintering temperatures. The addition of Ag<sub>2</sub>O particles to the Ag paste enhances the electrical conductivity and flexibility of the layers, as the Ag<sub>2</sub>O particles act as a bridge among Ag flakes [8], [9], [10]. Exploring the existing literature reveals the availability of a silver oxide-based paste that appears to be a low-cost, self-reducing paste for creating conductive layers printed by stencil printing and sintered by a hot plate [11]. Unfortunately, there is a lack of further investigation into designing other Ag<sub>2</sub>O paste compositions for use in creating conductive flexible structures with different printing technologies. Consequently, there is an urgent need to explore the modification possibilities of Ag<sub>2</sub>O particles for their application in other printing technologies.

Various preparation methods have been reported to form metal powders, and the choice depends on the materials used. Numerous papers related to the mechanical milling of Ag<sub>2</sub>O particles by planetary ball milling have been published. Following these studies, a planetary ball

mill was selected to modify Ag<sub>2</sub>O particles for polymer paste formation [12], [13].

The mechanical planetary ball milling method is widely employed in the industry due to its advantages, such as low cost and high yield. This milling process primarily induces changes in particle size and distribution and facilitates the reduction of Ag<sub>2</sub>O to Ag. Consequently, researchers have focused on synthesizing Ag nanoparticles through dry planetary milling of Ag<sub>2</sub>O particles, which falls within the field of mechanochemistry, dealing with chemical and physicochemical changes induced by mechanical energy [14]. Planetary ball milling involves rapidly accelerating the milling balls through both centrifugal and Coriolis forces, leading to an interaction between frictional and impact forces, releasing high dynamic energies [15]. The mechanical processing of Ag<sub>2</sub>O particles generates non-equilibrium intermediate phases due to the substantial energy release during high-speed impacts, causing a chemical reaction that rapidly reduces Ag<sub>2</sub>O particles to Ag [14].

However, no previous research has investigated the modification of Ag<sub>2</sub>O particles for their use in producing polymer paste for flexible electronics without reducing them into Ag. Modifying particles is essential, as it allows the adjustment of their electrical conductivity by controlling their size, morphology, and particle distribution [16]. Changing the size of Ag<sub>2</sub>O particles could potentially increase their reactivity [17], enabling the reduction of Ag<sub>2</sub>O particles to Ag at lower processing temperatures while achieving comparable electrical conductivity results. This addresses the challenge of decreasing the processing temperature of polymer pastes.

Our goal is to modify Ag<sub>2</sub>O particles using planetary ball milling to avoid their reduction into Ag. The wet planetary ball milling method was chosen, with methanol selected as the medium. Although this method has the disadvantage of potential powder contamination by the medium [18], powders created using the wet method are

subsequently analysed for contamination. This analysis is crucial to understanding its impact on the reduction of Ag<sub>2</sub>O particles to Ag during the thermal process of the printed layer. Additionally, the utilization of screen-printing technology for sample printing is based on an analysis of particle size measurements of milled particles in their original form. The particles prepared in this manner are intended for the production of a low-temperature processed polymer paste for printing polymer conductive layers in the field of flexible electronics.

## 2. EXPERIMENTAL MATERIALS AND PRODUCTION OF Ag<sub>2</sub>O PASTE

The production of Ag<sub>2</sub>O paste involves the preparation of fine powders suitable for use in screen printing. Particle size, distribution, and purity are analysed after 5 hours of milling before utilization.

### 2.1. Principle of planetary ball milling

Planetary ball milling (PBM) is a widely utilized high-energy ball milling method employed in the synthesis of nanoparticles [19], and the production of fine metal powder [20]. As shown in Fig. 1, a planetary ball mill comprises a milling jar fixed to a rotating sun wheel. The milling jars spin around their own axis and in the opposite direction of the sun wheel. The milling balls experience rapid acceleration driven by both centrifugal and Coriolis forces arising from the different rotation directions. Throughout the milling process, impact forces occur between the balls and the material, along with the milling of the particles themselves due to frictional forces [21].

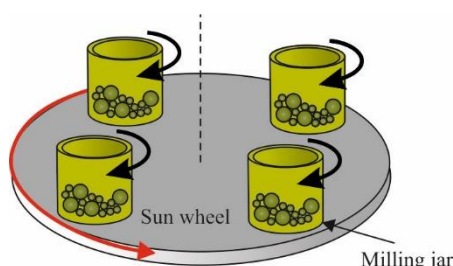


Fig. 1 Principle of planetary ball milling

Ball milling typically involves the cold welding and fracturing of particles until the saturation limit is reached [18]. Different forces introduced during milling result in the infusion of stresses into the powders, leading to the formation of lattice defects such as vacancies and dislocations between layered structures formed by cold welding. These defects enhance the surface activity of the powders and increase internal energy. Additionally, dislocations facilitate easier diffusion between layers, thereby reducing the distance between them. The cyclic welding and fracture processes contribute to the creation of a layered structure, where the layer thickness decreases with milling time, resulting in the formation of a finer powder [20], [22].

Fracturing and cold welding are integral aspects of ball milling, and maintaining a proper balance between the two is crucial for the effective functioning of alloying. In the case of ductile materials, cold welding tends to dominate,

whereas for brittle materials, fracture takes precedence. Process control agents (PCA) are introduced to achieve the desired equilibrium, with commonly used agents including methanol, ethanol, and water. These agents adsorb onto the material's surface, reducing cold welding between particles, preventing agglomeration, and facilitating particle milling. In the case of easily ignitable powders, the PCA acts as a heat sink, making wet milling more prevalent compared to dry milling. Despite its advantages, the primary drawback of this technology is the potential contamination caused by the reagents used [18].

A multitude of factors must be considered for effective high-energy planetary ball milling, including ball filling, the ball/powder ratio, and the diameter of the balls. The milling ball filling ratio (milling ball bulk volume/milling jar volume) is most effective at around 0.3, with the exact value contingent upon the milled material and the frictional forces generated by said material. At this ratio, there is no separation of the balls from the jar wall or from each other, ensuring continuous crushing of the material between the balls and between the balls and the vessel wall. However, this observation holds true only if the mill feed does not alter the movement of the balls [23]. The addition of particles increases viscosity and reduces particle mobility, consequently decreasing milling efficiency. Therefore, a viable solution involves increasing the ball diameter to provide higher kinetic energy or adding fluid to regulate viscosity [24]. The selection of the ball diameter is influenced by the rotation speed and the desired powder result. As the rotation speed increases, the ball diameter decreases [24]. Additionally, for particle synthesis requiring higher kinetic energy, ball diameters in the range of a few tens of millimetres are employed [12]. Conversely, for producing fine powders (characterized by a homogeneous particle size distribution and a diameter below 5  $\mu\text{m}$  suitable for screen printing) [25], balls smaller than 10 mm are utilized [26]. The typical ratio of balls to powder is 10:1, though a higher ratio of 20:1 can be employed to enhance milling efficiency (by increasing high-energy collisions), or a lower ratio can be used to decrease it [27].

### 2.2. Mechano-chemical milling of Ag<sub>2</sub>O powders

As mentioned earlier, the objective of our experiments is to modify the size of Ag<sub>2</sub>O powder particles (with a diameter below 5  $\mu\text{m}$ ) suitable for screen printing technology. Ag<sub>2</sub>O powder particles are known to be unstable during milling [13]. Therefore, our secondary goal is to conduct mechano-chemical milling to determine the optimal milling time and parameters that prevent the reduction of Ag<sub>2</sub>O particles to Ag. The particle modifications were executed using a wet planetary ball mill with methanol as the medium. The milling ball filling ratio is 0.2, and the ball-to-powder ratio is 8:1, chosen to mitigate high-energy collisions that could lead to the reduction of Ag<sub>2</sub>O to Ag during milling. The milling composition consists of balls, methanol, and Ag<sub>2</sub>O particles in a ratio of 8:1:1, utilizing 10g of particles for milling. All ingredients in the milling jar accounted for 44 ml, while the mill balls accounted for 29.5 ml (approximately 12.3% of the jar volume). Both the balls and the jars are made of agate. Agate balls with diameters of 10/20 mm were selected in a

count of 17/5. The parameters of the planetary ball milling are detailed in Table 1.

**Table 1** Planetary ball milling parameters

| Parameter                   | Volume |
|-----------------------------|--------|
| Sun wheel (rpm)             | 120    |
| Milling jars (rpm)          | 60     |
| Diameter of sun wheel (mm)  | 140    |
| Diameters of jars (mm)      | 80     |
| Volume of milling jars (ml) | 240    |

Following five hours of milling, the formed mass was dried at room temperature for 24 hours. Subsequently, Ag<sub>2</sub>O particles modified in this manner were sieved using a stainless-steel stencil with a mesh parameter of 400. These sieved particles can be utilized to produce the polymer paste. When the Ag<sub>2</sub>O particles are sufficiently fine and homogeneous (with a diameter less than 5 µm) and no Ag reduction occurs during the mechano-chemical processes in milling, we consider these conditions optimal for our purpose. These modified particles are then incorporated into the formulation of a polymer conductive paste, where Ag<sub>2</sub>O is primarily reduced to Ag through the thermal process.

### 2.3. Analysis of Ag<sub>2</sub>O Powder Particles

The Mastersizer 2000 was employed to measure the size and distribution of the particles, utilizing the laser diffraction technique. This method involves measuring the intensity of light scattered as a laser beam traverses through a dispersed particulate sample. The obtained data is then analysed to calculate the size of the particles responsible for creating the scattering pattern [28].

X-ray diffraction analysis was conducted using Philips X'Pert. The powder X-ray diffraction technique was employed to determine the phase composition. X-ray diffraction patterns were obtained in reflection mode using monochromatic high-intensity CuKα radiation (Lambda λ=1.54060 Å). The phase composition was analysed using the CMPR/Logic software.

### 2.4. Formulation of Ag<sub>2</sub>O-Based Paste

Ethylene glycol (≥99%) and glycerine (≥99.5%) were procured from CentralChem®. Hydroxyethyl cellulose powder with a viscosity of 145 mPa·s at a 1% concentration in H<sub>2</sub>O at 20°C was obtained from Sigma-Aldrich. Silver oxide powder particles (purity ≥ 99%) were obtained from VWR International and modified by wet planetary ball milling. All other chemicals were used without further purification. PET (polyethylene terephthalate) Mylar A film with a thickness of 50 µm from DuPont was used as the substrate. Glass tubes and plastic stirrers used in the experiments were cleaned with methanol.

The production of the paste involves mixing ethylene glycol, glycerine, and distilled water using a magnetic stirrer. Subsequently, ethyl cellulose was dissolved in this mixture to modify the viscosity of the paste and enhance adhesion between the printed layer and the substrate. Finally, Ag<sub>2</sub>O particles, comprising 50% of the total weight

of the paste, were added to the formed substance and mixed using a mechanical stirrer to create the polymer Ag<sub>2</sub>O-based paste.

### 3. SAMPLES PREPARATION

Using screen printing technology, 1, 2, and 3 layers of an Ag<sub>2</sub>O-based paste were printed on a 50 µm thick PET Mylar A substrate. Following printing, each layer underwent thermal processing in a laboratory oven, and the impact of vacuum on sheet resistance was monitored. The test sample pattern for the 4-point probe method was set at 10x20 mm.

The printed samples were subsequently thermally processed for a fixed duration of 10 minutes at temperatures ranging from 120°C to 160°C. The influence of vacuum during thermal processing on the resulting sheet resistance was investigated.

The 4-point probe test method was chosen to measure the sheet resistance of the printed layers. This method utilizes four conductors for the measurement, with two supplying current to the sample and two measuring voltage. When the sample's thickness is significantly smaller than the probe spacings, the sample can be treated as two-dimensional. In this context, the calculation of the sheet resistance can be determined by the formula:

$$R_{SH} = [\pi / \ln 2] * R \quad (1)$$

where R represents the measured resistance using the Agilent 4338B milliohm meter [29].

### 4. RESULTS

The particle size distribution of milled particles after 5 hours (S2) and in their original form (S1) was analysed. The primary objective of this analysis was to monitor whether milling resulted in a more uniform particle size distribution suitable for use in screen-printable paste. In Fig. 2, the particle size distribution of the particles in their original form is shown. Fig. 2 presents two peaks in the distribution curve, indicating two main groups of particles. These groups contain particles approximately 0.35 µm and 2.16 µm in size.

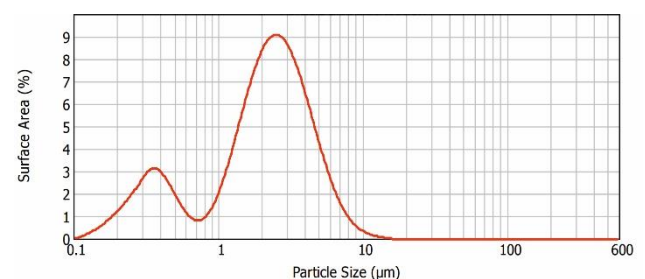


**Fig. 2** Particles size distribution of Ag<sub>2</sub>O particles in their origin form

The particle size of Ag<sub>2</sub>O particles milled by planetary ball mill in methanol for 5 hours exhibits a more homogeneous particle distribution in comparison to large-grain particles in their origin form, as shown in Fig. 3. Additionally, milling has decreased the particle size, which

ensures that clogging of the mesh by particles not occur during the printing. Thanks to the homogeneous distribution of particle sizes after milling, finely prepared particles in this way are a suitable option for their use in polymer self-reducing paste formulation.

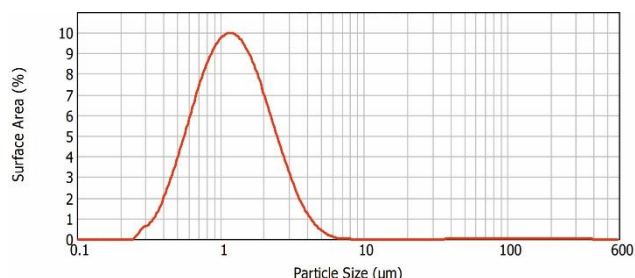


**Fig. 3** Particles size distribution of Ag<sub>2</sub>O particles after milling for 5 hours

The particle size comparison between S1 and S2 powders is listed in the following Table 1: D(0,1) - indicating that 10% of particles are smaller than a given value, D(0,5) - representing the median, and D(0,9) - signifying that 90% of particles are smaller than a specific value. Additionally, D (3,2) ("Sauter mean") denotes the diameter of a particle whose ratio of volume to surface area is the same as that of the entire sample [30].

**Table 2** The particle size comparison between S1 and S2 powders

| Samples | D(0,1) | D(0,5) | D(0,9) | D(3,2) |
|---------|--------|--------|--------|--------|
| S1      | 0.349  | 2.167  | 4.757  | 2.449  |
| S2      | 0.562  | 1.183  | 2.539  | 1.514  |

The median of the milled particles decreased from 2.167 μm to 1.183 μm, and the D(3,2) value decreased from 2.499 μm to 1.514 μm, as listed in Table 2. The table and the distribution curves clearly indicate that planetary wet milling resulted in the homogenization of particle distribution as well as size reduction.

The XRD patterns of Ag<sub>2</sub>O powder particles in their original form and after 5 hours of milling are presented in Fig. 4. The sample phase composition was identified as Ag<sub>2</sub>O (primitive cubic lattice,  $a=4.7263$  Å, space group no. 224) [31]. Following 5 hours of milling, the peaks corresponding to the Ag<sub>2</sub>O phase were not only reduced but also broadened or, in some cases, disappeared. These phenomena can be attributed to the reduction in the crystallite size of the Ag<sub>2</sub>O crystalline phase. Simultaneously, the cubic Ag phase (face-centered cubic lattice,  $a=4.0862$  Å, space group no. 225) [32] is crystallized from Ag<sub>2</sub>O through the milling process, as shown in Fig. 4. The Ag phase forms through milling in only 3% of the total composition.

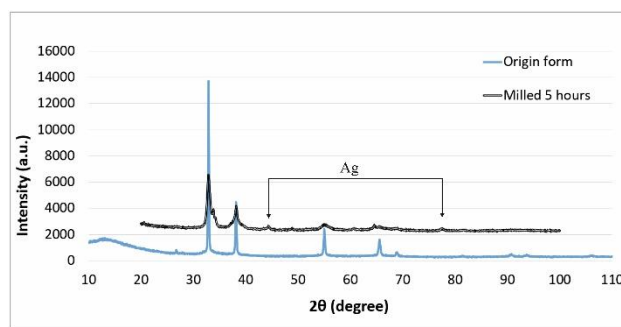


**Fig. 4** XRD patterns of Ag<sub>2</sub>O particles in their origin form and after 5 hours of milling

The sheet resistance of printed layers is a critical parameter that reflects the efficiency of thermal self-reduction of Ag<sub>2</sub>O to Ag. The findings suggest that the Ag<sub>2</sub>O-based polymer paste can chemically reduce Ag<sub>2</sub>O particles to conductive Ag during thermal processing at a low processing temperature of 120°C, as shown in Fig. 5.

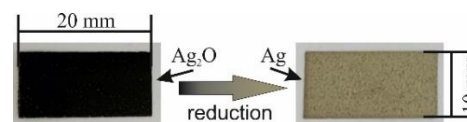


**Fig. 5** Printed Ag<sub>2</sub>O based polymer paste (left), after thermal processing (right)

The variation in sheet resistance with vacuum, processing temperature, and the number of layers is depicted in Fig. 6. The minimum sheet resistance for a single layer is recorded at 129.24 mΩ/sq under vacuum at 140 °C. For two layers, the lowest sheet resistance is observed at 81.61 mΩ/sq at a temperature of 130°C under vacuum. The optimum sheet resistance of 41.44 mΩ/sq is achieved at 120°C for three layers processed under vacuum. The results highlight that vacuum treatment reduces the sheet resistance of the printed layers, and processing three layers at 120°C is optimal for achieving a highly conductive Ag layer.

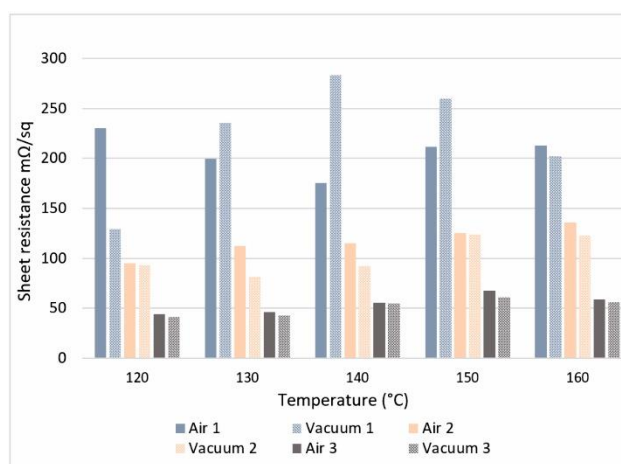


**Fig. 6** The impact of vacuum, processing temperature, and layers on sheet resistance

## 5. CONCLUSIONS

This paper details the production of a polymer self-reducing paste and the utilization of wet planetary ball milling to modify Ag<sub>2</sub>O particles within the paste. Our study demonstrates that wet planetary ball milling is a suitable method for modifying Ag<sub>2</sub>O particles without their reduction to Ag during milling, making them suitable for use in polymer paste and screen printing. Following 5 hours of milling, the particles reduced in size, reaching an average of 1.514 µm, indicating a 38% decrease. Additionally, the particle size distribution became more homogeneous. A polymer paste created from Ag<sub>2</sub>O particles prepared through this method, was screen printed and subsequently thermally processed to be reduced into highly conductive Ag layers. The results indicate that this self-reducing paste can produce highly conductive layers with a sheet resistance of 41.44 mΩ/sq through thermal processing at 120°C for 10 minutes under vacuum. Achieving this sheet resistance requires the printing of three layers of Ag<sub>2</sub>O paste.

Our experiments demonstrate that wet planetary ball milling of Ag<sub>2</sub>O particles is an effective method for modifying their size and distribution, making them suitable to produce polymer conductive self-reducing paste used in the creation of Ag conductive layers.

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## BIOGRAPHIES

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