



# Structural Analysis and Material Characterization of Silver Conductive Ink for Stretchable Electronics

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**Abstract:** Stretchable electronic systems have become more popular in various applications such as medical, fabric, flexible sensors for personalized health care, etc. There are two major parts of flexible and stretchable circuit boards that are substrate (a plastic material) and conductive ink (formulated polymer with conductive metal). According to electrical measurements, conductive ink plays a very important role in stretchable electronic equipment. The main objective of this paper is to develop a silver (Ag) based conductive ink and characterize its mechanical and electrical properties. Conductive ink is prepared by mixing an epoxy resin, cross – linking agent, additives (adhesion promoter), organic solvent, catalyst and silver flakes all together. ASTM D412 Type C dog bone shaped cutter is used to make three samples of conductive ink. The stress-strain analysis of conductive ink is carried out using universal testing machine (UTM). The conductivity is measured using two-point probe digital multi-meter. Also, the microstructural analysis, morphology and characterization are done by scanning electron microscopy (SEM). The images are taken after curing and tensile testing. The formulated ink possesses high conductivity and stretchability up to 137% strain. The achieved conductivity of the ink is  $4.167 \times 10^4$  S/m. The maximum stress before failure, yield stress, Young's and tangent moduli are calculated as 1.195 MPa, 0.86 MPa, 5.72 MPa and 2.08 MPa, respectively. The SEM analysis indicates that the distribution of silver particles is uniform and in a good density throughout the sample.

**Keywords:** Stretchable electronics, Silver conductive ink, Characterization, Tensile test, Scanning electron microscopy

## 1. Introduction

Stretchable electronic circuits (SEC) is a technology that has been under research for last 10 years. It has been improving day by day from rigid printed circuit boards (PCBs) to the bendable, twistable and stretchable circuit boards [1,2]. The stretchable electronic circuits mainly comprised of flexible substrate, flexible conductive ink and electronic devices like light-emitting diode (LED), transistors, capacitors, resistors, etc. These circuits are used in many applications such as wearable electronics [3-5], flexible and stretchable electronics [6,7], radio frequency identification (RFID) tags [8,9], health monitoring devices [10-12], image capturing machines for human motion [13,14], smart textiles [15-17], solar cells [18,19], sensors, robotic skins, and medical appliances [20-22]. Moreover, in recent studies,

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the stretchable electronic circuits are very helpful in transferring data in different wireless communication systems [23,24].

Although, various stretchable electronic devices have been developed by maintaining electrical properties under high strain. There are many procedures to fabricate the stretchable electronic circuits systems in which rigid devices combine with the stretchable conductive ink in order to get large and reversible stretching [25,26]. Various techniques of printing desired patterns on flexible substrates are available in which inkjet [27], screen [28], gravure [29] and aerosol printings [30] commonly used. The stretchability of these circuits can be determined by stretchable interconnects. The substrates are very important part in stretchable circuits. Mostly polyimide, polyethylene terephthalate, polydimethylsiloxane (PDMS) and polycarbonate are used as substrate. The other important element of a good electronic device is the conductive ink.

The important factors to control the stretchability and flexibility of electronic circuits are material and design selection for substrate and conductive ink [22,31]. In previous studies, the stretchability of a circuit can be controlled by using a proper mold for substrate and conductor because the stretchable conductor depends on the geometry of a circuit by controlling width and thickness. In order to achieve a high quality of stretchable printed circuit, the material used for conductive ink must possess high conductivity, low resistivity and good adhesion with substrate.

Conductive ink is the core element of stretchable printed circuits that has been functional in many fields such as RFID, batteries, electronic product monitors, and automotive LEDs [32-34]. The common conductive elements of ink are silver, copper, nickel, gold, carbon nanotubes or graphene, solvents and other fillers [35-37]. Many contradicting factors are taken into account during the making of conductive ink. In past years, various research has been done on the fabrication of conductive inks. The main drawbacks of developed inks were that they had short shelf lives at room temperature and required high drying and curing temperature and relatively long drying and curing times. Therefore, a good conductive ink must show high adhesion with substrate and be stable during storage. It must also have low viscosity at high metal contents [35]. The binders such as PDMS, acrylics [38], alkyls [39], cellulose [40] and resins [41] are used in conductive inks because these binders connect conductive filler elements to each other and show good electrical conductivity. However, due to the presence of binders, higher temperature and longer time are required in curing and sintering processes to remove them [42]. Hence, the flexible substrates do not bear such high temperatures [43]. Therefore, conductive ink should be cured at temperatures below 150°C or 120°C to be compatible with stretchable and flexible substrates [35].

Conductive ink consists of an epoxy resin (binder), a stabilizing agent, additives, organic solvent, conductive metal, viscosity controller and catalyst. An organic solvent determines the basic properties of ink and conductive metals like Ag ( $\sigma = 6.3 \times 10^7 \Omega^{-1} \text{m}^{-1}$ ), Cu ( $\sigma = 5.96 \times 10^7 \Omega^{-1} \text{m}^{-1}$ ), Au ( $\sigma = 4.42 \times 10^7 \Omega^{-1} \text{m}^{-1}$ ), and Al ( $\sigma = 3.78 \times 10^7 \Omega^{-1} \text{m}^{-1}$ ) [35]. A polymeric substance acts as a stabilizing agent that helps ink to be stable to aggregate and precipitate during the metallic dispersion. The different characteristics of conductive ink formulation are viscosity, surface tension and wettability [44]. The printing quality can be affected by them and they are also used to determine the drop size, drop placement accuracy, satellite formation and wetting of the substrate [45,46].

Now a days, silver-based conductive inks are used in electronic devices due to high conductivity and oxidative stability of silver [47-59]. Also, the oxide of silver remains conductive compared to copper and aluminum whose oxides are non-conductive [36,50]. There are two major factors on which the conductivity of silver-based ink depends: pack-ability and sintering. The pack-ability of silver powder is determined by its morphology and size distribution. The density of silver-based conductive ink can be obtained by pack-ability which means higher the density, higher will be electrical conductivity. Sintering process is also used to increase the conductivity of conductive ink [60]. It is done after ink applied on the substrate. This process helps to remove the protective agents from the surface of silver conductive ink and thus established a dense and conductive path throughout the printed surface. It is noted that conductivity of silver can be achieved above 150°C sintering temperature [61]. On the other hand, low temperature sintering methods give higher resistivity of conductor due to which cracks produced on the surface and electrical breakdown occurred [62]. Moreover, in order to achieve high conductivity at low sintering temperature (below 150°C or equal to 100°C), silver nano hexagonal platelets (AgNHPs) are prepared by self-assembly on graphene surface [63]. Similarly, Mo Lixin [64] produced nano silver ink of low resistivity (4.6  $\mu\Omega\text{cm}$ ) at 140°C, which is very close to the resistivity value of bulk silver (1.58  $\mu\Omega\text{cm}$ ) on paper substrate. Fernandes [65] studied the material properties of different conductive ink formulations using silver nanoparticles and obtained resistivity values from 3.3 to  $5.6 \times 10^{-6} \Omega\text{.cm}$ . The conductive ink made of silver flakes demonstrated good stretchability due to their flat shapes and large contact area between flakes [66]. Kumar [67] used silver flakes to develop conductive paste and achieved resistivity of about  $1.2 \times 10^{-3} \mu\Omega\text{.cm}$ . Also, Suikkola [68] opted silver flakes to make stretchable conductive ink that have resistivity of about 23.2  $\mu\Omega\text{.cm}$  below 150°C (about 125°C) sintering temperature and withstand strain about 74%. Therefore, silver-based conductive ink with moderate sintering and high electrical conductivity is used.

In this paper, a simple and low-cost method was presented to develop silver based conductive ink comprised of silver flakes, dispersant, and epoxy resins. The proposed formulation produced at room temperature, resulting in a longer shelf life with less curing time and temperature. The mechanical and electrical properties of silver conductive ink were examined using uniaxial tensile testing and two-point probe digital multi-meter, respectively. In addition, the surface morphology was also analyzed by scanning electron microscopy (SEM).

## 2. Materials and Methods

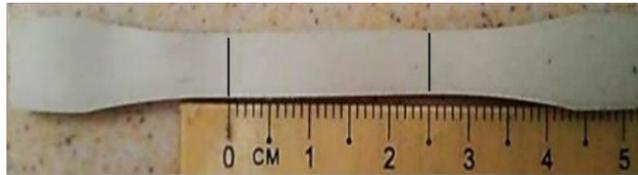
### 2.1 Materials and Ink Preparation

Poly (dimethylsiloxane) hydroxyl terminated (PDMS-OH), (3-glycidyloxypropyl) trimethoxysilane (ETMS), octamethylcyclotetrasiloxane (D4), toluene, silver flakes, acetic acid and dibutyltin dilaurate (DBDTL). The properties of these materials are describe in Table 1.

**Table 1 - Material components of conductive ink and their properties**

Components	Materials	Properties
Epoxy resin	PDMS-OH	Mol. wt. $110 \times 10^7 \text{ g/mol}$ , Viscosity $50 \times 10^3 \text{ cSt}$
Cross-linking agent	ETMS	Mol. wt. $236.34 \text{ g/mol}$ , Purity $\geq 98\%$
Organic solvent	D4	Density $0.956 \text{ g/ml}$
Viscosity controller	Toluene	Mol. wt. $92.14 \text{ g/mol}$ , Purity $99\%$
Conductive material	Silver flakes	Size: $2-3.5 \mu\text{m}$
Catalysts	DBDTL	Mol. wt. $631.56 \text{ g/mol}$ , Purity $95\%$
	Acetic acid	Purity $99\%$

A conductive dispersion was formed by mixing silver flakes with PDMS-OH epoxy resin for 24 hours using a magnetic stirrer at 280 rpm, so that, all the particles were fully dissolved in the PDMS. After that, organic solvent (D4) and cross-linking agent (ETMS) were added into the mixture and again stirred for 5-10 minutes. Finally, the catalysts were added in the solution to speed up the reaction. The mixture was then cast into a rectangular shaped container of thickness 2 mm. After curing for 24 hours at room temperature, dog bone shaped (ASTM D412 Type C standard) specimens [69] were cut from the conductive ink sheet using appropriate cutter. Fig. 1 shows the final shape of conductive ink sample.



**Fig. 1 - Silver conductive ink sample**

### 2.2 Material Characterization

The reliability and durability of developed silver conductive ink were checked by means of uniaxial tensile test, electrical conductivity measurement and SEM imaging. Many studies have been carried out on stretchable electronic circuits in terms of conductivity and stretchability. The conductivity is the reciprocal of resistivity. Mathematically, resistivity was calculated as  $\rho = RWt/L$  [70], where R is the resistance of conductive ink measured by two-point probe multi-meter, W is the width, L is the length and t is the thickness of the sample.

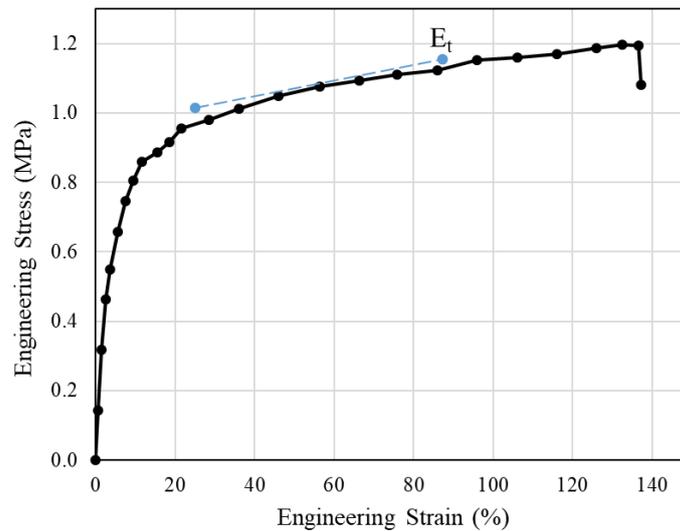
The other important challenge for a good conductive ink is to have excellent mechanical properties. This is done by performing tensile testing. Three samples of conductive ink were tested under UTM. The cross-head speed of 10 mm/minute at room temperature with 10 kN load cell was applied and samples were gripped at gauge length of 25 mm at room temperature. The experimental findings were used to measure the values of engineering stress and strain.

Another measurement technique, named as scanning electron microscopy (SEM) was used to detect the damages, defects and stresses produced in conductive ink after applying load. The surface morphology of prepared conductive ink was also carried out before undergoing tensile test.

## 3. Results and Discussions

The engineering stress-strain curve is obtained by uniaxial tensile testing as shown in Fig. 2. It is noted that the maximum stress and yield stress for formulated silver conductive ink before failure are 1.195 MPa at 137% strain and 0.86 MPa respectively. It has two slopes of linear and non-linear curves. The stress-strain curve shows the non-linearity of conductive ink because of the presence of polymeric substance PDMS. The modulus of elasticity (E) is determined by taking the slope of a tangent to the stress-strain curve through the origin. It is useful by considering minimal amount of changes in stress. The stiffness of the material in inelastic region is described by calculating the tangent modulus. The tangent modulus ( $E_t$ ) is the slope of line tangent to the stress-strain curve at a point of interest in the plastic region [71]. It can be calculated at the various points depending on their positions in the plastic region. In this study, the

tangent modulus is also computed due to the non-linear behavior shown in stress-strain diagram. The calculated values of Young's and tangent moduli are 5.72 MPa and 2.08 MPa, respectively.



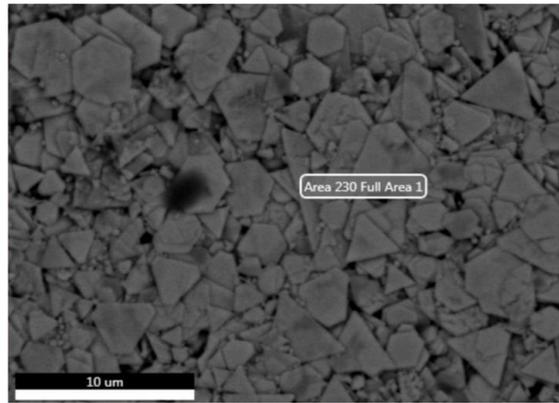
**Fig. 2 - Engineering stress-strain curve of silver conductive ink**

The stretchability, electrical and elastic properties of silver conductive ink also depend on the weight percentage of silver particles use in formulation. The proposed formulation of silver conductive ink has approximately 70% (volume fraction) of silver particles. The conductive ink with higher silver contents possesses higher conductivity and minimum elongation at rupture. The addition of more silver particles also increases the viscosity of the ink due to which the sample becomes more fragile [72]. In order to increase the stretchability of conductive ink, the amount of elastomer can be increased but as a result a weak bonding between silver particles and PDMS will occur that allow rapid increase in resistivity. The resistivity and conductivity of current formulation are obtained (@  $R = 0.1 \Omega$ ,  $W = 6 \text{ mm}$ ,  $t = 1 \text{ mm}$  and  $L = 25 \text{ mm}$ ) as  $0.024 \times 10^{-3} \Omega\text{m}$  and  $4.167 \times 10^4 \Omega\text{m}$ , respectively. The formulated conductive ink possesses higher conductivity and lower resistance because of the two reasons. One is the usage of larger size silver flakes in this study due to which the contact area between flakes and PDMS increases while on the other hand, the application of correct dispersant with proper amount helps in the dispersion of silver particles uniformly [4]. The organic solvent (polymer stabilizer) during sintering process holds the silver flakes dispersed in the ink and evaporates from the solution which results in the reduction of conductor volume. Due to the shrinkage of conductive ink, the adjacent silver flakes started attracting towards each other and contact area between the flakes increased, therefore, conductivity of ink increased. The recent study on silver based conductive ink, carried by Zhong [73], shows that the silver ink can stretch up to 47% strain, which is lower than our current formulation. Hence, our silver ink formulation exhibits high conductivity with the maximum stretchability up to 137%. The micro-structure of silver flakes in conductive ink can be seen in Fig. 3 which shows that the silver particles are uniformly rooted in the elastomeric copolymers.

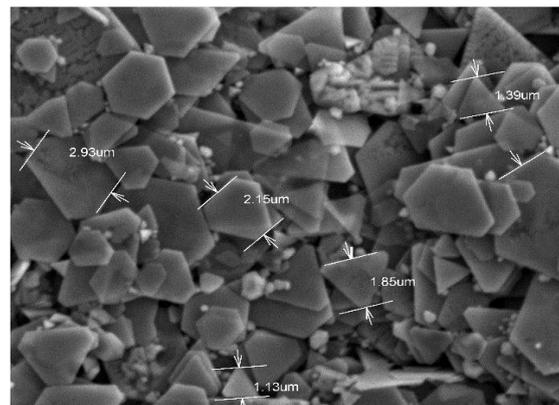
Finally, the energy-dispersive x-ray spectroscopy (EDX) of silver conductive ink is carried out as shown in Fig. 5. The highest peak in the graph shows the maximum amount of silver particles present in the formulation of conductive ink. Approximately 93.69% by weight silver particles are present in the sample. The distribution and size of silver particles under SEM scanning after curing at room temperature is illustrated in Fig. 4. It can be seen that silver particles are in the flake shape and the size varies from  $1 \mu\text{m}$  to  $3 \mu\text{m}$ .

#### 4. Conclusion

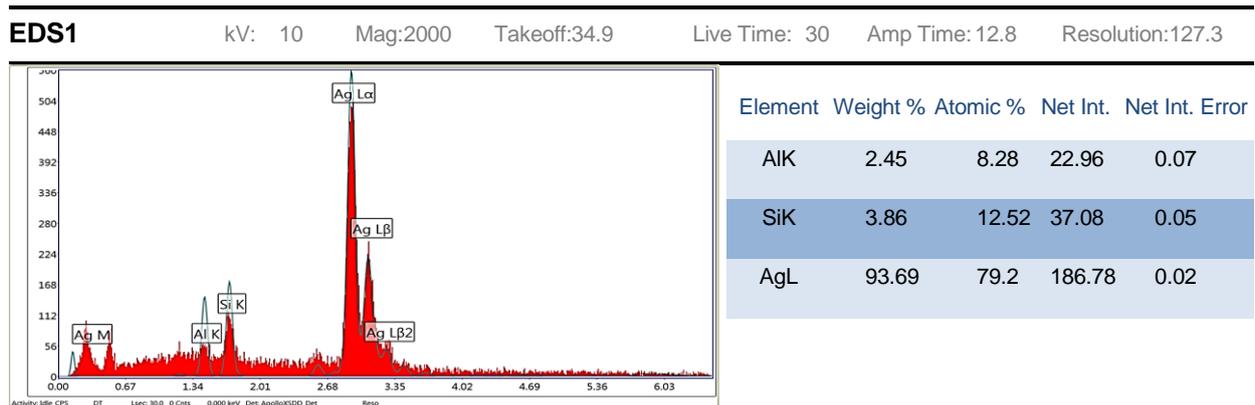
The conductive ink develops using silver flakes and PDMS-OH epoxy resin in this study. ASTM D412 type C standard is used for tensile testing of the ink. As a result, the maximum stress before failure and yield stress are obtained as 1.195 MPa and 0.86 MPa, respectively. The formulated ink has achieved maximum stretchability up to 137% with the modulus of elasticity 5.72 MPa and tangent modulus 2.08 MPa. The ink possesses higher conductivity, i.e.,  $4.167 \times 10^4 \text{ S/m}$ , due to the presence of large size of silver flakes with higher solubility in organic solvent. Finally, the EDX analysis shows the maximum amount of silver flakes, 93.69% by weight, present in the formulation, whereas, SEM provides the uniform distribution of silver flakes of size varies from 1 to  $3 \mu\text{m}$ .



**Fig. 3 - Microstructure of silver flakes**



**Fig. 4 - Size and distribution of silver particles under SEM**



**Fig. 5 – EDX spectroscopy of silver conductive ink**

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