Challenges and Opportunities in Direct Write Technology Using Nano-metal Particles

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Abstract

Direct write technology assists the electronic industry in their effort to miniaturize electronic circuits and enhance speed of printing capability. The technology allows printing various patterns without employing a mask or a resist with an enhanced speed with the aid of computer.

This paper describes the current status of the synthesis of conductive inks, a key to success in this technology. There are numerous factors to overcome in manufacturing inks which meet all the necessary conditions of conductivity, viscosity, and stability. In order to achieve the required conductivity, nano-particles used in the ink have to be made from gold, silver or copper. Inherent problems involved in these metal powders include high melting point, coagulation, impurities, cost, and, in the case of copper, oxidation. The direction of research currently being carried out in meeting and overcoming various challenges in this technology is reviewed and discussed. Other salient applications of nano-sized metal powders are also briefly examined.

Keywords: nano metals, direct write, catalysis, reduction, inverse micelle

Introduction

The power of nano-technology was first mentioned at an international conference in 1959 by Dr. Richard Feynman, a Physics Nobel Laureate when he commented on nano-science in relation to atomic theory. It was suggested that one day all of the information known to the world will be stored in a space of the size of a grain. The significance of this statement was hardly comprehended at the time, but as time progresses many people feel that we are approaching closer to the reality of the nano-world.

The term “nano” is used in conjunction with objects of a size between 10Å and 1000Å, meaning 1 nm and 100 nm respectively. One nanometer is $10^9$ meters which is too small for most people to grasp its meaning and potential applications. In order to get perspective on this measurement, let us imagine a comparison between an object of micro-size and an object of nano-size in terms of a comparison between the wave-length of ultra-violet light and the wave-length of X-rays. When the size of an object is comparable to the wave-length of infrared and visible light, the size is referred to as micro-size, which we are more accustomed to and is often known as the colloidal size.

When the size of an object is in the nano-size range, the characteristics and behavior of the material is changed dramatically. This might be illustrated with silver, which has an atomic weight of 108, an atomic diameter ($b$) of 2.9 Å, and a density ($\rho$) of 10.5 g/cm$^3$. A cube if silver 1 cm on a side would have a surface area of 6 cm$^2$, or a specific surface of 0.6 cm$^2$/g. The specific surface of a cubic or spherical particle is simply given by $6/\rho d$ where $d$ is the diameter of the particle. For a silver particle 1 $\mu$m in diameter, the specific surface is $6 \times 10^7$ cm$^2$/g, whereas that of a particle 10 nm (0.01 $\mu$m) in diameter would increase sharply to $6 \times 10^5$ cm$^2$/g. If a solid is made up of atoms of linear dimensions, $b$, then the molecular volume is approximately $b^3$ and the fraction of atoms in the surface layer is approximately $b^2$ and the fraction of atoms in the surface layer is approximately $6(b/d)$. Thus, for a 1-mm particle, about 0.2 % of the atoms will be in the surface, but in the case of a 10-nm particle about 20 % of the atoms lie in the surface. So for the 1-cm cube, only 2 or 3 atoms in ten million are surface

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atoms, but when divided into $10^{12}$ particles of 1-μm, one atom in 450 is a surface atom. At 10 nm, one silver atom in five is a surface atom, and surface effects dominate. So when dealing with nano-particles, we must be concerned with large numbers of particles, extremely small size, large specific surface area, and particles dominated by surface effects.

In 1856, Michael Faraday first prepared colloidal gold by reducing an aqueous solution of gold chloride with phosphorous to yield a ruby-colored liquid, proving that he had finely divided gold particles. He found that a small addition of salts to the solution changed the color to blue, and studied various phenomena associated. More modern work shows that gold nano-sized particles exhibit red. Gold nano-sized particles exhibit red, orange, and purple colors depending upon their size and shape, and the melting point of gold can be lowered from 1060 K for bulk gold, to about 300-400 K when the size becomes 5-10 nm. In addition, chemical reactivity changes significantly when the size of particles enters in the nano-size range and many nano-sized particles are used in conjunction with catalytic reactions.

There has been a great deal of interest in the synthesis of conductive ink using nano-sized metal powders since such ink finds numerous applications in the electronic industry. Numerous attempts have been made to produce conductive ink using primarily gold and silver nano-sized particles but commercial production of such an ink has not yet occurred. If successful, the applications of the ink are numerous. Examples can be found in display backplanes, radio frequency identification (RFID) antennas and tags, printed sensors, smart packages, e-paper, advanced layered electronics and many more.

Micro-fabrication of electronic components at the submicron scale will also help industry in its effort to miniaturize electronic circuits. Many researchers believe that the direct write technology will undoubtedly revolutionize the electronic industry. The term direct write process represents a printing process in which electronic circuit patterns are written directly from a pen-like system without going through a series and the tedious process of fabricating the patterns with masking and etching. The direct write process is flexible, versatile and accurate to a high degree of precision since a computer is an essential part of the process. Manufacturing sub-micron sized electronic circuits using traditional lithographic techniques is a very time-consuming and expensive process.

Reduction of metal ions into their elemental state in an aqueous solution is routinely carried out by hydrometallurgists. The skills and fundamental techniques used by this branch of metallurgy can easily be applied to manufacturing nano-metal powders in solution with a few significant variations. In this paper, the authors will attempt to introduce and discuss how such small metal powders can be synthesized and the challenges one faces during the process of synthesizing such nano-sized metal powders.

**Synthesis of Nano-Metal Powders**

Various methods are used to synthesize nano-metal powders. Four commonly used methods are given below:

- Laser/Plasma Method
- Electro-Chemical Method
- Vacuum Decomposition Method
- Wet Method

**Laser/Plasma Method**

This method uses a metal target bombarded with high energy with an intensity flux greater than $10^7$ watts/cm$^2$. As a result, the atoms on the surface of the target will generate plasma gas. The temperature of the system can rise up to 10,000 deg-K and about $10^4$-10$^5$ atoms on the 0.01 cm$^2$ target area are generated in $10^{-8}$ seconds. Atoms produced in such a system are usually in nano-size but the cost of the process is usually very high.

Metal gas produced in such a system can then be cooled off and various sizes of nano-metal powders can be manufactured by adjusting the rate of cooling. In this process, Ar or N$_2$ gas is used as a carrier gas to prevent oxidation.

**Electro-Chemical Method**

This process method is the same technique used in the electrochemical deposition of metal ions in aqueous solution often used in hydrometallurgical processes. In this method, metal salts are dissolved in aqueous media which is then subjected to an electrical field. Positively charged metal ions will be attracted to the cathode and form elemental metal by receiving electrons from the electrode.

$$M^{+} + ze \rightarrow M$$

The metals collected on the surface of the cathode are separated from it and broken into various sizes. It
is very difficult to control the size of metal powders generated in this fashion, however. Principles involved in this process are described well in common references used in hydrometallurgy.\textsuperscript{3,4}

**Vacuum Decomposition Method**

This method uses vacuum and heat to decompose metal salts. Size control is also a problem with this method.

\[ \text{MCl}_2 + \text{heat} \rightarrow \text{M} + \text{Cl}_2 \]

Information on the temperature of decomposition of various metal salts is readily available in the literature.\textsuperscript{5}

**Wet Method**

This is the most common method in the synthesis of nano-metal powders. This method is a relatively easy method and also very effective. Metal salts are dissolved in liquid media, and then a reducing agent is introduced to the system to reduce metal ions to elemental metal. There are, in general, two types of carrying out this method. One is to reduce metal ions in solution by adding a reducing agent directly into the system. The other method is very similar to the above process but the reduction is carried out in the presence of organic chemicals. These will be discussed in more detail below.

**a. Direct Reduction in Aqueous Phase**

When metal ions are introduced in water, these metal ions are uniformly dispersed in the liquid phase. As a reducing agent is introduced to the solution containing metal ions, the metal ions are subjected to reduction resulting in an elemental state upon reduction if the reductant used is effective.\textsuperscript{6-17} It is important to note that reductants suitable for some metals are not necessarily suitable for others. Significant difference in the emf (electro-motive force) value between the metal ion and the reducing agent must exist in order to achieve an effective reduction. Difference in the emf is a necessary, but not sufficient, condition for satisfactory reduction.

As metal particles are generated, they should be individual metal atoms first. However, the metal atoms in the aqueous phase are unstable by nature, and these metal atoms try to get together and grow. In addition, some metals serve as nuclei for others to grow on. The rate of growth of these metal powders will depend upon various variables, including the concentration of metal ions, the type of reductant, temperature, and very frequently the pH of the system.

In general, the lower the concentration of metal ion and reducing agent the smaller the size of metal powders which results.\textsuperscript{6} The size of the produced metal powders is relatively small when the rate of reduction is slow. Consequently, less powerful reductants are favorable for generating smaller sizes of metal powders.

When metal powders produced by reduction are highly charged, the growth of the particles is adversely affected. Unfortunately, most metals, except gold and some of other precious metals, are subject to dissolution at low pH: therefore this method of producing small-sized particles has its limit. On the other hand, if the pH of the solution is raised too much, most metals are subjected to hydration or oxidation to become hydroxides or oxides, which may not be desirable in many applications.\textsuperscript{6}

Another way of controlling the size is to add dispersants. Dispersants frequently used may include starch, silicates, and various polymers.\textsuperscript{5,18} This approach is widely used in the synthesis of nano-metal powders but it should be noted that these dispersants are impurities added to the system, which could cause problems in later applications.

**b. Choice of Reductants**

Table 1 lists selected various metals with their standard emf value, melting point, and conductivity, which are compared with those of the hydrogen ion.
as a reference. When these metal ions are dispersed in water and an appropriate reducing agent is introduced, these metals will be reduced to elemental metals.

As noted from this table, noble metals such as gold, silver, platinum and copper are easily reduced by most common reductants but many other metals would require stronger reductants to be reduced. In addition to the reducing power, i.e. emf, the melting point of these metals is an important factor in certain applications. For example, in most electronic industry applications, good conductivity is essential to the function of the resulting product.

Most of the ink produced to date requires a post-heat treatment. The ink usually contains liquid, such as water, containing organic chemicals, which have to be removed to secure acceptable circuits with a desired value of conductivity. As a result, a post-heat treatment is needed to remove water and organic matters. Since most substrates are made of organic materials, the temperature applied in this heat process should not exceed the temperature beyond which the substrate can be damaged. In addition, it is necessary to ensure good mechanical adherence between the ink materials and the substrate. It is also important to maintain good fluidity of the ink with an acceptable level of viscosity.

Table 2 lists commonly used reductants with their emf values. As noted, most of these reductants will easily reduce gold, silver and copper, but in order to reduce most other metals, only a few of these reductants will be effective. It should be noted, however, that some of the reductants that produce gases as products are usually effective reductants: the partial pressure of these gases would be very low, as their solubility in water is low. As a result, the standard emf values can be misleading in representing their true power of reduction. In other words, the emf values of these reductants can be very small, as long as these gases are immediately removed from the system.

As will be discussed later, the stronger the power of reductants in terms of the emf becomes, the faster the reduction reaction will generally take place. However, a fast reduction does not necessarily represent desirable conditions for the formation of the desired size of nano-particles. It is frequently found that the slower the reduction is the smaller the final size of the nano-particles becomes. Another consideration should be taken into account in selecting the reductant is the kind of products it produces. In general, inorganic products such as sulfur or other solids are undesirable, since they become undesirable impurities which will stay with the nano-particles.

The majority of the studies on the manufacturing of nano-metal particles have been conducted on gold and silver. Since gold and silver are very noble as noted in Table 1, the reduction of these metals in solution can be easily carried out using even mild reductants. In view of these metals being noble and also environmental concerns, organic based reductants have been widely used in this practice. These include trisodium citrate (C6H5O7Na3), formaldehyde (CH2O), polyethylene glycol (C2nH4nO2n), glucose (C6H12O6), ascorbic acid (C6H8O6), tannic acid (C6H2O15), polyvinylpyrrolidone (PVP: C6H10NO), polyvinylalcohol (PVA: C6H10O5), oleyl amine (C17H35N), and many more. In addition, the literature mentions a host of other organic reductants. These include alkyl acid phosphate (CnH2nPO4 – amyl), dendrimers, 3-thiopheneacetic acid (C4H9O3S), superhydrides, glucose (C6H12O6), aspartic acid (C4H7NO4 – HO2CCH(OMe)CO2H), sodium formaldehyde sulfoxylate (CH2NaOSO2), arginine (C6H12N4O3), 1,2-hexanediol (C6H12O3), tetrakis (hydroxymethyl) phosphonium chloride ((HOCH2)4PCl), polyacrylamide (CH2CHCONH2), quercetin (C21H12O10), Geranium leaf, aldehyde (RCOCH), phosphorous, dimethylformamide (C3H6N), triocetyl phosphine, dextrin, dimethyamine borane, and toluene.

It should be noted however, that the most widely used reductants in manufacturing nano-metal powders is sodium borohydride, followed by hydrazine, and occasionally hydroxylamine hydrochloride, which are frequently used in hydrometallurgical applications. These reductants are very powerful and can be applied not only to gold, silver and copper, but also to other metals including iron, cobalt, and nickel. When these more powerful reductants are used, the reduction of ions is almost immediate, unlike with some organic reductants which are inherently weak reductants and often require high temperatures. The
c. Oxidation of Metal Powders

As noted earlier, most investigations into manufacturing nano-metal powders have reported on gold and silver. However, there is a natural incentive to produce nano-metal powders using copper. This is because copper exhibits the second highest conductivity among metals, and is very abundant. In terms of economical consideration, the price of copper is about 1/85 and 1/6,000 respectively of silver and gold based on the price in May of 2009.

As can be seen in Table 3, gold and silver are difficult to oxidize but copper is easily oxidized with a small amount of oxygen present. This indicates that most metals except gold and silver are easily oxidized in aqueous media. This is a problem in manufacturing nano-metal particles other than gold and silver. Prevention of oxidation of other metals including copper is almost impossible even in the inert gas atmosphere. The prevention of oxidation of these oxides can be accomplished with the help of CO or H₂ gas, as shown in Table 4. This means that oxidation of copper nano-particles can be eliminated if the synthesis of the copper nano-particles is conducted in the presence of CO or H₂. However, handling these gases is rather cumbersome, and practitioners prefer to avoid using such gases when possible.

### Table 3

<table>
<thead>
<tr>
<th>Metal Oxides</th>
<th>ΔG°,25°C cal/mol</th>
<th>P₀₂, atm</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuO</td>
<td>-30,400</td>
<td>2.60 × 10⁻⁵</td>
</tr>
<tr>
<td>Au₂O₂</td>
<td>48,000</td>
<td>1.58 × 10⁻⁵</td>
</tr>
<tr>
<td>Ag₂O</td>
<td>2,600</td>
<td>6.51 × 10⁻⁷</td>
</tr>
</tbody>
</table>

### Table 4

<table>
<thead>
<tr>
<th>Metal Oxides</th>
<th>CO/CO₂</th>
<th>H₂/H₂O</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuO</td>
<td>1.35 × 10⁻⁴</td>
<td>2.43 × 10⁻⁷</td>
</tr>
<tr>
<td>Au₂O₂</td>
<td>2.22 × 10⁻⁶</td>
<td>2.95 × 10⁻⁴</td>
</tr>
<tr>
<td>Ag₂O</td>
<td>6.08 × 10⁻⁸</td>
<td>1.09 × 10⁻⁴</td>
</tr>
</tbody>
</table>

d. Concentration Effect

The concentration and size of these nano-metal powders play an important role in creating an acceptable conductive ink. When metal ions suspended in water are subjected to reduction by a reducing agent, metal ions will be reduced to a metallic state, first as an atom, and then will grow by combining with other reduced metal atoms. The growth of these metals in water is a very fast process exhibiting the growth by a few micron sizes in a couple of minutes, if not seconds.

It is obvious that the more diluted these ions are in the system, the easier it is to maintain particles in the state of dispersion, and hence prevent agglomeration. However, this creates a dilemma, since an effective ink should maintain a certain concentration of these ions in the first place. It should be noted that there is a threshold concentration of these ions in the solution in the production of the conductive ink. The main objective of the ink is to render the final product sufficiently conductive, and therefore, the concentration of metal powders in the ink should be high enough to have sufficient amounts of metal powder accumulating on the substrate to ensure good conductivity through the patterns.

In order to illustrate the importance of the metal concentration to the formation of the ink, let us assume that we desire to make a one micron size pattern consisting of 1 μm width × 1 μm depth, using 100 nm size silver particles dispersed in the ink. When the ink is deposited on the substrate and water is subsequently evaporated via the post heat-treatment, only metal powders will remain on the substrate. Let us also assume that the metal powders at the 1 μm depth are all deposited on the substrate to form a monolayer. At this stage, it is vital to have sufficient amounts of metal powder in the ink to have a continuous metal powders aligned in the drawn pattern, so that electrons can flow through these metal powders. In order to achieve this objective, a monolayer formation, at the minimum, of these particles on the substrate is required. Table 5 shows the percent of the metal powders concentrated on the substrate for a given concentration of metal in the ink.

As noted in the table, when the concentration of solids is 0.1%, which is equivalent to 1000 ppm of metal powders in the solution, the solution will yield only 0.14% coverage, which is far from the monolayer required. Even at the concentration of 40%, which is equivalent to 620,000 ppm of solids, the solution will yield less than 100% monolayer coverage. It should be noted that even if the coverage is 100%, there is
no guarantee that the overall conductivity will be satisfactory, due to the unavoidable contact resistance between particles. It is noted that a 75% concentration of solids in the solution is by no means normally recognized as a fluid ink, but is rather a paste. This is one of the most difficult challenges to overcome in making a successful conductive ink.

Even if the resulting ink product in the substrate is multi-layered, there exists the problem of packing, which leads to voids resulting in poor conductivity. Since the melting point of silver is 960˚C, the contact resistance is very difficult to resolve, due to the fact that most substrates are made of organic compounds and will be damaged over 250˚C, a recommended maximum temperature allowed to sustain the integrity of the organic substrate used in the electronic industry. One way to overcome this problem is to mix silver with a metal or alloy whose melting point is rather low. As seen in Table 1, the melting point of some metals, such as tin, indium, and gallium, is less than 250˚C. Therefore, mixing silver with these metals yields better electrical contact after heat treatment at this temperature. Even if the melting point of a metal is more than this temperature, when this metal is mixed with other metals, the resulting eutectic temperature can be less than 250˚C (see Fig. 1). As seen in this figure, when indium and tin are mixed at about 50%, the melting point drops to about 120˚C. Such an attempt has been carried out in the authors’ laboratory and the results are encouraging.

f. Reduction with Organic Phase

Many investigators have synthesized gold and silver nano-particles using the organic phase in the presence of the aqueous phase with various surfactants. The so called “inverse micelle method” is frequently used in the synthesis of nano-metal particles, and utilizes such a two-phase system. As depicted in Fig 2, a group of metal ions dissolved in a water droplet are surrounded by organic surfactants which have a special affinity with the metal ions. These metal ions can be negative, as in the case of AuCl\(^{-}\) or positive as in the case of Ag\(^{+}\) inside the droplets.
These water droplets can be a result of emulsification of water in oil phase. Metal ions can form a group in the aqueous phase with the aid of surfactants which have affinity with these ions. For negative ions, cationic surfactant such as alkyl amines, and for cationic ions, anionic surfactants such as carboxyl and sulfonate are used to surround the metal ions. These metal ions are then subjected to reduction with the aid of a reductant such as sodium borohydride, citric acid, or alkane tetrakis phosphonium chloride.

The tail end of the surfactants is hydrophobic, and therefore they are attracted to the oil phase. These droplets can then be easily transferred to the oil phase as shown in Fig. 3. The oil phase is then separated from the water phase and subject to drying. After drying and evaporating, the residual organic phase will result in nano-sized metal powders which can be stored in an air-tight container for future use. The size of the reduced nano-metal powders is determined by the size of water droplets in oil as in the continuous phase.

The size of water droplets in the oil phase is determined by an effective emulsification of the oil/water mixture. The parameter known as “W,” which is the molar ratio of water to surfactant for the emulsification system is an important factor governing the size of these droplets.

In general, the less water present in comparison to the surfactant, the smaller the water droplets are and hence the smaller the produced nano-metal powders becomes. Surfactants used in the emulsification can be cationic, like cetyltrimethylammonium bromide, anionic, like bis(2-ethylhexyl)sulfosuccinate, and nonionic, like Triton X-100. Nano-metals produced in the water droplets can grow by agglomeration within the confinement of the size of the water droplets but the maximum size is limited by the size of the droplets.

Such prepared nano-particles in the oil phase can be stored for the future use. Even if the nano-particles in the oil phase become somewhat flocculated, they can be easily re-dispersed at a later time by methods such as ultra-sonic wave if necessary. The organic phase can be easily evaporated to isolate the nano-particles, as mentioned earlier. The surfactants adhered to metal nano-particles help metal nano-particles re-disperse. These surfactants can be removed by dissolution with a proper solvent, by evaporation or even by burning.

The reduction of metals trapped in the water droplets can be done while they are in the aqueous phase or after transferring into the oil phase. If these metal powders are reduced while in the water phase, after reduction, they can be transferred to the oil phase. The rest of the process will be identical as described above for the direct reduction process. In order to reinforce the stability of the water droplets, thiol group organic chemicals are often added, since thio group organic chemicals have affinity of these metal ions.

Nano gold and silver powders can be synthesized in the aqueous phase but in the presence of organic stabilizers such as poly-vinyl pyrrolidone (PVP) which also happens to be a reducing agent. The molecular weight of this organic chemical ranges between 10,000 and 60,000, and it serves as a dispersing agent to separate metal ions from each other. A solution containing metal ions is added to the solution containing this organic chemical and then the mixture is usually heated to about 60°C. Silver is then reduced to give a pink, purple, red or green color, depending upon the size and shape.
Other Applications of Nano-Metal Particles

Applications of nano-metal powders are found in materials, devices, and process technology. Nanoparticles are used in catalysts, nano-membrane, nano-carbon tubes, high strength materials, composite materials, and medical materials. Applications in nano-devices are found in short-wave electronic devices, molecular devices, bio-electronic devices, and optical devices. In addition, the nano-technology has applications in process technology including nanopatterning, nano-circuit, organic and inorganic nanomaterials processing, nano-electrical system, nano-computers, nano-measuring processes, and nano-medical processes.

Let us take a close look at a number of practical examples. Nano-sized gold and silver offer various colors depending upon size and shape. This property is used in paint applications. There are evidences that nano-particles can be used in deodorant and sunscreens, but much more investigation on the side and after effects of such applications is required for safe applications in these areas.

One of the most remarkable and promising applications of nano-metal powders would be in catalysis. It has been found that oxidation of carbon monoxide is possible even at room temperature on gold nano-powders. Cartoons showing the reactions between carbon monoxide and oxygen, and also cyanide and ferric ions, on gold nano-particles are shown in Figs. 4 and 5. In this case, since metal powders are nano-sized, huge surface area per given volume is available. In the case of cyanide with ferric ions, since gold has a special affinity with cyanide, gold nanoparticles will catch cyanide, helping ferric ion to react with cyanide. Therefore, the chemical reaction will be facilitated simply because of the availability of a large surface area, and, at the same time, the affinity of gold with cyanide, enabling it to hold cyanide for the reaction with ferric ions.

Summary and Comments

It is apparent that the fundamentals of hydrometallurgy, known to many who have been working on applied interfacial phenomena such as, mineral processing researchers, or chemical engineers, are easily applied to the manufacture of nano-metal powders. Manufacture of nano-metal powders utilizes the principles and technologies which have been already employed in these or many other related areas for a century. For example, mineral process engineers who are familiar with the metal reduction technology and interfacial phenomena used in froth flotation can immediately contribute their knowledge to the advancement of this exciting and emerging technology.

Nano-technology offers exciting opportunities, not only making nano-sized metal powders which can be used in the electronic industry, but also in medical, chemical, pharmaceutical, agricultural, civil, mechanical, electronics, materials, and environmental industries, and many other areas.

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References


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