

STUDY ON INKJET PRINTING OF NANOSTRUCTURES OF SILVER ON VARIOUS SUBSTRATES

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INTRODUCTION:

Inkjet printing is a computer-based printing that creates an image by ejecting fixed amounts of solution or “ink” filled in the cartridge. Inkjet printing is a versatile process used for deposition of liquid-phase materials. Examples of materials which have been printed this way includes polymers/nanocomposites, nanoparticles, carbon nanotubes (CNT), Graphene and MoS₂ nanosheets. Conductive inks can be printed in order to produce printed electronics, which is now a field of tremendous progress. Printed Electronics are being used for a variety of applications such as Photovoltaic cells, Light Emitting Diodes (LEDs), Transmitters, Supercapacitors, Photo sensors, Strain Sensors, Pesticide detection techniques, Gas Sensors, E-Textiles/Smart Clothing, etc. In comparison to traditional methods of manufacturing electronic devices, printing technique has a clear and overwhelming vantage of the production costs.

Properties such as oxidation resistance, low resistivity and water dispersibility has allured research enthusiasts greatly towards silver-based conductive inks over other conductive inks. The ink used in conventional method of conductive printing of silver nanostructures consisted of Ag nanowires (NW) or nanoparticles (NP) dispersed in solvents. Efficacy of such a printing method relies on creating solutions having appropriate flow properties to ensure adequate jettability of the ink through the nozzle heads of the cartridge. In case of printing AgNWs, this could be extremely challenging as they have a high aspect ratio and this could lead to blocking of nozzles. In addition, the high density of silver leads to relatively lesser amount of silver nanowires deposited for a given volume of ink printed on the substrate.

On the contrary, the salt printing technique developed by our group involves a far simpler process as this involves printing of salt solutions which is

exposed and developed on the substrate to obtain various morphologies of silver nanostructures accordingly. The advantages of this technique over other methods of Inkjet printing of AgNWs/AgNPs are that this technique can be carried out in room temperature and has a lower cost compared to other printing methods. Many printing techniques use the costly Dimatix Printer whereas we use Office Deskjet Printer. Hence, the developed technique seems to be a highly optimized one among all the methods available in literature for fabrication of silver nanostructures on different types of substrates.

CHEMICALS USED FOR THE EXPERIMENTS:

- Silver Nitrate(AgNO_3) Granules
- Potassium Halides, KX (X= Cl,Br,I)
- DI Water
- IPA solution
- Ethanol
- ID-78 (KBr and KCl variants)
- Acetone
- PDDA (Poly DADMAC)
- PSS (Poly Sodium 4-Styrene Sulphate)
- PVA (Poly Vinyl Alcohol)
- Labolene

STUDY OF VARIOUS PRINTER SETTINGS FOR IDENTIFYING THE MOST APPROPRIATE CHOICE

I. The below trials were printed with the following properties on MS Word –

- Quality -Normal Paper
- Colour
- Print in max DPI-NO
- Print in Grayscale-NO(except trial 9)

Observation of cartridge motion:

- Goes from Left End(LE) to Right End(RE) of cartridge rod
- Halts for a while
- Comes to middle section of the paper where 5 cm square has to be printed
- Prints and finally settles back to RE

TABLE 1: Speed calculated for various paper settings

SL NO.	Type of Paper	Speed Calculated (cm/s)
1	Plain Paper, Normal Quality	17.1
2	HP Photo Paper	19.6
3	HP Matte Brochure or Professional Paper	20.4
4	HP Matte Presentation Paper	21.2
5	HP Glossy Brochure or Professional Paper	18.0

6	Other Photo Inkjet Papers	19.8
7	Other Matte Inkjet Papers	18.7
8	Other Glossy Inkjet Papers	17.5
9	Repeat- HP Glossy Brochure or Professional Paper	18.0
10	HP Photo Paper(Grayscale)	17.1

II. The below trials were printed with the following properties on MS Powerpoint –

- Quality-Normal Paper
- Colour
- Print in max DPI-YES
- Print in Grayscale-High Quality Grayscale

TABLE 2: Notable changes in cartridge motion for change in paper type

SL NO.	Type of Paper	Cartridge Motion	Speed Calculated (cm/s)
1	Other Glossy Inkjet Papers	Cartridge moves to RE of rod every 15 th time it oscillates from LE to RE of square	14.9
2	HP Glossy Brochure or Professional Paper	Cartridge moves to RE of rod every 17 th time it oscillates from LE to RE of square	11.1

3	HP Matte Brochure or Professional Paper	Cartridge moves to RE of rod every 15 th time it oscillates from LE to RE of square	8.61
4	HP Photo Paper	Cartridge moves to RE of rod every 2 th time it oscillates from LE to RE of square	13.3

OBSERVATION & CONCLUSION:

Print setting finalised was '*HP Matte Brochure or Professional Paper*' with '*Max DPI*' & '*High Quality Grayscale*' as it has the least speed and was observed to deposit most amount of ink.

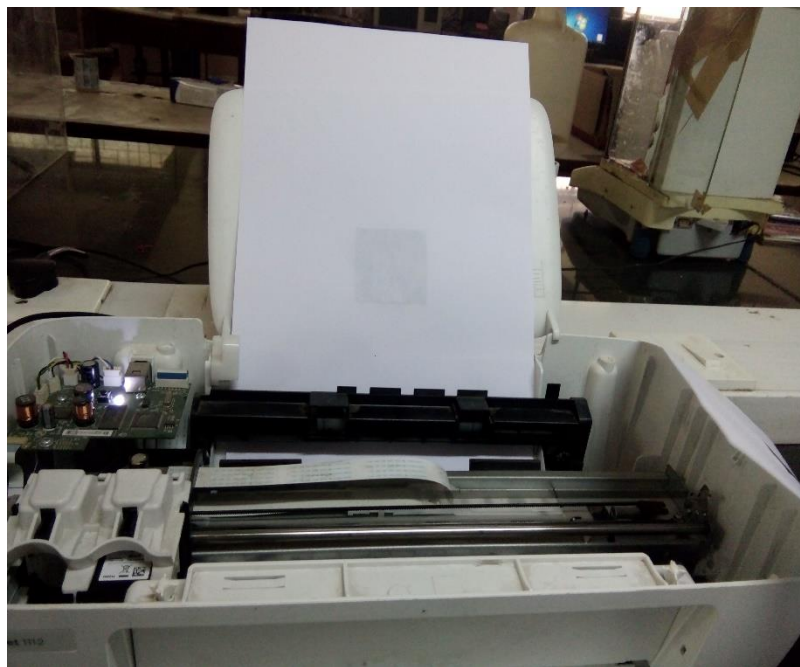


Fig 1: Taking prints using HP DeskJet 1112 printer and HP Black Ink Cartridge 803

PRINTING DI WATER TO DETERMINE (AMOUNT BEING PRINTED)

$\mu\text{L}/\text{cm}^2$ OF PRINTING INK USED AND SILVER LOADING

Data used for Calculations in *Table 3-*

- Area printed=5 cm square=25 cm²
- Density of DI water at room temperature(32°C), $\rho=0.995$ g/mL
- 0.850 g AgNO₃ was dissolved in 5 mL DI Water to make a solution of 1 M concentration.
- Mass fraction of Ag in 1 g of AgNO₃=0.635

TABLE 3: Calculation table to determine silver loading

(Sample calculation is provided in appendix A)

SL NO .	Wt. of Cartridge +DI water	(Wt. of Cartridge +DI water) after 5 successive prints	DI water used for 5 prints	DI water used for a single print	Volume of DI water used for a single print	DI water used for a single print per unit area	Amt. of AgNO ₃	Amt. of Ag
	g	g	g	g	μL	$\mu\text{L}/\text{cm}^2$	mg/cm ²	mg/cm ²
1	19.602	19.376	0.226	0.045	45	1.8	0.306	0.194
2	19.475	19.227	0.248	0.050	5.0×10^1	2.0	0.340	0.216
3	19.263	19.040	0.223	0.045	45	1.8	0.306	0.194
4	19.200	18.977	0.223	0.045	45	1.8	0.306	0.194
5	19.095	18.870	0.225	0.045	45	1.8	0.306	0.194

Amount of DI water used for a single print per unit area= $(1.84 \pm 0.0152) \mu\text{L}/\text{cm}^2$

Amt. of Ag = $(0.198 \pm 0.009) \text{ mg}/\text{cm}^2$

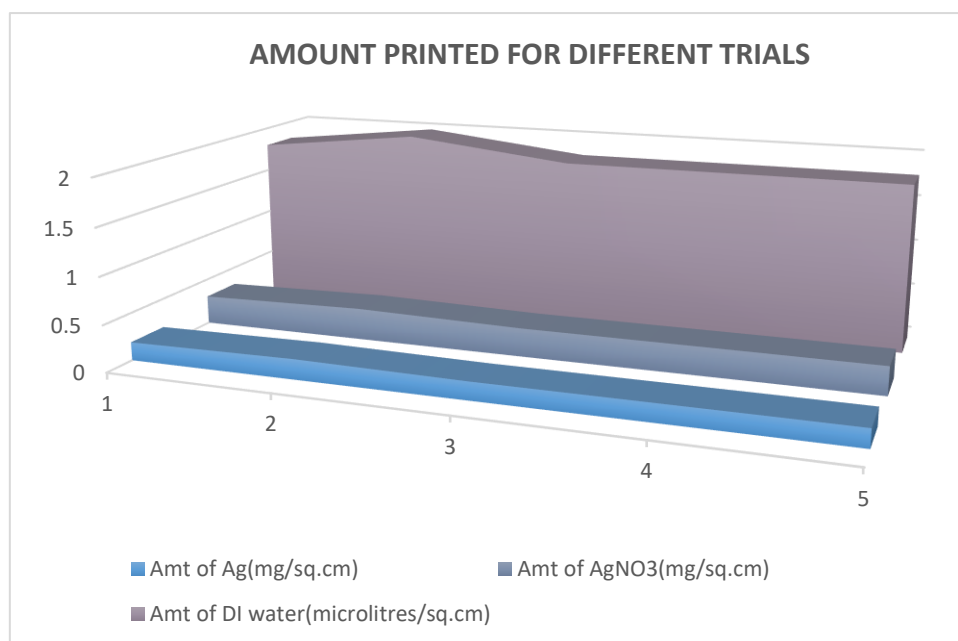


Fig 2: Amount of Ag, AgNO₃ and DI Water printed for various trials

CONDUCTIVITY OF AgNO_3 ON DIFFERENT MATERIALS

AIM:

To observe conductivity of square sheet of 5 cm×5 cm dimensions on the following materials-

- PET(Poly ethylene terephthalate)
- Natural Rubber Sheet
- Normal Quality Paper
- HP Photopaper

PROCEDURE:

I.PRINTING

1. 5 mL of 1 M AgNO_3 solution was prepared for which 0.850 g of AgNO_3 was dissolved in 5 mL DI water & sonicated for 2 min. The solution was covered with Aluminium foil as AgNO_3 could decompose on exposure to light.
2. 7×7 cm square of PET, Photopaper and Natural Rubber sheet was cut.
3. The rubber sheet & PET pieces were roughened using sandpaper in order to improve surface wettability (Fig 3).



Fig 3: Wettability comparison of normal and roughened substrates respectively: Left-PET sheet; Right- Natural Rubber sheet

4. Check for appropriate roughening by dropping and spreading some DI water on your material.
 5. 5×5 cm squares were printed on blank paper using black ink to understand position for fixing the various materials.
 6. Cartridges were drained and cleared using tissue paper and DI water.
 7. The cartridge lid was closed and printing was carried out on normal papers until complete removal of DI water was confirmed. You can also opt for printing at increased resolution so that more DI water gets used up.
 8. The bottom part of cartridge has to be cleaned with a tissue paper dipped in ethanol or IPA. Following that, wiping of the alcohol with a fresh tissue is also necessary.
 9. The materials were attached by sticking double-sided tape strips around the 5 cm black square. Scotch tapes were also used after sticking material using double-sided tape. Note that the scotch tape should remain outside of the 5 cm black square.
 10. Flow of AgNO_3 on tissue was ensured before every time of printing AgNO_3 . Then AgNO_3 was printed and kept for drying.
 11. Post drying, AgNO_3 was printed another time. The '*print-dry*' cycle was repeated until a total of 5 layers of AgNO_3 got coated. Note that the alignment of paper inside the printer matters.
 12. After printing cycles were over, the cartridge was cleaned, again using DI water and the cartridge was drained out completely using tissue paper.
- Before printing any solution, it is important to make sure that you have saturated the cartridge with the same solution.

II. PREPARATION OF DEVELOPER SOLUTION

1. 500 mL beaker was washed using Labolene, DI water and the remaining DI water was wiped off using acetone.

2. 400 mL of developer solution was prepared (ID-78) which consists of:

- 400 mL of DI Water
- 20 g of Sodium Sulfite-desiccated (Na_2SO_3)
- 4.8 g of Hydroquinone ($\text{C}_6\text{H}_6\text{O}_2$)
- 24.8 g of Sodium Carbonate (Na_2CO_3)
- 0.2 g of Phenidone ($\text{C}_9\text{H}_{10}\text{N}_2\text{O}$)
- 0.16 g of Potassium Bromide (KBr)

3. The solution was sonicated for 10-15 minutes to ensure homogeneity and solubility of all of its components.

III. PRINTING OF THE DEVELOPER ON THE MATERIALS

1. The cartridge was cleaned using DI water.

2. A small strip of AgNO_3 printed material was cut out.

3. The dimensions of the strip and the print area in the printer was roughly matched by printing the ruler-measured dimension of the strip using black ink on normal paper.

4. The strip was attached to the paper and the developer was printed over it 5 times on each material. It was made sure that proper drying occurred in between the successive prints (Fig 4).

NOTE: Developer needs to be sonicated. Also, cartridge bottom has to be cleaned using ethanol/IPA dipped tissue and a clean tissue.

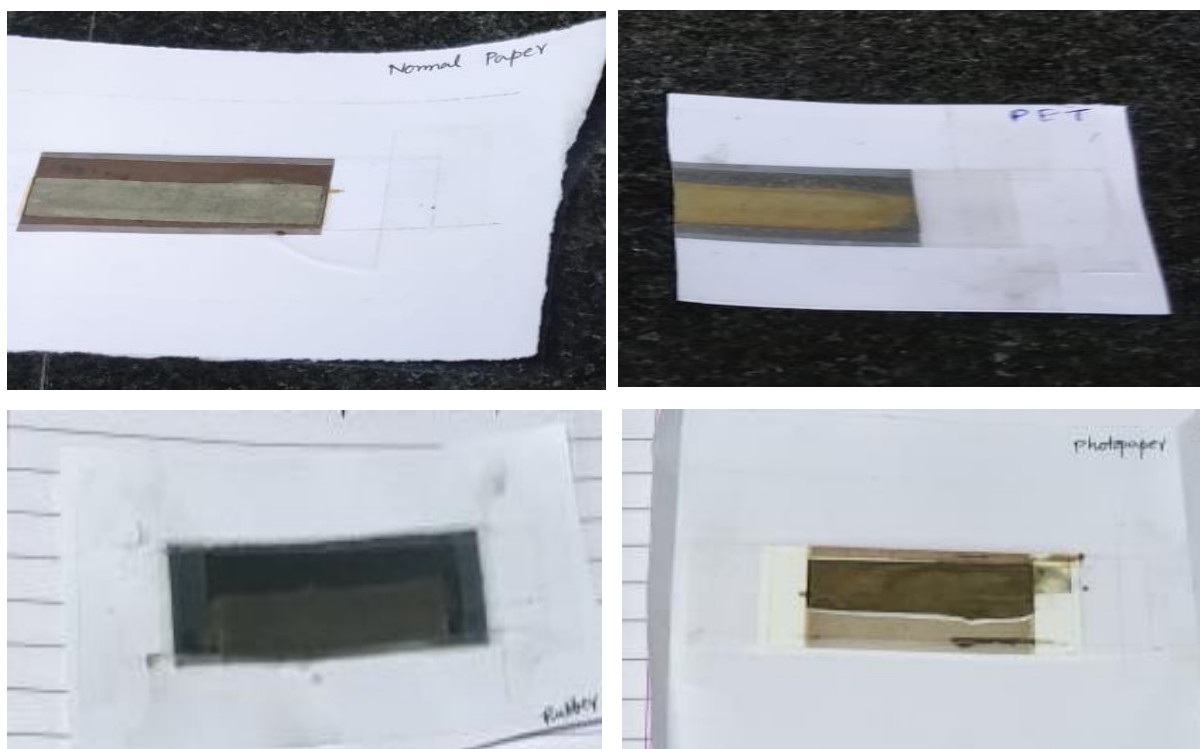


Fig 4: Different substrates printed with ID-78 (KBr) developer: Top Left- Normal Paper; Top Right-PET; Bottom Left-Natural Rubber; Bottom Right- Photopaper

RESULT:

On printing developer on the 4 materials, no substance is found to conduct electricity.

IV. DIRECT DIPPING OF DEVELOPER ON THE ABOVE MATERIALS

AIM: To understand whether the above made print-developed strips of AgNO_3 did not conduct due to inadequate amount of developer solution printed.

PROCEDURE:

1. The developer solution was sonicated for around 10 minutes.

2. The developed strips obtained from III above were dipped in developer solution for 15 minutes and then dipped into a tub of DI water to stop development (Fig 5).

3. After proper drying of the strips, the conductivity was checked.

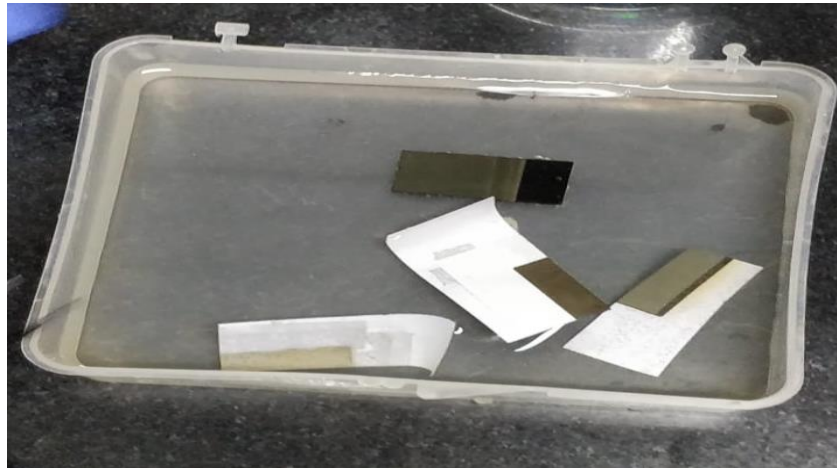


Fig 5: Picture showing development of substrates (Normal Paper, Natural Rubber, PET and HP Photo paper) by dipping into a tub of ID-78(KBr) developer

RESULT:

On further dipping the strips after printing with the developer solution, the materials conduct electricity.

CONCLUSION:

Since the materials conduct now, we can conclude that the amount of developer printed was not sufficient. Hence, we can drop off the method of printing developer and undertake the conventional development procedure of immersing the prints.

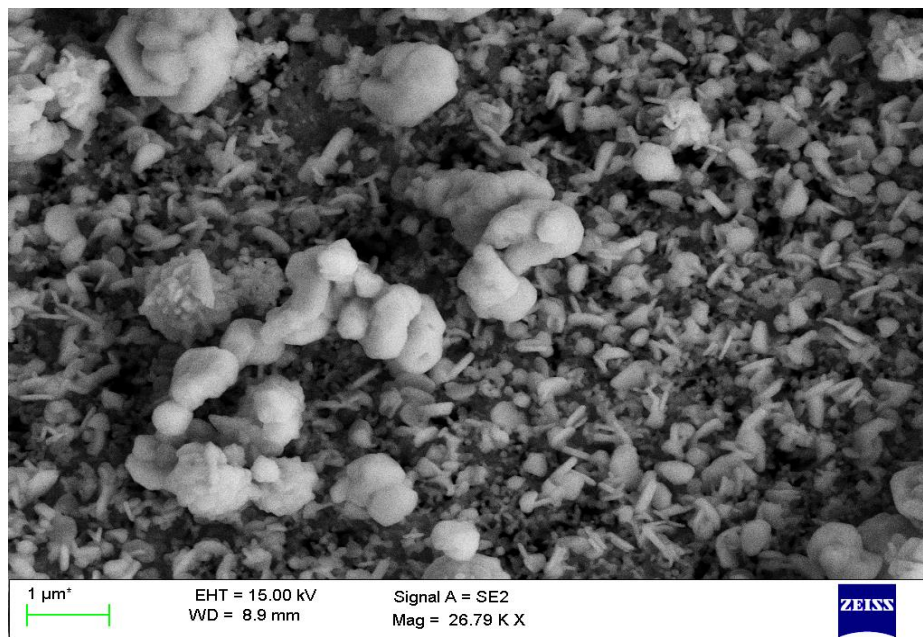
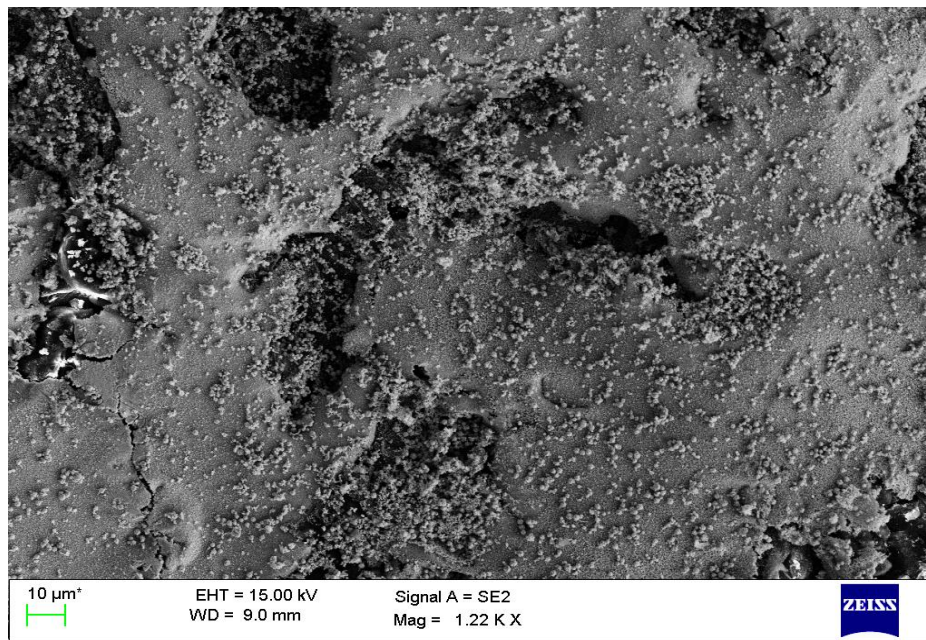


Fig 6: SEM images of PET sheet

APPLICATION OF AgNO₃-IPA SOLUTION ON PET AND POLYIMIDE **(WITHOUT PRINTING) FOLLOWED BY DIRECT DEVELOPMENT**

1. A. A 5 cm square of PET was cut.
B. Polyimide sheet was stuck on PET and a 5 cm square was cut out of it.
2. AgNO₃-IPA solution was spread evenly on both the materials. Around 40 μ L of solution was used for this purpose. It was noticed that the surface wettened without roughening.
3. The materials were let to dry. Meanwhile, the developer solution was sonicated for about 10 minutes.
4. After sonication and drying of printing materials, they were dipped in ID-78 developer for about 15 min
5. The developed materials were dipped in DI water for 5-6 minutes and kept for drying inside the laminar hood.



Fig 7: Polyimide Tape available in Markets (IC: [google.com/images](https://www.google.com/images))

RESULT & INFERENCE:

The developer hadn't evenly spread on the surface of AgNO₃-IPA coating unlike the case of a roughened surface. Hence this method can be dropped off and replaced with the roughening method.

THE BASICS OF SALT PRINTING:

The salt-based inkjet printing of silver nanostructures is based on the conventional black and white photography process. Photographic film and paper are made up of photosensitive grains of silver and halides. In our method, we print aqueous layers of silver salt and reducing agent (KX) alternatively on normal paper which are low-priced & commonly available to carry out the photographic process. This will form a percolating conductive network of silver nanostructures upon enough halide light exposure followed by development. It was understood from literature that morphologies can be controlled by the type and concentration of the halides used. Printing only silver bromide results in thick and sparse network of nanowires, whereas on printing silver chloride, we would be getting silver nanoparticles. However, introducing larger ion such as iodide into the halide motivates a nanowire formation. For example, by printing a solution of KBr:KI in the ratio 95:5 on silver salt results in a morphology consisting of a mixture of nanowires and nanoparticles(Fig 7).

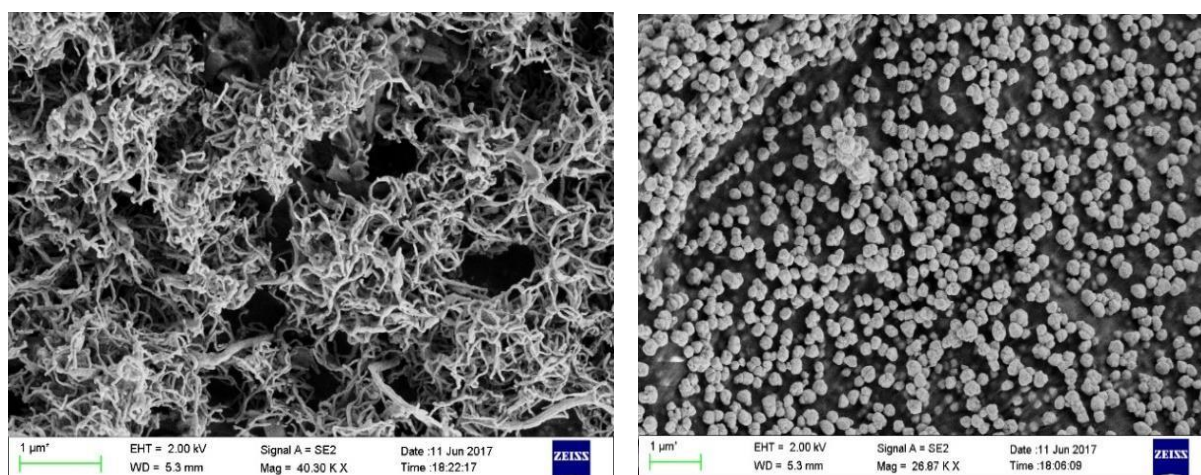


Fig 8: Effect of halide on morphologies: Left-AgNWs formed by using KX and AgNO_3 ; Right- AgNPs formed by using KCl and AgNO_3

After printing and adequate drying, the substrate is exposed to halide light where nucleation takes place. In simple words, when photons from light come in contact with the silver halides, electrons are ejected from the valence band of the halide and move to the conducting band. This electron then combines with a silver ion in motion and ends up forming atomic silver (Ag). The place where this occurs is called the 'latent image centre'. Note that 3 or 4 atoms are necessary for obtaining a stable latent image centre. Thus, a latent image is formed on our exposure step which is invisible (hence the name 'latent') to the naked eye (Fig 8). The formation of silver metal is logarithmically proportional to the intensity of light which leads to varying latent image centre densities. Also, latent image centre density is the reason for variation in morphology on use of different halides. Larger ions such as I^- improve the mobility of Ag^+ due to more lattice defects in the halide grains resulting in a relatively high latent image density.

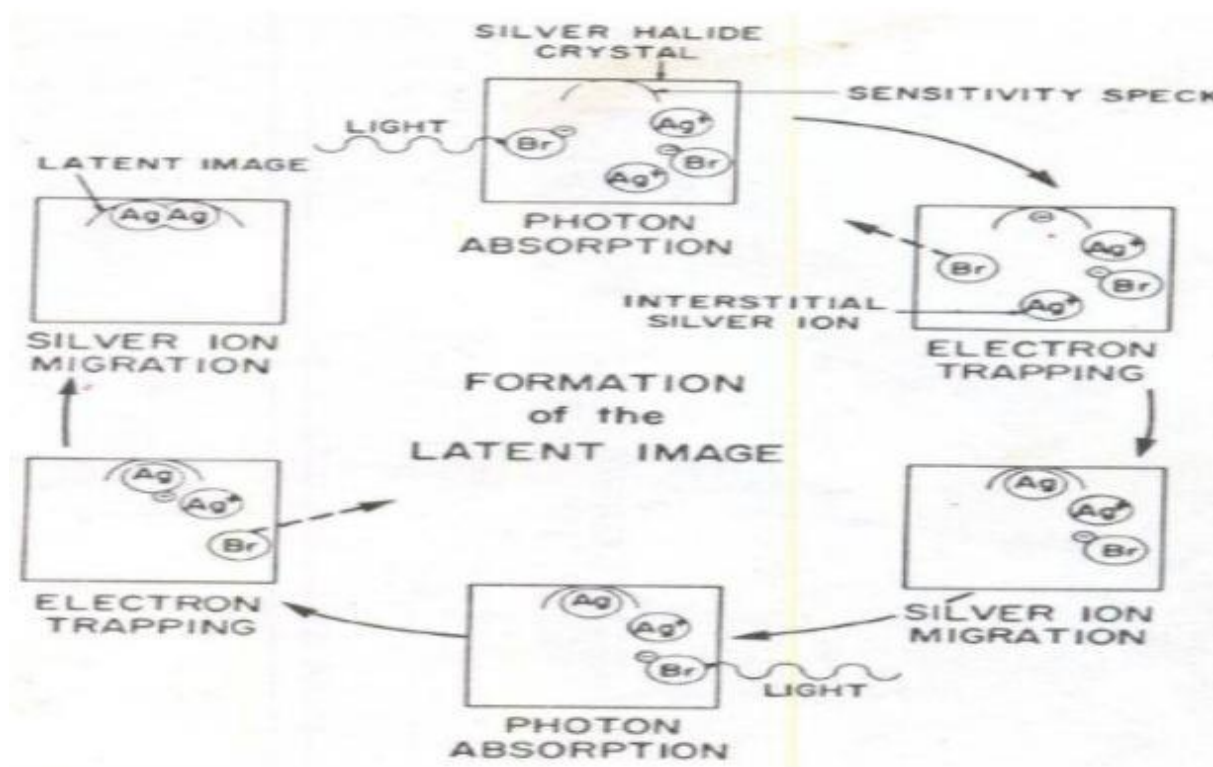


Fig 9: Formation of latent image (Image Source: www.google.com/images)

Developers are basically reducing agents which are used to make the latent images visible by acting on the exposed light-sensitive silver-halide crystals. Silver clusters formed during light exposure acts as deep electron traps and receive electrons when immersed in a developer. The electrons then attract Ag^+ . Finally, AgX nanoparticles reduce to AgNP which leads to a visible image. A large number of developers have been formulated such as ID-68R, ID-78, IF-2, etc. <http://bjornburton.org/ilford.html.gz> showcases a list of recipes for making developers along with applications to outweigh one over the other for our specified purpose. Fixer does the work of fixing the image and make it permanent and washing with DI water cleans the substrate from all the previous chemicals it was exposed to, thus terminating the reactions happening, if any.

PRINT-EXPOSE-DEVELOP OF BILAYERS OF AgNO₃-KCl

AIM:

1. To understand the process of '*Print-Expose-Develop*' method of generating silver nanostructures.
2. To observe difference in morphology by using different developers- ID-78 developers (one containing KBr and other containing KCl)

PROCEDURE:

1. 1 M AgNO₃ was prepared by dissolving about 0.850 g of AgNO₃ in 5 mL DI water. Similarly, about 0.745 g of KCl was dissolved in 5 mL DI water to obtain solution of 2 M concentration. Higher concentration of KCl is chosen in order to ensure that all Ag gets converted to its halide.
2. The prepared solutions were sonicated for 2 minutes and both the developers were sonicated for 10 minutes each.
3. 2 separate cartridges were opened up for printing of AgNO₃ and KCl respectively. Each cartridge was filled with the corresponding solution. It was also made sure that the cartridges got saturated with the solution it has to print by printing individually on plain paper.
4. A bilayer of AgNO₃-KCl was printed starting with KCl (referenced as K) and followed by AgNO₃ (referenced as A). After every print of the cartridges, their bottoms were cleaned using tissue dipped in IPA and further with a dry tissue too. A total of 6 prints were carried out one on top of other, in two different patterns-KAAKAKA (letters 'A I S' in both the sets) and KAKAKA (remaining letters). A single print consists of 2 sets of letters 'A I S W A R Y A' for observation under different development conditions.

Also, note that successive prints should be made only after proper drying of the previous layer.

5. After printing the 3 bilayers, the samples were exposed under halide lamp for 15 minutes.

6. One set of letters (A I S W A R Y A) were then dipped in ID-78(containing KBr) developer and other set of letters were dipped in ID-78(containing KCl) for 15 minutes. The developed prints were washed in a tub of DI water and kept for drying inside the laminar hood.



Fig 10: An example of set of letters fabricated on normal paper using 'print-expose-develop' technique

Hypothesis:

The set of letters (A I S W A R Y A) developed using ID-78(containing KBr) is assumed to have wire-like morphology and the set of letters developed using ID-78(containing KCl) is assumed to have particle-like morphology. The morphology can be verified by carrying out a SEM analysis.

RESULTS:

1. The letters developed using ID-78(KBr) has lower resistances than *corresponding* letters developed using ID-78(KCl) developer. This means that the former set conducts more which could be possibly due to the hypothesised wire-

like morphology in comparison to a lesser conducting, particle-like morphology for the latter set.

2. The resistance of KAAKAKA print is far lower than resistance of KAKAKA print.
(For ex: 4 Ω and 100 Ω respectively, i.e. about 25 times)

STUDY OF THE EFFECT OF EXPOSURE TIMES

AIM:

1. Analyse the effect of different exposure times and constant development time using the '*Print-Expose-Develop*' method.
2. Print equilateral triangles and check whether resistance across every edge is around the same.

PROCEDURE:

1. 1 M AgNO_3 was prepared by dissolving about 0.850 g of AgNO_3 in 5 mL DI water. Similarly, about 0.745 g of KCl was dissolved in 5 mL DI water to obtain solution of 2 M concentration. Higher concentration of KCl is chosen in order to ensure that all Ag gets converted to its halide.
2. The prepared solutions were sonicated for 2 minutes and ID-78(containing KBr) developer were sonicated for 10 minutes.
3. AgNO_3 and KCl cartridge were filled with the corresponding solutions. It was made sure that the cartridges got saturated with the solution it has to print by printing individually on plain paper.
4. A bilayer of AgNO_3 -KCl was printed starting with KCl (referenced as K) and followed by AgNO_3 (referenced as A). After every print of the cartridges, their bottoms were cleaned using tissue dipped in IPA and further with a dry tissue too. A total of 6 prints were carried out one on top of other in the pattern-KAKAKA. A single print consists of 11 equilateral triangles (edge length= 3 cm).

Also, note that successive prints should be made only after proper drying of the previous layer.

5. After printing the 3 bilayers, the 10 of 11 triangles were separately exposed under the halide lamp for varying times, i.e. 1 to 10 min, in increments of 1 minute. The last triangle was left unexposed.

6. As soon as exposure time of each triangle got over, it was developed using the ID-78 developer for 15 minutes and washed in DI water. All of the 10 samples were kept inside the laminar hood for adequate drying.

RESULTS:

TABLE 4: Resistances of substrates fabricated using 'Print-Expose-Develop' technique on normal paper with different exposure times

Exposure time (min)	Averaged resistance of 3 edges of the equilateral triangle (Ω)
No exposure (0 min)	Non-conducting
1	4.7
2	4.6
3	4.6
4	5.1
5	4.9
6	4.8
7	4.5
8	5.0
9	3.3
10	4.5

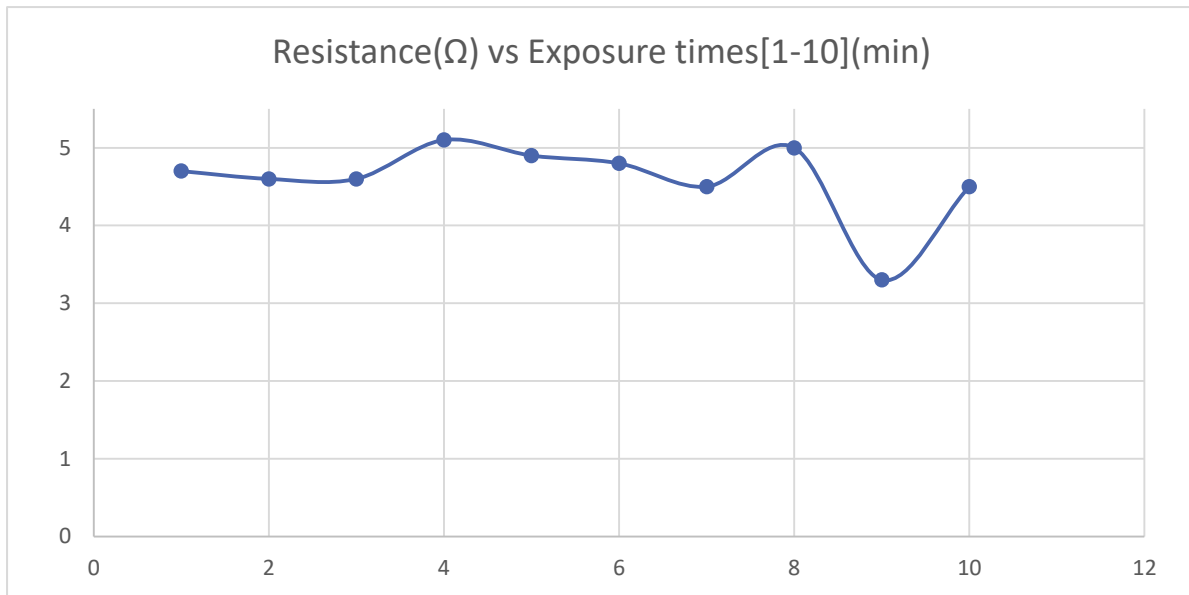


Fig 11: Curve showing variation of measured resistance with different exposure times (1-10 min)

CONCLUSION:

Exposure to halide light is necessary for obtaining conductive structure while printing $\text{AgNO}_3\text{-KCl}$. On the contrary, it is not necessary to expose the substrates for long durations as it doesn't seem to cause any major difference in the resistances obtained.

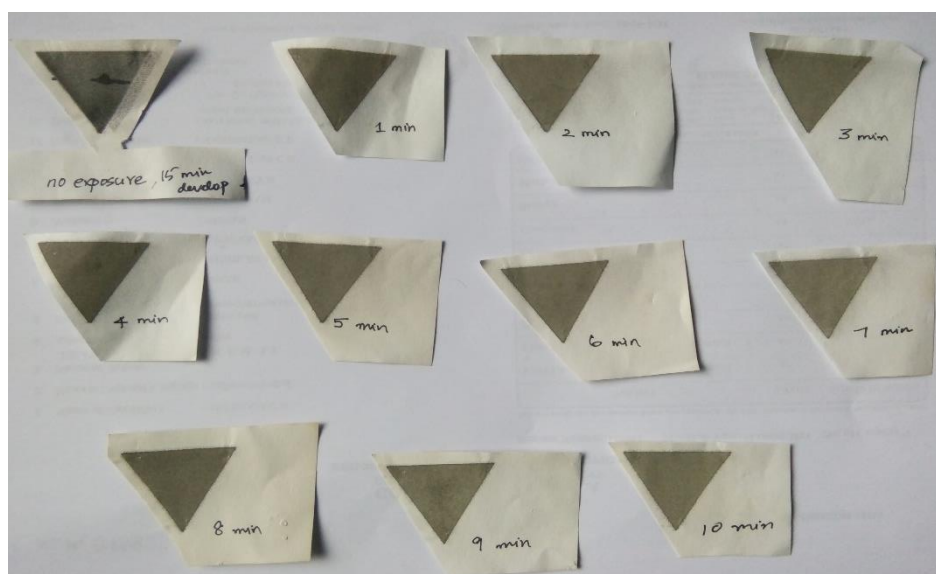


Fig 12: Equilateral triangles fabricated on normal paper using 'Print-Expose-Develop' technique with varying exposure times

STUDY OF THE EFFECT OF DEVELOPMENT TIMES

AIM:

Analyse the effect of constant exposure times and different development time in the conventional '*Print-Expose-Develop*' method using Natural Rubber

PROCEDURE:

1. 1 M AgNO_3 was prepared by dissolving about 0.850 g of AgNO_3 in 5 mL DI water. Similarly, about 0.745 g of KCl was dissolved in 5 mL DI water to obtain solution of 2 M concentration. Higher concentration of KCl is chosen in order to ensure that all Ag gets converted to its halide.
2. A Piece of Natural Rubber Sheet was roughened with sandpaper and good wetting was obtained for printing (Fig 12a).

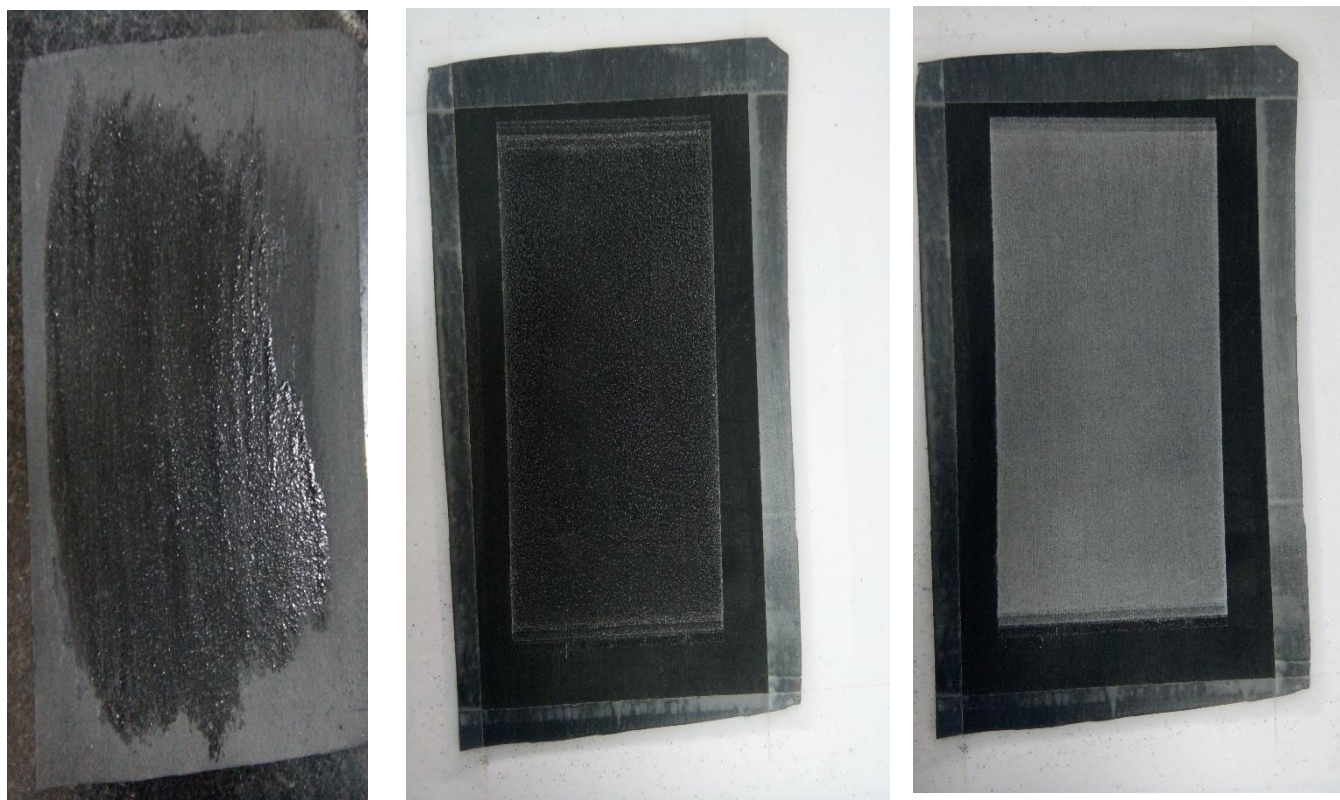


Fig 13: a (Leftmost image)-Hydrophilicity of roughened Natural Rubber sheet; b (Middle image) - After printing and drying of 5 layers of KCl; c (Rightmost image) - After printing and drying of 5 layers of AgNO_3

3. The prepared solutions were sonicated for 2 minutes and ID-78(containing KBr) developer was sonicated for 10 minutes.

4. AgNO₃ and KCl cartridge were filled with the corresponding solutions. It was made sure that the cartridges got saturated with the solution it has to print by printing individually on plain paper.

5. The rubber sheet was attached to paper using scotch tape. 5 consecutive layers of KCl (referenced as K) and AgNO₃ (referenced as A) were printed each. After every print of the cartridges, their bottoms were cleaned using tissue dipped in IPA and further with a dry tissue too.

Also, note that successive prints should be made only after proper drying of the previous layer (Fig 12b, c).

6. After printing the rubber sheet was cut into a number of pieces and all the pieces were exposed together under the halide lamp for 3-4 minutes.

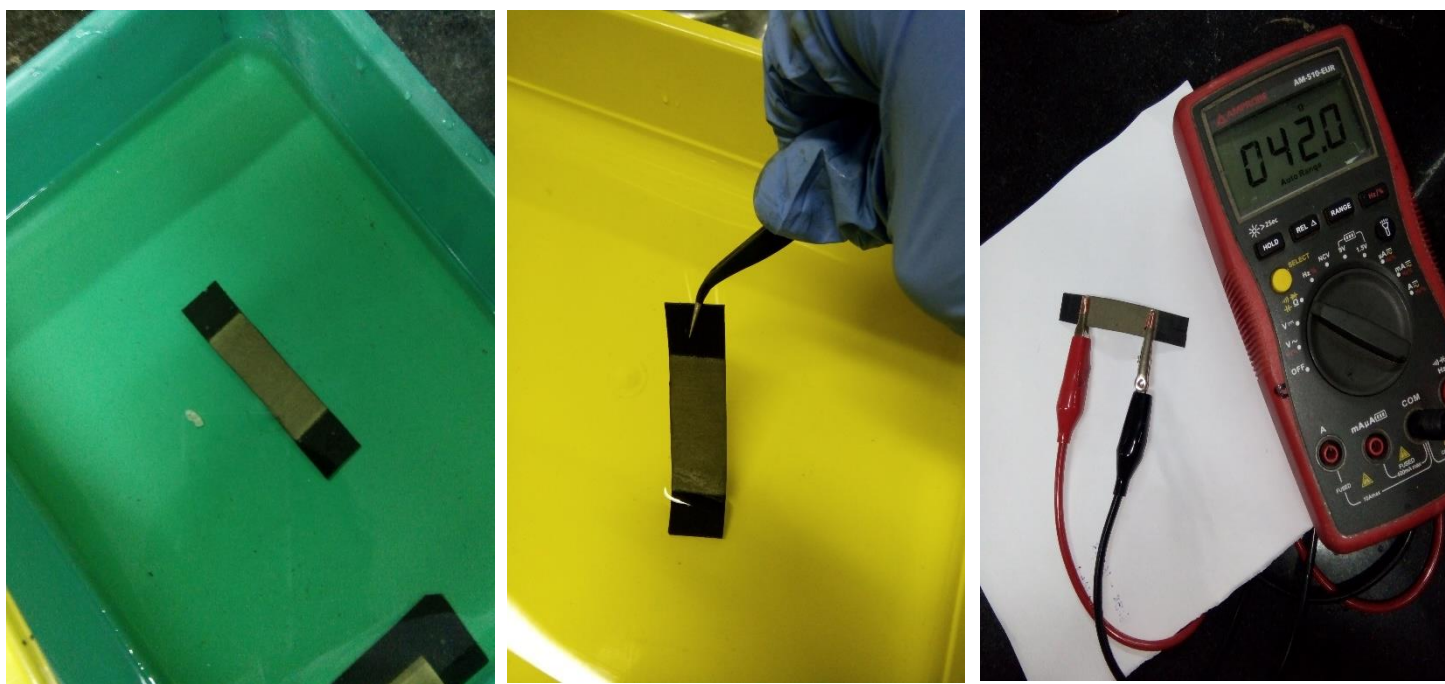


Fig 14: Leftmost- Developing in ID-78 (KBr); Middle- Washing with DI water; Rightmost- Checking resistance after drying

7. As soon as exposure time got over, each figure was developed using the ID-78(KBr) developer for varying times, i.e. from 0-5 minutes, each with an increment of one minute and washed in DI water later (Fig 13). Do not forget to use a tweezer. All of the samples were kept inside the laminar hood for adequate drying.

OBSERVATION:

Development Time (min)	Resistance (Ω)
No development (0 min)	Non-conducting
1	42
2	51.8
3	31.4
4	45.8
5	41.8

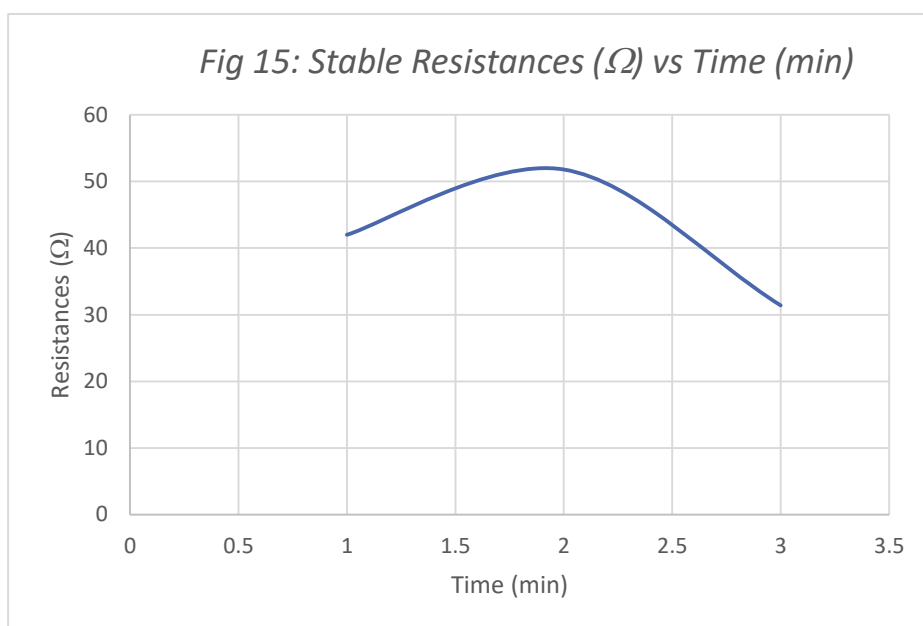


TABLE 5: Resistances of silver nanostructures fabricated on Natural Rubber using 'Print-Expose-Develop' technique with different development times

INFERENCE:

Prints obtained on Natural Rubber Sheet appear to be very uniform. As development times vary, there was no unique trend observed, though there was no conduction with no development. Hence, we cannot predict the development time required for obtaining some specific value of resistance.

STRAIN SENSORS

Flexible and stretchable electronic devices are used in health monitoring systems, robotics, human-machine interfaces, etc. Strain sensors can be broadly classified into Piezoresistive, Piezoelectric and Piezocapacitive. Piezoresistive strain sensors respond to mechanical strain due to change in resistance, one of which our group is trying to develop. The change in resistance occurs due to propagation of cracks when a tensile or compressive stress is applied as explained in the literature. Stretchability and sensitivity are two parameters which decide the quality of the sensor. Also, sensors need to have considerably fast response speeds. Strain sensors with low sensitivities can be used for human body detections such as for pulse rates, heart rate, small body movements and speech detection. Materials such as CNT, CuNWs, AgNWs, etc have been used for similar applications. However, developing strain sensors via the simplest method of 'Print-Expose-Develop' unique to our lab is novel. Our group had found modified forms of Dragonskin, known as 'Ecoflex' which has properties similar to skin and good stretchability and sensitivity. But printing AgNWs on Dragonskin/Ecoflex has become a challenge. The remaining part of this project involves our trials to print on the surface of Dragonskin.

PREPARATION OF DRAGONSKIN

PROCEDURE:

1. Part A and Part B of 'Dragon Skin' (a product of Smooth-On) was weighed in 1:1 ratio (around 6 g) to make a semi-liquid solution of *Dragonskin*.



Fig 16: Part A and Part B components available in the market for preparation of 'Dragonskin' (Image Courtesy: www.google.com/images)

2. The solutions were mixed. It was noticed that bubbles were being formed during mixing and pouring, which could lead to non-uniformity of the final product formed.
3. In order to remove the bubbles, the mixture was degassed inside a desiccator for about 20 minutes. For opening the desiccator after degassing, some air was let to get inside the desiccator vessel.
4. The degassed *Dragonskin* solution was slowly poured on a 220-grit sand paper. The purpose of pouring in on sandpaper is to make it more sensitive to strain.

5. For complete solidification of *Dragonskin* on sandpaper, we have to wait for about 16 hours (as per the product specification). Then, it was peeled off from the sandpaper.



Fig 17: Low wettability of the surface of Dragonskin on sprinkling DI water

OBSERVATION:

Dragonskin was prepared without trapped bubbles in it. But, the material doesn't have considerable wettability (Fig 13).

INFERENCE:

It is necessary to improve wettability of the surface in order to print on it for wearable sensor applications.

IMPROVING HYDROPHILICITY OF THE SURFACE

Wettability of a surface is determined by the force balance of the substance on the surface accounting adhesive and cohesive forces. For adherence of a material on another, wetting is important. If water can wet a surface, we call it 'Hydrophilic' and if not 'Hydrophobic'. A measure of contact angle of the liquid on the substrate can give us an idea of how much/how much not it wets the surface. An instrument known as 'Goniometer' is used for this purpose. As the contact angle gets lower, wettability increases. The least wetting will be for substance with a contact angle of 180° , when the liquid tends to achieve the shape of a sphere.

Various methods have been tried by our group in order to achieve improved hydrophilicity of Dragonskin, which is our substrate of interest. One of the most common ways of increasing adhesion is by roughening the surface. As we have seen earlier, roughening materials using sandpaper such as PET and Natural Rubber increases its hydrophilicity rendering it to be printable. Unfortunately, such a simple technique did not come to fruition on Dragonskin (Fig 14). This compels us to go in search of better and feasible methods such as Plasma Treatment. In this process, we let oxygen plasma oxidise the outer layer



Fig 18: Hydrophobicity of Dragonskin(cured on PET) after roughening with sandpaper

of the surface/polymer and increase the number of polar groups, directly improving the printability or coatability. Plasma treatment also makes the product clean. On the contrary, while plasma treatment improves wetting, its effect doesn't last

very long. So, we carry out other secondary procedures on a plasma treated substrate as described in the following sections.

I. PLASMA TREATMENT AND ELECTROLYTE COATING (PDDA-PSS) OF DRAGONSKIN

1. The Dragonskin has to be plasma cleaned (Fig 15) for 8 minutes.
2. Sonicate PDDA (Poly DADMAC), which is a positive electrolyte and PSS (Poly Sodium 4-Styrene Sulphate), a negative electrolyte each for 10 minutes.
3. The Dragonskin sheet was then immersed in a beaker of PDDA for 15 minutes. This will lead to a coating of PDDA on the Dragonskin.



Fig 19: Harrick Plasma Cleaner (Image Courtesy: www.google.com/images)

4. After dipping into PDDA, the Dragonskin was washed with DI water (immersing for 1-2 minutes) and then dipped into beaker containing PSS for 15 minutes. DI water washing is done before coming in contact with both electrolytes in order to prevent the combination of negative and positive electrolytes.

5. The 'PDDA-DI water-PSS-DI water' cycle was repeated 3 times, thus forming 3 bilayers. It could be observed that surface of Dragonskin has got completely wet.



Fig 20: The picture shows the improved surface wettability on coating Dragonskin with Electrolytic solution

OBSERVATION:

It was observed that the surface of Dragonskin has achieved increased wettability. Also, wiping the PDDA-PSS coated Dragonskin with tissues or any other wipe can lead to removal of the coating.

CONCLUSION:

Eventhough the objective of improving wettability has been achieved, it will not persist as the coating can be easily removed. Moreover, this method appears to be tedious for which we should have had a more fruitful outcome. Hence, this method is not the most feasible solution.

II. CHALK COATING

Chalk is a soft, white, porous, sedimentary carbonate rock, a form of limestone composed of the mineral calcite (CaCO_3) [Wikipedia]. Chalk powder is an easily available and cheap material. Its use as an abrasive encouraged us to try out coating solutions and pastes of chalk on Dragonskin (Fig 17).

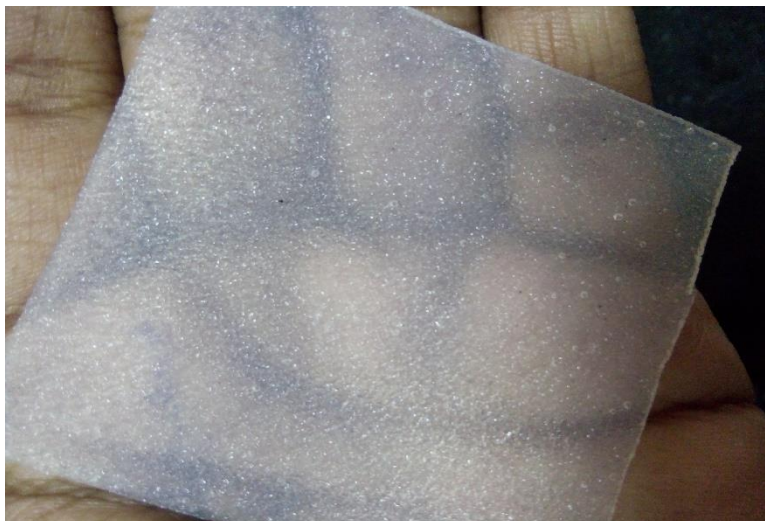


Fig 21: Dragonskin coated with 20 wt% chalk powder solution

It was observed that on coating pastes, we get very good wettability. But the chalk deposited can be scraped off from the surface without too much of effort, though it won't come out by casual wiping. Above all, this method leaves us with a disadvantage of non-uniform coating (which leads to uneven surface properties) when coated using a roller or immersion coating. Therefore, we need to further explore the literature available.

III. PVA COATING

PROCEDURE:

1. Dragonskin was prepared on PET sheet
2. 1 wt% PVA solution was prepared by dissolving powder of PVA in DI water. As PVA doesn't dissolve properly, we magnetically stir the beaker on *Tarson's Hotop-Digital Hot Plate* (at rpms greater than 300). It was left overnight (stirred for around 17 hours). In the morning, we got a homogenous solution.
3. Dragonskin surfaces were cleaned with IPA solution and kept inside hot air oven for 40 minutes at 110° C
4. Then it was plasma treated for 5 minutes and immersed in PVA solution for 10-15 minutes.
5. After that, it was dried in ambient conditions. Then, it was heated at 110°C for 15 minutes on *Tarson's Hotop-Digital Hot Plate*. The Dragonskin was kept on copper taped aluminium foil as it was found that on partial heating, Dragonskin sticks to aluminium somewhat and could be difficult to be separated from aluminium foil.
6. Later, it was cooled to room temperature. It was found that wettability of surface has enhanced to a good extent.

OBSERVATION & CONCLUSION:

Compared to PDDA-PSS coating, PVA coating makes the surface more wettable. Unlike the case of chalk coating, PVA coating seems to be more uniform to the naked eye.

Almost similar to the case of PDDA-PSS coating, PVA coating can be removed by continuous wiping with materials such as tissue paper.

In conclusion, the result of the PVA coating is demotivating, accounting for the long waiting times required in the procedure.

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APPENDIX A

SAMPLE CALCULATION FOR FINDING SILVER LOADING

Reading No.1:

Wt. of Cartridge+DI water = 19.602 g

(Wt. of Cartridge+DI water) after 5 successive prints = 19.376 g

DI water used for 5 prints = 19. 376- 19.602 g = 0.226 g

DI water used for a single print = $\frac{0.226}{5} = 0.045$ g

Density of DI water at room temperature (32°C), $\rho=0.995$ g/mL

Volume of DI water used for a single print = $0.995 \times 0.045 = 45$ μ L

Area printed=5 cm square=25 cm²

DI water used for a single print per unit area = $\frac{45 \mu\text{L}}{25 \text{ sq.cm}} = 1.8 \mu\text{L}/\text{cm}^2$

1 M solution of AgNO₃ was prepared, i.e. 5 mL DI Water has 0.850 g AgNO₃.

Amt. of AgNO₃ used for a single print per unit area= $0.85 \text{ g} \times \frac{1.8 \frac{\mu\text{L}}{\text{cm}^2}}{5 \text{ mL}}$
 $= 0.306 \text{ mg}/\text{cm}^2$

Mass fraction of Ag in 1 g of AgNO₃= ratio of mol wt of Ag to AgNO₃= 0.635

Amt. of Ag (Silver loading) = $0.635 \times 0.306 = 0.194 \text{ mg}/\text{cm}^2$

APPENDIX B

BRIEF DESCRIPTIONS OF TECHNIQUES/INSTRUMENTS USED IN THIS PROJECT

B1. Sonicator

Sonication is the act of applying sound energy to agitate particles in a sample. Ultrasonic frequencies (>20 kHz) are usually used, leading to the process also being known as **ultrasonication** or **ultra-sonication**. In the laboratory, it is usually applied using an *ultrasonic bath* or an *ultrasonic probe*, colloquially known as a *sonicator* [Wikipedia].

Dispersion and deagglomeration by ultrasonication are a result of ultrasonic cavitation. When exposing liquids the sound waves that propagate into the liquid result in alternation high-pressure and low-pressure cycles. This applies mechanical stress on the attracting forces between the individual particles. Ultrasonic cavitation in liquids causes high speed liquid jets of up to 1000 km/hr. Such jets press liquid at high pressure between the particles and separate them from each other. Smaller particles are accelerated with the liquid jets and collide at high speeds. This makes ultrasound an effective means of dispersing but also for the milling of micron-size and submicron-size particles.

Reference: https://www.hielscher.com/nano_01.htm



Fig 22: Branson 2510 Sonicator (Image Source: www.google.com/images)

B2. Plasma Treatment

In plasma, gas atoms are excited to higher energy states and also ionized. As the atoms and molecules 'relax' to their normal, lower energy states they release a photon of light, this results in the characteristic “glow” or light associated with plasma. Different gases give different colours. For example, oxygen plasma emits a light blue colour. A plasma's activated species include atoms, molecules, ions, electrons, free radicals and photons in the short wave ultraviolet (vacuum UV, or VUV for short) range. This mixture then interacts with any surface placed in the plasma.

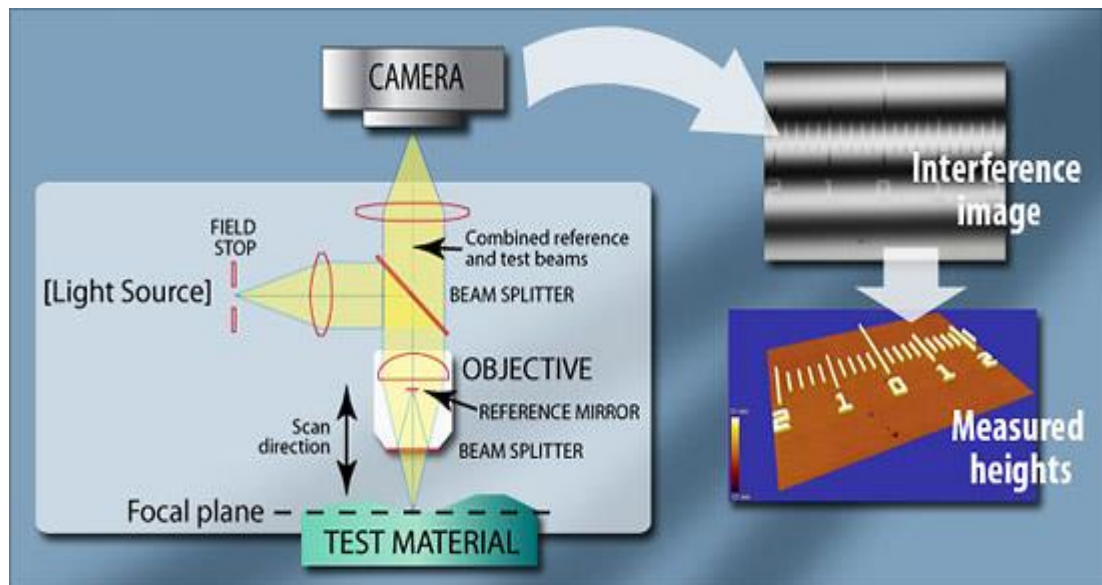
If the gas used is oxygen, the plasma is an effective, economical, environmentally safe method for critical cleaning. The VUV energy is very effective in the breaking of most organic bonds (i.e., C–H, C–C, C=C, C–O, and C–N) of surface contaminants. This helps to break apart high molecular weight contaminants. A second cleaning action is carried out by the oxygen species created in the plasma (O_2^+ , O_2^- , O_3 , O, O^+ , O^- , ionised ozone, metastable excited oxygen, and free electrons). These species react with organic contaminants to form H_2O , CO, CO_2 , and lower molecular weight hydrocarbons. These compounds have relatively high vapour pressures and are evacuated from the chamber during processing. The resulting surface is ultra-clean. If the part to be treated consists of easily oxidized materials such as silver or copper, inert gases such as argon or helium are used instead. The plasma activated atoms and ions behave like a molecular sandblast and can break down organic contaminants. These contaminants are again vapourised and evacuated from the chamber during processing.

References: Wikipedia; plasmaetch.com

B3. Optical Profilometry

In Profilometry, we study the topography of a surface. This can be surface morphology, roughness, analysis of cracks, scratches, heights of crests & troughs, etc. Two types of Profilometers are present- Contact type and Non-contact type. An Optical Profilometer is a non-contact type. The elementary working principle of the Optical Profilometer is illustrated in the Fig 21.

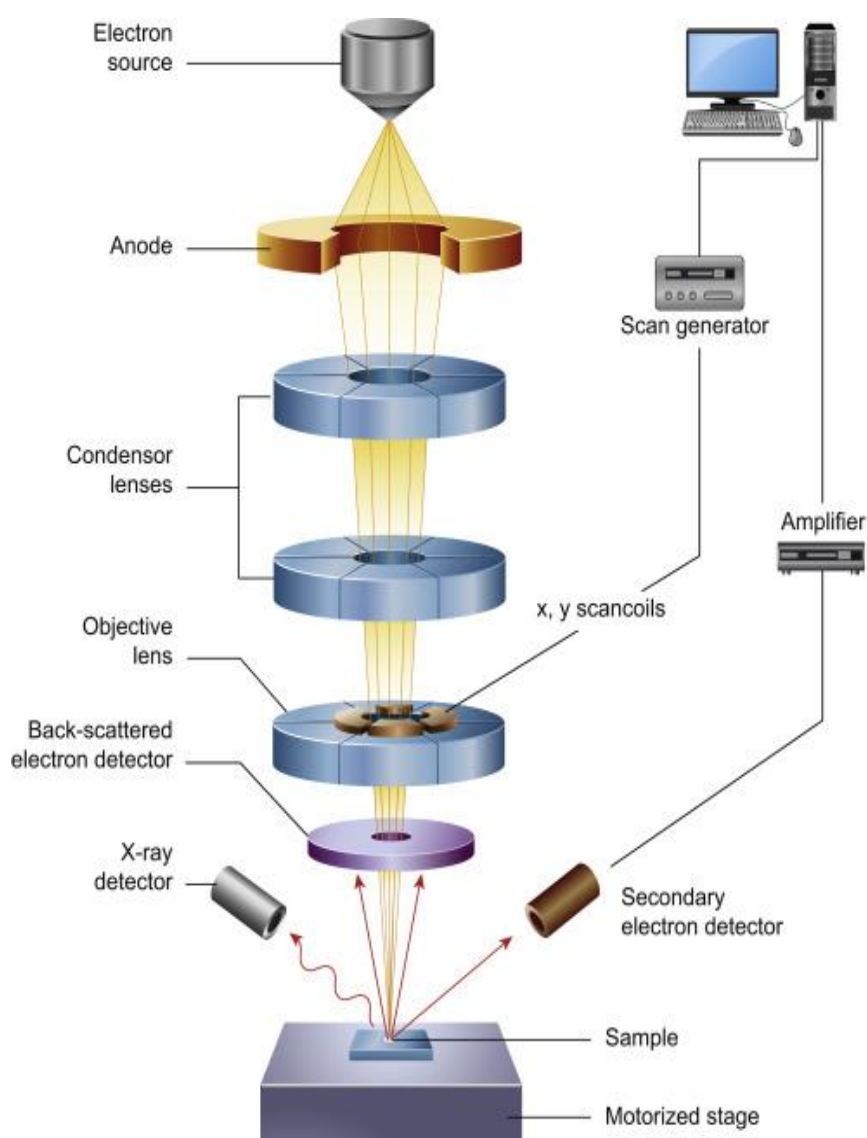
In our project, we had used an Optical Profilometer to understand surface morphology of samples obtained from printing and direct development (without exposure). But this analysis was a failure as AgNPs fabricated on the substrate did not reflect the light considerably.



*Fig 23: Illustration of working of an Optical Profilometer
(Image Source: www.zygo.com)*

B4. SEM (Scanning Electron Microscopy)

The purpose of SEM is that it provides detailed images of the surfaces of the analyte. SEM focuses on the sample's surface and its composition, so SEM shows (only) the morphology of samples. SEM is based on scattered electrons, i.e. electrons emitted from the surface of a specimen. Fig 22 illustrates the main features of SEM. While sample preparation, it is coated with a thin layer of a heavy metal such as gold or palladium. Image is formed when the secondary or backscattered electrons arising from interaction of electron beam and metal coated specimen are collected. The resulting image is displayed on a computer screen. SEM can resolve objects as close as 20 nm. The



magnifying power of SEM is up to 50,000X. A major advantage of SEM is that it allows for a large amount of sample to be analysed at a time. Also, SEM provides a 3-dimensional image.

Reference:
microbeonline.com

Fig 24: Features of SEM (Image Source: sciencedirect.com)

B5. TEM (Transmitting Electron Microscope)

Transmission electron microscope is used to view thin specimens (tissue sections, molecules, etc). TEM can show many characteristics of the sample, such as internal composition, morphology, crystallization etc. Here, electrons are used as “light source”. TEM is based on transmitted electrons and operates on the same basic principles as the light microscope. The sample in TEM has to be cut thinner (70-90 nm) because electrons cannot penetrate very far into materials. Transmitted electrons hit a fluorescent screen giving rise to a “shadow image” of the specimen with its different parts displayed in varied darkness according to their density. The image can be studied directly by the operator or photographed with a camera (Fig 23). Advantage of TEM over SEM is that TEM has much higher resolution. It can resolve objects as close as 1 nm i.e. down to near atomic levels. The magnifying power of TEM is up to 2 million times. Unlike SEM, only small amount of sample can be analysed at a time using TEM. Also, TEM provides a 2-dimensional picture.

Reference: microbeonline.com

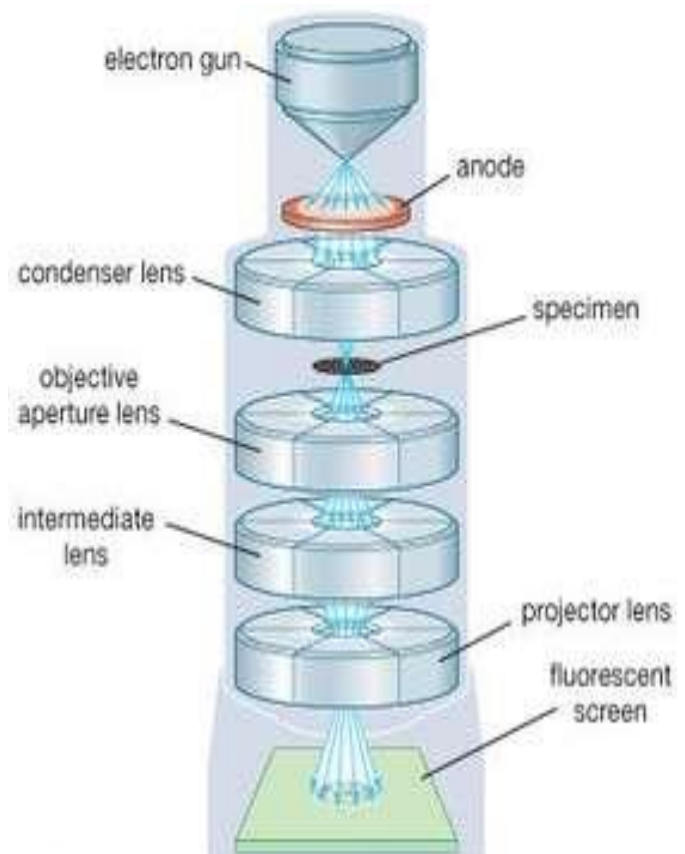


Fig 25: Features of TEM (Image Source: 2009 Encyclopaedia Britannica, Inc.)

APPENDIX C

CALIBRATION OF WEIGHING BALANCE

The weighing balance used in our lab is OHAUS® Adventurer™ Model AR0640. As per the user manual of this model (production discontinued), Adventurer™ balances are calibrated before shipment, however, calibration can be affected by changes in location, temperature, or rough handling.

AR0640 balance can be calibrated in two ways:

- **Span calibration:** Span calibration resets the balance's weighing range using **two** weight values- zero and a weight value at or near the balance's capacity.
- **Linearity calibration:** Linearity calibration minimizes deviation between actual and displayed weights within the balance's weighing range. **Three** weight values are used- zero, a weight value within the balance's weighing range and a weight value at or near the balance's specified capacity.

PROCEDURE:

It was checked whether the bubble towards the back of the weighing balance is properly aligned. This is done in order to ensure exact horizontal positioning of the balance. Both the types of calibration were performed as mentioned in the user manual of AR0640. For span calibration, 50 g weight was used and for linearity calibration, 20 g and 50 g weights were used. The weights used were BRASS BULLION STANDARD weights of Class 1 type which was present in our lab.

PRECAUTIONS:

Use gloves and tweezers while handling the weights. Also, wipe the weights clean using tissues.

CALIBRATION (Cont.)

Calibration Masses

Before beginning calibration, make sure masses are available. If you begin calibration and realize calibration masses are not available, exit the procedure by pressing and holding **Mode Off** until balance returns to weigh mode. The balance will retain previously stored calibration data. Calibration should be performed as necessary to ensure accurate weighing. Masses required to perform the procedures are listed in the following table.

CALIBRATION MASSES

CAPACITY	LINEARITY MASSES	SPAN ONLY MASSES
65g	20g/50g	50g
110g	50g/100g	100g
150g	100g/150g	150g
210g	100g/200g	200g
310g	100g/300g	300g
510g	300g/500g	500g
1500g	500g/1500g	1500g
3100g	1000g/3000g	3000g
4100g	2000g/4000g	4000g

Masses must meet or exceed ASTM Class 1 Tolerance.
Calibration masses are available as accessories.

Span Calibration

1. With the balance turned ON, press and hold **>O/T<** until **CAL** is displayed.
2. Release **>O/T<**, **-C-** is momentarily displayed followed by the value of the calibration mass which is to be placed on the pan.

Do not disturb the balance when -C- is displayed. Incorrect calibration may result.

3. Place the indicated span calibration mass on the pan.
4. Press **>O/T<**, **-C-** is momentarily displayed, then the weight of the mass on the pan is displayed.
5. Remove the calibration mass from the pan. The balance is now calibrated and returns to the weighing mode.

Linearity Calibration

1. With the balance OFF, press and hold **>O/T<** until **MENU** is displayed, then release it and **UNITS** is displayed. Press **Mode Off**, **LIN** is displayed.
2. Press **>O/T<**, **-C-** is displayed followed by the value of the first mass which must be placed on the pan.
3. Place the required mass on the pan and momentarily press **>O/T<**, **-C-** is displayed followed by the value of the next mass to be placed on the pan.

Do not disturb the balance while -C- is displayed.

4. Place the second required mass on the pan and momentarily press **>O/T<**, **-C-** is displayed. When the weight on the pan is displayed with the stability indicator, the balance is calibrated and returns to the weighing mode.
5. Remove the calibration masses from the pan.

CARE AND MAINTENANCE

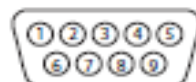
To keep the balance operating properly, keep the housing and platform clean. If necessary, a cloth dampened with a mild detergent may be used. Keep calibration masses in a safe dry place.

RS232 COMMAND DATA TABLE

Command Character	Description
P	Print command
T	Tare command
?	Displays current mode
XS	X=0 (zero) Stable off
XS	X=S Stable on (default setting in balance)
XS	X=A Auto print on stability

NOTE: Print commands entered through the computer are temporary. When the balance is turned off, it will return to balance menu settings when turned on again.

- 1 N/C
- 2 Data Out (TXD)
- 3 Data In (RXD)
- 4 N/C
- 5 Connected to pin 8
- 6 N/C
- 7 Ground
- 8 Connected to pin 5
- 9 N/C



TROUBLESHOOTING

SYMPTOM	PROBABLE CAUSE	REMEDY
No Display.	Power Adapter not connected.	Connect AC Adapter.
Incorrect weight reading.	Balance out of calibration. Balance was not zeroed before weighing.	Calibrate the balance. Press >O/T< with no weight on the pan, then weigh item.
Calibration procedure does not work.	Incorrect calibration masses being used.	Use correct masses.
Unable to display weight in a particular weighing unit.	Weighing unit not activated in menu.	Use Units menu to set desired units ON (see menu).
Balance won't store selections made in menu.	END selection was not used to exit menu.	You must use END to exit each menu and save selections.