CHAPTER 4

Experimental arrangement and equipments used

4.1 Introduction

Sample preparation, experimental measurement and technique used play a vital role in experimental research work. In this chapter, detailed discussions on the methodology of powdered sample preparation, fabrication of thin film, characterization of thin film and other important techniques used to study the different electrical and optical parameters of the material have been discussed.

4.2 Materials used for sample preparation

Pure aniline, $\text{C}_6\text{H}_5\text{NH}_2$ (99.99% purity) in liquid state was procured from M/S Sigma-Aldich Chemical Co., USA. It is the main ingredient to prepare polymerized aniline i.e. polyaniline in the laboratory. Other materials used in the preparation of samples were hydrochloric acid (HCl) (99.9% purity) and ammonium peroxysulphate ($\text{NH}_4\text{S}_2\text{O}_8$) (99.9% purity) were collected from Across Organics, Mumbai. Potassium tetrachloroaurate (KAuCl₄) of 99.9% purity) was procured from Sigma-Aldich Chemical Co., USA. 1-methyl 1-2 pyrrolidinone (NMP of 99.9% purity) and dimethyl sulfoxide (DMSO of 99.9% purity) were procured from Merck Specialities Pvt. Ltd., and Fine-Chem Ltd., Mumbai respectively and used as suitable solvent for polyaniline. For fabrication of junctions with aluminium metal (99.99% purity) and zinc oxide ZnO (99.99% purity) the materials were purchased from Sigma-Aldich Chem Co., USA. Some other materials like Ag (99.9% purity), In (99.9% purity), CdSe (99.9% purity) and Zn (99.9% purity) were procured from Across Organics, Mumbai, India.

The indium tin oxide (ITO) coated glass substrates were purchased from Sigma-Aldich Chemical Co., USA. Better quality molybdenum boats, collected from Vacuum
Techniques Pvt. Ltd., Bangalore, used for thermal evaporation of Al, ZnO, Zn, In and Ag to fabricate junctions and electrodes etc. as required. In some cases, instead of boat, other types of heating coils like helix, basket etc. were also used, depending upon the suitability of the materials for evaporation.

4.3 Sample preparation

4.3.1 Chemical synthesis of polyaniline powder

Chemical synthesis of polyaniline in the form of emeraldine salt was done by the general procedure using redox polymerization of aniline in presence of an oxidant, ammonium peroxidosulphate (APS) and using HCl as dopant. Freshly distilled aniline (4 ml) in 50 ml of 1M HCl solution at 2 to 3 °C was stirred for 20 minutes and subsequently added 1M APS solution (25 ml) drop wise at a rate of 5 drops/minutes. The process continued till the whole quantity was added and the solution turned green. Stirring of the compound continued for about 3 hours after which the solution was kept undisturbed for overnight. Next day, the precipitate was treated with tetrahydrofuran to eliminate other oligomers and was filtered using funnel. The product was dried in an oven for 24 hours at 45 °C to get the powder [163].

4.3.2 Chemical synthesis of gold nanocomposite polyaniline powder

For nanocomposite materials, polyaniline was synthesized by normal procedure of oxidation polymerization of aniline hydrochloride using ammonium peroxidosulphate as an oxidant. Freshly distilled aniline (8 mL) in 200 mL of 1M HCl solution at 3 °C (keeping in ice chamber) was stirred for 15 minutes and subsequently 1 M APS solution (50 mL) was added to it at the rate of 5 drops per minute thereafter added the gold solution (KAuCl₄, 50 mL) freely by opening the burette to the main solution. The stirring of solution was continued for about 2 hours as before and the resulting solution
was kept overnight for complete polymerization. The precipitate was washed out by using first tetrahydrofuran (3 mL tetrahydrofuran in 15 mL distilled water) and secondly by sodium hydroxide (NaOH) solution to eliminate any oligomers and then dried in oven at 45 °C for 20-24 hrs. Thus the powdered gold nanocomposite polyaniline sample was obtained [164].

4.4 Substrate selection

Substrate is the supporting materials on which thin film is deposited. It may be crystalline, amorphous or polycrystalline depending on the nature of the film to be deposited on it. The surface of substrate influences thin film properties considerably. The substrate should have the desirable properties [165] e.g. the surface should be highly smooth and perfectly flat, high mechanical strength to enable the substrate to withstand strain during processing and monitoring, high resistivity, good thermal conductivity, it should have same co-efficient of thermal expansion as that of the deposited film, nonporous, low cost and easily available etc.

The commonly used substrate materials are glass, mica, ceramic, stainless steel etc. Glass is the most widely used substrate as it satisfies most of the properties required. In this study microscopic glass slides of thickness 1.35 mm (Upen Instrument, Nashik, India) were used. For fabrication of junction and to study its properties, the surfaces of the substrates should be conducting. To make it conducting a metallic layer is required on its surface as base electrode. Generally, indium tin oxide (ITO) layers are deposited on it as conducting medium and named as indium tin oxide (ITO) coated glass substrate. In this case ITO layer serves as one of the electrodes. Substrates of appropriate sizes (2.5x2.5 cm² and 1.8x2.5 cm²) were then cut out from the glass or ITO coated glass slides for use.
4.5 Cleaning of substrate

To have the proper adhesion and formation of a thin film, the surfaces of the substrates should be highly clean and free of all contaminations. Sticking of different impurity atom, fingerprints, dust particles etc. are the sources of contaminants. The removal of these contaminants using different cleaning process mainly depends on the types of substrates and contaminants present. Different types of chemical reagents e.g. acid, alkali or alcohol with proper concentrations are used to remove the contaminants by breaking the bonds between the contaminants molecules as well as between the contaminants and substrates. Acid removes any oxide layer and greases into water soluble compounds. The ultrasonic cleaning process is also used to remove all types of contaminants e.g. greasy particles, fingerprints etc. It enhances the dissolution of residues sticking on the substrate by the intense local stirring action by the shock waves created in the solvent.

The substrates of appropriate sizes 2.5x2.5 cm$^2$ and 2.5x1.8 cm$^2$ cut from the ITO coated glass slides were first washed with ordinary detergent solution and then these were treated in a mixture of nitric acid and isopropyl alcohol. These substrates are then washed by freshly prepared distilled water. Also these substrates were thoroughly cleaned with methanol and then rinsed with deionized water. To avoid recontamination of any particles, dust etc. proper drying and storing of cleaned wet substrates is important and utmost precautions were taken in drying the substrates in an atmosphere free from any contaminations. In case of glass substrates, the desired size substrates were kept in a solution of potassium dichromate for 24 hours and then washes with distilled water. This was then again boiled in solvent like methanol for further cleaning. These were finally rinsed in deionized water. The substrates are kept vertically in a clean
petry dish to avoid water stick mark on it. After that these were put inside a cleaned closed stainless steel oven for drying for an hour at about 80 °C.

4.6 Designing of masks for sample preparation

To study the various characteristics of the films and junctions, different convenient shapes are given. It is obtained by masking substrates during the deposition of materials on the substrates. The materials of the masks should be chemically inactive with the vapour atoms and stable over the temperature range used for the deposition of the materials. In our purpose, freshly and chemically clean good quality mica sheet were used for making different shaped masks according to need. These were cut by razor blades and micro punch. Aluminium and molybdenum foils may also be used as mask. The masks were cleaned by acetone, distilled water and finally dried properly by hot air blower.

4.7 Preparation of thin film for study

4.7.1 Drop coating method for polyaniline film

The powdered sample of polyaniline was dissolved in 1-methyl 1,2-pyrrolidinone (NMP) in a beaker with continuous stirring for about 4 hours. The solution was filtered for use and drops of the solution with the help of small syringe were placed over a chemically cleaned glass slide using as substrate. The drops were spread over by small glass spoon to make it in the form of film. For fabrication of junction, cleaned ITO coated glass substrate was used. The substrate was dried in a specially made vacuum chamber which was fitted with a heater and a temperature controller. The temperature was maintained at 45 °C for drying in vacuum for about 4 hours. Four sets of ultra thin films of (p)polyaniline in the ranges from 200 Å to 400 Å were produced in a cycle to study various parameters. One set of film so produced was
taken to the thermal evaporation unit for deposition of electrodes. For gap type cell geometry, sample was made by using two co-planer electrodes separated by a gap to study electrical parameters. Two electrodes of pure silver were deposited onto the film prepared on glass substrate keeping a gap of 1 mm using a mask. For fabrication of junction, high purity Al foil for Schottky barrier or (n)ZnO material (99.99% purity) for heterojunction was vacuum deposited onto the (p)polyaniline film prepared on ITO coated glass substrate in the form of small disc or rectangular shaped electrodes. The schematic diagrams of gap type and sandwich type structures are shown in Figure 4.1.

4.7.2 Drop coating method for gold nanocomposite polyaniline film

In the similar manner as discussed above, powdered sample of gold nanocomposite polyaniline was dissolved in 1-methyl 2-pyrroldinone (NMP) in a beaker with continuous stirring for about 4 hours. The solution was filtered and drops of it were placed over a glass slide already cleaned by chemical wash and subsequently in ultrasonic bath. The drops were spread over by small glass spoon to make it in the form of film. For fabrication of junction, ITO coated glass substrate was used after proper cleaning. This was dried in a specially made vacuum chamber which was fitted with a heater and a temperature controller. The temperature was maintained at 45°C for drying in vacuum for about 4 hours. Four sets of ultra thin films of (p)gold nanocomposite polyaniline in the ranges from 200 Å to 500 Å were produced in a cycle to study various parameters. One set of film so produced was taken to the thermal evaporation unit for deposition of electrodes. For gap type cell geometry, sample was made by using two co-planer electrodes separated by a gap to study electrical parameters. Two electrodes of pure silver were deposited onto the film prepared on glass substrate keeping a gap of 1 mm using a mask. For fabrication of junction, high purity Al foil for Schottky barrier or
(n)ZnO material (99.99% purity) for heterojunction was vacuum deposited onto the (p)gold nanocomposite polyaniline film prepared on ITO coated glass substrate in the form of small disc or rectangular shaped electrodes. The schematic diagrams of gap type and sandwich type structures are shown in Figure 4.1.
Figure 4.1: Schematic diagrams of the samples (a) gap type, (b) sandwich type with circular type barrier material, (c) sandwich type with rectangular shaped barrier material, (d) sandwich type with circular shaped barrier material and connecting electrode and (e) sandwich type with rectangular shaped barrier material and connecting electrode.

4.7.3 Electrochemical method of preparation for polyaniline and gold nanocomposite polyaniline film

The preparation of polyaniline and gold nanocomposite polyaniline thin film were performed by electrochemical method using Autolab Potentiostat/Galvanostat (EcoChemie, Netherlands, Model 101N) with NOVA software in which working electrode was replaced with PANI/ITO and AuNPs/PANI/ITO. Platinum wire and Ag/AgCl (3 M KCl) were used as counter and reference electrodes respectively. Prior to the electro-polymerization, the ITO coated glass plate (0.80 cm²) was thoroughly cleaned with methanol and then rinsed with de-ionized water. Polymerization of aniline was achieved in a potentiodynamic mode in 0.2 M aniline per 1 M HCl solution following methodology of Radhapyari et al. [166]. The nanocomposites of AuNPs-PANI were prepared by electrochemical deposition of a mixture of HCl (1.0 M), aniline (0.2 M) and 500 µL AuNPs (2 mg mL⁻¹ of KAuCl₄) and thoroughly sonicated for 15 minutes; introduced in a three-electrode electrochemical cell of Autolab Potentiostat/Galvanostat (EcoChemie, Netherlands, Model 101N). The cell consists of
Ag/AgCl (3 M KCl) as reference, Pt wire as counter electrode and ITO coated glass plate (0.80 cm²) as working electrode. The electro-polymerization was demonstrated at scan rate of 20 mVs⁻¹ for seven (7) cycles in the potential range -2.0 V to 1.1 V. The p-type AuNPs/PANI/ITO structure formed on the electrode is washed with de-ionized water to clean off the untreated AuNPs-PANI film. Two set of films were prepared in the range of 110 nm to 130 nm thickness.

4.8 Measurement of film thickness

The thickness of the fabricated films was measured by profilometer (Vecco Dektak 150) at IIT, Guwahati, India by scratching the films carefully with a sharp tweezers or a toothpick avoiding to scratch into the substrate. Thicknesses were measured in three different positions of the films and average was calculated. Results have been discussed in the appropriate chapter of results and discussions.

4.9 Microstructural, morphological and compositional analysis

The structural, morphological and compositional analyses are the important parts of any study. The materials can be characterized structurally, physically and chemically with the help of sophisticated analytical instruments viz. X-ray diffractometer (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM) etc.

4.9.1 X-ray diffraction studies of the sample

X-ray diffractometer is a very useful analytical instrument for determining the whole range of detail information viz. amorphous, crystalline, polycrystalline, crystal structure, orientation, crystalline size, lattice constant, defects, stresses and strain developed in the samples etc. Philips X-ray diffractometer (Philips X’ Pert Pro) with CuKα radiation of wavelength 1.54056 Å was used to make X-ray diffraction spectra. The diffractometer operated at 40 KeV and 30 mA. X-ray diffractogram analysis
including the peak search was done by computer programming Philips X’ pert software.
XRD pattern of all the samples were found to be similar to other workers in this area.
The details report of the samples has been discussed in the appropriate chapters.

4.9.2 Scanning electron microscope (SEM) analysis

In order to know the morphology of polyaniline sample and presence of gold
nanoparticles in the gold polyaniline matrix, the scanning electron microscope was
used. The SEM spectra were taken using SEM (JEOL Asia PTE Ltd. Singapore/JEOL
JSM) with operational voltage of 15 KV at Tezpur Central University, India.
Observations have been reported in the results section of the film.

4.9.3 Compositional analysis

The quantitative elemental compositional analysis of the prepared samples was
done using energy dispersive X-ray analysis (EDAX) at IASST, Guwahati, which
informed the percentage of different elements present in the sample. Details have been
explained in the appropriate chapter.

4.9.4 Transmission electron microscope (TEM) analysis

To know the morphology of polyaniline sample and presence of gold
nanoparticles in the gold polyaniline matrix, the transmission electron microscope was
used (Model-JEM-2100) at North Eastern Hill University, Shillong, India. Observations
have been reported in the results section of the film.

4.10 Optical analysis of the thin films

4.10.1 UV-Visible spectrometer study

For optical transmission, absorbance and reflectance measurement of the
sample, a UV-Visible spectrometer (Shimadzu, Model UV-1800) was used in CSIR-
NEIST, Jorhat. The range of spectrometer is 200 nm to 900 nm.
4.10.2 FTIR analysis of the films

To study the chemical bonding and functional groups present in the sample, Fourier transformed infrared (FTIR) spectrometer (Perkin Elmer System 2000) was used at CSIR-NEIST, Jorhat, India.

4.10.3 Photoluminescence study of the films

Photoluminescence spectra of polyaniline and gold nanocomposite polyaniline were recorded to study the effect of illumination (using PL setup Perkin Elmer Singapore PTE Ltd, Singapur) at Tezpur Central University, India.

4.11 Thermogravimetry analysis (TGA)

The stability of polyaniline and gold nanocomposite polyaniline with respect to temperature were studied with the help of thermogravimetry analysis instrument (recorded by STD Q600 V 20.9 Built 20) at CSIR-NEIST, Jorhat, India.

4.12 Deposition of electrode materials for studying electrical properties of thin films and junctions

4.12.1 Vacuum coating unit

To deposit metals, alloys and compounds which involve evaporation of the material in vacuum, vacuum coating unit is used, where high vacuum is created to minimize the interaction between materials of the newly fabricated film and residual gases. In our purpose this unit has been mainly used to deposit metal e.g. aluminium and inorganic substances e.g. ZnO etc. on polyaniline and gold nanocomposite polyaniline films to prepare Schottky junctions and heterojunctions respectively. Also for using as proper electrodes materials to the films for various measurements, metallic films were deposited by vacuum coating unit. Schematic layout of a vacuum coating unit with different important parts has been drawn in Figure 4.2. In these studies, for
fabrication of junctions (both Schottky junctions and heterojunctions) and electrodes materials on the films, a vacuum coating unit [Vacuum Techniques (P) Ltd., Bangalore-58 (Model VT-ACG-03)] was used. A diffusion oil pump was used to evacuate the chamber having speed of about 500 lit/sec backed by a double stage rotary pump, run by a single phase motor with a magnetic admittance cum isolation valve and having suction capacity of about 300 lit/min. This diffusion pump is capable of producing vacuum in the range of $10^{-5}$ to $10^{-7}$ torr. A Pirani and a Penning ionization gauge were used to measure the vacuum created by the pumps, inside the chamber. The diffusion pump is connected to the chamber through a water cooler isolation valve called baffle valve which could adequately baffle direct entry of oil vapour molecule from the diffusion pump to the chamber. The coating chamber mainly consists of a bell jar approximately 500 mm diameter, made of stainless steel fitted with an L-shaped neoprene gasket at its base. The system was provided with air admittance valve for admitting air, needle valve for introducing any gas if required to be, a movable shutter, which can be moved from the outside is fitted inside the chamber to control the deposition on the substrate. The bell jar with the gasket is placed on a stainless steel base plate which is firmly joined by welding to the top of the baffle valve and is fitted with numbers of electrodes to facilitate external connections for heater, thermocouples, thickness monitor unit, power supply etc. A filament source heater in the form of boat or helical or basket shaped coil were clamped at the two ends of two upper plates and fixed by steel screws. Before every deposition it is required to clean all the parts like, bell jar, neoprene gasket base plate and all other elements inside the bell jar. A specially designed aluminium circular plate having numbers of slits of different sizes are kept inside the chamber. The substrates and the masks are kept on that plate for exposing to the vapour. The mask
holder was supported by three metallic rods within the bell jar, the distance of which from the source heater could be adjusted easily. Arrangement of heater source, mask, substrate, sample etc. inside the chamber is shown in Figure 4.3.

Figure 4.2  Vacuum Coating Unit. (A, Bell jar; B, Neoprene rubber gasket; C, Base plate; S, Substrate holder; R, Shutter; D, Filament; G and F, Pressure gauge; N, Vent; H, Baffle; I, Diffusion pump; J, Water cooling coil; K, Diffusion pump heater; P, Rotary pump; O, Magnetic valve fitted to the pump)

Figure 4.3: Schematic diagram of the thermal evaporation chamber
4.12.2 Electrodes deposition for gap type sample

![Diagram](image)

(a) Glass substrate

![Diagram](image)

(b) Glass substrate, Polyaniline thin film

![Diagram](image)

(c) Glass, Gap of polyaniline film, Electrode material

Figure 4.4: Fabrication of gap type structure (a) glass substrate, (b) polyaniline thin film on glass substrate and (c) gap type polyaniline sample on glass and silver electrode

For the study of electrical conductivity at different environments viz. at vacuum, air and gases gap type samples were prepared. In the present case, silver metal was selected as electrode materials as it makes ohmic contact to polyaniline and it has high conductivity and good adhesion with glass substrates also has mechanical stability with
low stress [167]. For gap type structure, rectangular shaped semiconductor film was first fabricated on glass substrate by drop coating method. Over this film two rectangular films of silver with a spacing of 2 mm was vacuum deposited using suitable mask. These two metallic films were used as electrodes for measurement (Figure 4.4).

4.13 Junction fabrication for these films

Metal-semiconductor junction (Schottky junction) and heterojunction were fabricated for studying the junction characteristics and various parameters.

4.13.1 Metal-polymer junction

[(p)polyaniline and (p)gold nanocomposite polyaniline with aluminium metal]

![Diagram showing the structure of the junctions](image)

(a) Indium Tin Oxide
Glass substrate

(b) Barrier material Al circular shape
Polyaniline thin film
Indium Tin Oxide coating
Glass substrate

(c) Barrier material Al rectangular shape
Polyaniline thin film
Indium Tin Oxide coating
Glass substrate

Figure 4.5: Fabrication of sandwich type structure (a) ITO coated glass substrate, (b) Al/(p)polyaniline junction (circular type) on ITO coated glass substrate and (c) Al/(p)polyaniline junction (rectangular type) on ITO coated glass substrate
To study the different parameters of metal-semiconductor junction aluminium metal (Aluminium foil of 99.99% purity) was deposited onto polyaniline film to form Schottky barrier junction. Sandwich type junction was fabricated on the ITO coated glass substrate. The ITO layer on the glass substrate works as base electrode for ohmic contact. On this ITO layer polyaniline film or gold nanocomposite polyaniline film were deposited by electrochemical method (discussed above). The barrier metal aluminium was deposited onto the films in the form of small disc shaped spots of areas 0.01 cm$^2$, 0.03 cm$^2$ and 0.05 cm$^2$ by vacuum deposition using suitable mask. These form the ITO/(p)polyaniline/aluminium and ITO/(p)gold nanocomposite polyaniline/aluminium Schottky junctions as desired. The schematic diagram of the junctions is shown in Figure 4.5.

4.13.2 Preparation of polymer heterojunctions

[(n)ZnO/(p)polyaniline and (n)ZnO/(p)gold nanocomposite polyaniline heterojunction]

To study the (n)ZnO/(p)polyaniline and (n)ZnO/(p)gold nanocomposite polyaniline heterojunctions sandwich type samples were prepared. These junctions were prepared over ITO coated glass substrates. ZnO (99.99% purity) was then vacuum deposited onto the already prepared polyaniline films in the shape of small disc or rectangle using suitable mask. Indium metal was used as upper electrode which was vacuum deposited over ZnO disc. Thus a ITO/(p)polyaniline film/(n)ZnO/In heterostructure was made. The schematic diagrams of the junctions are shown in Figure 4.6.
4.14 Equipments used for measuring various parameters

4.14.1 Source meter

For almost all the measurements of electrical parameters, a source meter was used. With this equipment, measurement can be accurately made and computer can be connected for monitoring and storage of data for different characteristics. In the present case, different electrical characteristics of films and junctions including gas sensing properties, Keithley Source Meter (model 2611A, 1-Channel, 100fA, 200V/100nV, 1.5DC/10A Pulse) with LabTracer software was used.

Figure 4.6: Fabrication of sandwich type heterojunctions (a) (n)ZnO/(p)polyaniline heterojunction (circular type) on ITO coated glass substrate with In electrode and (b) (n)ZnO/(p)polyaniline heterojunction (rectangular type) on ITO coated glass substrate with In electrode
4.14.2 LCR-Q meter for capacitance-voltage characteristics

The capacitance developed in the Schottky junction and heterojunction at different bias voltages was measured using an autocompute LCR-Q meter (Aplab 4910) with measuring frequency at 1 KH. The samples were kept in a shield chamber to avoid any noise during measurement. The different bias voltage was supplied using a variable D. C. power supply (Scientific Electronic Model: PS 25). The schematic diagram is shown in Figure 4.7.

![Diagram](image.png)

Figure 4.7: LCR-Q meter for capacitance-voltage characteristics

4.14.3 Sample holder

![Diagram](image.png)

Figure 4.8: Schematic diagram of the sample holder
It is a specially designed device to hold the sample properly during measurement. This was a thick rectangular teflon plate. This plate was fixed inside a rectangular frame of perspex by four screws. The electrodes, in the form of strips were fixed at desirable length of the film which could hold the sample by spring action. The sample holder was properly placed inside a shielded box to avoid outside disturbance. The schematic diagram of the sample holder is shown in Figure 4.8.

4.15 Arrangement for measurement

4.15.1 Experimental arrangement for measuring electrical parameters

All electrical measurements were done in a specially designed chamber as shown in Figure 4.9. The chamber can be evacuated if required by a rotary pump. The sample holder with sample is kept inside the chamber. The source meter is connected to the sample through the feed throughs in the chamber. The source meter is computer controlled by the software, Lab Tracer provided with the equipment. The sample can be kept at desired temperature by a digital temperature controller fitted in the chamber. The temperature is sensed by a thermocouple connected to the sample.

![Figure 4.9: Experimental arrangement for studying various electrical parameters of films and junctions](image-url)
4.15.2 Arrangement for studying gas sensing properties

A separate arrangement was made for studying gas sensing properties of the films and junctions. The detailed process is reported below.

4.15.2.1 Preparation of sample for gas sensors

The ultra thin films of polyaniline and gold nanocomposite polyaniline prepared on glass substrate were taken to a vacuum evaporation unit for deposition of electrodes. Gap type cell configurations (as shown in Figure 4.4) were used for measurement. For this, pure silver (99.99%) metal was vacuum evaporated on to the film keeping a small gap using suitable mask. The masks were specially prepared for this type of cell configuration of film of area $13.8 \times 10^{-2}$ cm$^2$.

4.15.2.2 Arrangement for measurement

The samples were kept in a specially designed glass chamber fitted with inlet and outlet pipes. This chamber can be evacuated by a rotary pump fitted to the chamber. The chamber was fitted with a temperature controller to study the temperature effect on the gas sensitivity. Four probe methods were used to study the change of electrical parameters during the study. Pure carbon dioxide gas was taken from a gas cylinder fitted with regulator and was connected to the chamber. The sample was connected to DC power supply (Systronics) and the current produced in the sample was recorded by a digital precision multimeter (Fluke model no. 8846A). For the comparative study on the gas sensitivity, three states of chamber atmosphere of the samples were recorded viz, vacuum, air and CO$_2$ gas. Conductivities of the samples were measured first in air then in vacuum and finally in gases. The differences in readings in three cases were recorded. For gas measurement, the chamber was first evacuated to about $10^{-2}$ torr and then carbon dioxide gas was introduced at a small rate to occupy the space and the
corresponding conductivity of the sample was measured. The CO₂ gas sensitivity of the film at different temperatures was also recorded within the temperature range 303K – 333K to see the effect of temperature. The schematic arrangement for the measurement is shown in Figure 4.10 [168].

Figure 4.10: Schematic diagram for studying gas sensing properties of films
4.16 Photographs

Photograph 4.1: Four Indium Tin Oxide coated glass substrates ready for fabrication of film

Photograph 4.2: Polyaniline and gold nanocomposite polyaniline thin films (greenish colour) on ITO coated glass substrate
Photograph 4.3: Vacuum coating unit used for electrode deposition

(Vacuum Technique Pvt.Ltd.)

Photograph 4.4: Actual gap type samples of polyaniline and gold nanocomposite polyaniline fabricated for studying different parameters
Photograph 4.5: Junctions of Al/(p)polyaniline and Al/(p)gold nanocomposite polyaniline fabricated for studying different parameters

Photograph 4.6: Experimental arrangement with Keithley source meter (model-2611A) for studying the various electrical characteristics of thin films and junctions