Chapter 2

EXPERIMENTAL TECHNIQUES

2.1 Introduction

Analytical instruments are key to elucidate system parameters and characteristics. In the case of PS based devices the surface morphological, structural, optical, electrical and surface chemical features are to be analyzed, for characterization. In this chapter, the characterization techniques, and the instruments used for these studies are briefly given.

2.2 Formation of Porous Silicon

The porous silicon samples have been prepared using the electrochemical etching of silicon wafer. The figure 2.1. shows the experimental setup used in the preparation of Porous Silicon using a constant current source. The experimental arrangement consisted of a PVC container having the electrolyte, an inert cathode, Silicon anode and an ammeter. The cathode is a small piece of platinum foil. The n-type silicon wafer is the anode. In this experiment n-type silicon it is necessary to illuminate the wafer during the aniodization process in order to create the holes (h+) required by the chemical reaction to form the porous material [1,2]. So, light illumination on the wafer during the aniodization process is essential for the formation of n-type PS. i.e. the light illumination of the wafer during the aniodization process can be taken as a formation parameter.
for the 11-type PS. The electrolyte is prepared by using desired proportions of Hydrofluoric acid (HF) and ethanol. [3,4].

![Experimental setup for the fabrication of Porous Silicon](image1.png)

**Figure 2.1.** Experimental setup for the fabrication of Porous Silicon

2.3 Deposition of Doped Polymer Films

The optimized PS samples prepared using the electrochemical etching of silicon wafer have been coated with doped polymer by dip coating method. The doped polymer has been dissolved in Toluene and solutions of different concentrations of doped polymer have been made. The optimized PS samples have been dip coated in the polymer solutions to prepare the polymer / PS heterojunctions.

2.4 Scanning Electron Microscopy - SEM Study

The surface morphology, homogeneity and grain size of the PS samples have been studied by Scanning Electron Microscopy (SEM). Scanning Electron Microscope is the most versatile and commonly used
instrument to study the surface topography of the film. The low energy (50 eV) secondary electrons emitted from the surface of the specimen provide the basis for a different type of imaging. The electron beam can be concentrated to a small probe (~ 20 Å diameter) that may be deflected across the specimen in the faster fashion using scanning coils. The secondary electron can be detected above the specimen, and as image showing the intensity of secondary electrons emitted from different parts of the specimen can be displayed on a CRT. This scanning image is particularly useful for examining the morphology of metals, alloys, thin films of metal and semiconductors and crystalline materials. The convenience of Scanning Electron Microscopy is partly due to the long working distance between the final lens and the sample surface and partly due to the ability to directly study almost any free surface. Further SEM offers several modes of operation. The most widely employed is secondary electron imaging, which gives images of better than 100 Å resolutions almost unlimited depth of field and good contrast between most cell components [5].

JEOL JSM-5610 LV scanning electron microscope has been used for the morphological studies on PS samples. The SEM micrographs provide information about the surface homogeneity, surface morphology of the PS samples.
2.5 Structural Characterization- XRD Study

Structural characterization of PS using X-ray diffraction (XRD) analysis plays a vital role from fundamental research as well as applied research. Among the available structural characterization techniques, X-ray diffraction method is very simple, non-destructive and suitable for estimating the crystallographic parameters.

When an X-ray beam falls on matter, scattered X-radiation is produced by the atoms. These scattered waves spread out spherically from the atoms in the sample, and the interference effects of the scattered radiation from different atoms cause the intensity of the scattered radiation, to exhibit maxima and minima in various directions. The equipment consists of a X-ray generator, diffracting element, proportional counter and single channel pulse height analyzer. The specimen is mounted in the centre of the diffractometer and rotated by an angle around an axis in the film plane. The counter is attached to an arm rotating around the same axis by an angle 20. Only the \((hkl)\) planes parallel to the film contribute to the diffracted intensity.

The X-ray diffraction patterns of PS samples have been recorded by using the following configuration based X-ray diffractometer.

X-ray generator make & model : X’pert Pro
Filter : Nickel
Radiation : Cu K\(_a\) with wavelength =1.5412A
The diffraction peaks obtained were indexed using standard JCPDS (Joint Committee on Powder Diffraction Standards) cards and the crystalline parameters can be evaluated.

### 2.6 Photoluminescence and Electroluminescence Studies—Luminescence Spectrometer

Fluorescing samples (both films and solids) can be analyzed, using this spectrometer generally on excitation with higher energy radiation such as UV (with wavelength selected). These samples give light in the lower energy region (usually in the visible region), which when wavelength scanned, yields emission spectrum characteristics of the given luminescent sample.

Electromagnetic radiation from an Ultra violet-Visible source passes through a wavelength selected and through the cell as in a spectrometer. Unlike the measurement of absorption in a Spectrometer, however a portion of the emitted radiation that exits from the cell is measured. Because the luminescent radiation can be emitted in broad band that are centered at different wavelengths, a second wavelength selector is required in the path of the emitted radiation between the cell and the detector. The emitted radiation is not usually measured in-line with the exciting radiation, as in absorptive measurements, owing to possible spectral interference from the exciting radiation. Photoluminescence has been measured at many angles relative to the incident radiation and at many locations within the cell. The most common practice is to measure the emitted radiation of 90 degree
from the path of the exciting radiation and at the centre of the cell. The
signal from the detector is amplified, if required and routed to a read out
device.

High pressure xenon lamps emit a continuum of radiation that is
useful throughout the ultraviolet-visible and into the near-infrared spectral
regions. In order to emit constant intensity radiation at a particular
wavelength the lamp requires a stable power supply. In this context lasers
have proved to be useful radiative sources. Spectrometers are required,
whenever it is necessary to scan the wavelength of either excitation
radiation or the luminescent emission radiation.

Scanning instruments can be used to obtain two types of spectra. If
the wavelength at which the emission observed is held constant, the
wavelength at excitation occurs (excitation spectrum) can be scanned. If
the wavelength of the excitation radiation is fixed then the wavelength at
which the emission occurs (emission spectrum) can be scanned. If the
wavelength of the excitation radiation and the applied voltages are fixed
then the wavelength at which the emission occurs (electroluminescence
emission spectrum) can be scanned. For the present study (both PL and
EL), Perkin Elmer LS-55 luminescence spectrometer has been used.

2.7 Fourier Transform Infrared (FTIR) Spectroscopy

Infrared (IR) spectroscopy is one of the most powerful analytical
tools for elucidation of the molecular structures of inorganic and organic
compounds. It has a very wide range of applications from identification of compounds to material science in the semiconductor industry. From the IR analysis it is possible to identify the functional groups on surfaces and adsorbents, molecular composition of the materials. Infrared spectroscopy is based on absorption of IR radiation causing transition in the sample from one vibrational state to another, higher energy vibrational state [7].

Consequently, the infrared spectrum is a set of absorption bands whose intensity and frequency provide information of structure and bonding in the molecule. FTIR instruments employ interferometer techniques in the collection of spectral information and the spectrum is obtained as an inverse Fourier-Transform of the interferogram. FTIR spectroscopy is non-destructive, structurally specific and, unlike the high vacuum technique, FTIR is able to provide spectral insitu information about solid/liquid interfaces. In the present study, Shimadzu 8400s FTIR spectrometer having a wave number range of 4000 - 400 cm"1 has been used to identify the species and bonding on the surface layer of PS.

2.8 Current-Voltage (I-V) Characteristics

The doped polymer thin film has been deposited on PS so that p-polymer/PS/n-Si structure is formed. For ohmic contact at the back surface of n-Si substrate and the top surface of the polymer film, have been coated with silver paste and now Ag/doped polymer/PS/Si/Ag structure is obtained. When the polymer side is given a positive voltage and n-Si
substrate is given a negative voltage, the structure is forward biased. The circuit for measuring I-V is given in figure 2.2. For different currents (I), voltages (V) have been found and these values have been used to plot the I-V characteristics of the device. The reverse characteristics have also been similarly drawn.

![Figure 2.2 Forward Biasing of Doped Polymer/PS Heterojunction](image)

**Figure 2.2 Forward Biasing of Doped Polymer/PS Heterojunction**

### 2.9 Conclusion

In this chapter, a brief account of the characterization techniques and procedures which have been used to characterize the PS and doped polymer/PS are given.
2.10 References