Chapter 6

Nitrogen Modifications in Camphoric Carbon Films

6.1 Introduction

With the introduction of nitrogen (N) in the area of carbon research, the scope of carbon has increased manifold. Nitrogen incorporation in carbon is reported by many researchers [1-8]. The motivation behind this attempt can be divided into two broad categories; synthesis of crystalline carbon nitride alloy (C_3N_4) and doping of carbon in order to convert undoped p-type carbon to n-type. The properties of crystalline phase of carbon nitride(CN) alloy was reported by Liu and Cohen [9-10]. They have proposed β-C_3N_4, a form of CN alloy analogous to β-Si_3N_4 , should have hardness closer to diamond. a super hard material. Carbon nitride already has shown considerable interest in the field of protective coating for magnetic and optical materials [11]. Recently Nitta et al [12] has reported photoconductivity from amorphous-CN_x films which is encouraging for its application in optoelectronic devices in the future.

However, undoped carbon is reported to be lightly p-type and doping is essential for the application of carbon in electronic devices. Veerasamy et al. reported n-type doping in carbon using phosphorus powder [13] and nitrogen gas [14]. Since nitrogen has smaller radius compared to phosphorus and is close to that of carbon, the former would be preferred. Further, the nitrogen, being gas phase has the advantage of better control of dopant concentration over phosphorus in physical deposition systems. The ability to dope using nitrogen gas has shown a new direction for the application of the carbonaceous material in electronic devices. At present, there are numerous reports of attempts to use nitrogen gas and ion as a doping source [14-18]. We have been working on carbon film obtained from camphoric carbon soot target which reveals better properties compared to carbon film obtained from conventional graphite target (Chap. 4). In previous chapter (Chap. 5), we have shown successful doping of phosphorus in camphoric carbon film. Our objective of present work is to investigate the effect of nitrogen gas in camphoric carbon film by using PLD.

In this chapter, we will present some optoelectrical and structural properties of the nitrogen incorporated film as a function of nitrogen partial pressure (NPP) in the PLD chamber. The deposited films are examined by Raman, optical absorption, resistivity and X-ray photoelectron spectroscopic investigations.

6.2 Experimental Details

Film deposition conditions are same as presented earlier. In brief, the camphoric carbon soot is used as a target material. To incorporate nitrogen in the film, we have introduced N_2 gas in the PLD chamber via leak valve. The pressure of the N_2 was varied between 10^{-4} to 5×10^{-1} Torr. Prior to insert of N_2, the chamber was evacuated till about 10^{-6} Torr. The films are deposited on p-Si and quartz substrates. Structural properties of the films are studied by Raman and X-Ray Photoelectron spectroscopy (XPS) while electrical and optical properties are investigated by conductivity and spectral transmittance/ reflectance measurements.

6.3 Results and Discussions

6.3.1 X-ray Photoelectron Spectroscopy

The presence of nitrogen in carbon films is detected by XPS spectra. Figure 6.1 shows the XPS spectra for the undoped carbon film and the film deposited at 5×10^{-1} Torr NPP (the spectrum of undoped film is vertically shifted for clarity). The peak at about 284 and 400 eV are due to photoelectrons excited from the carbon C1s and nitrogen N1s levels, respectively. Atomic % of N in the film is determined from XPS spectral measurements in these two regions.

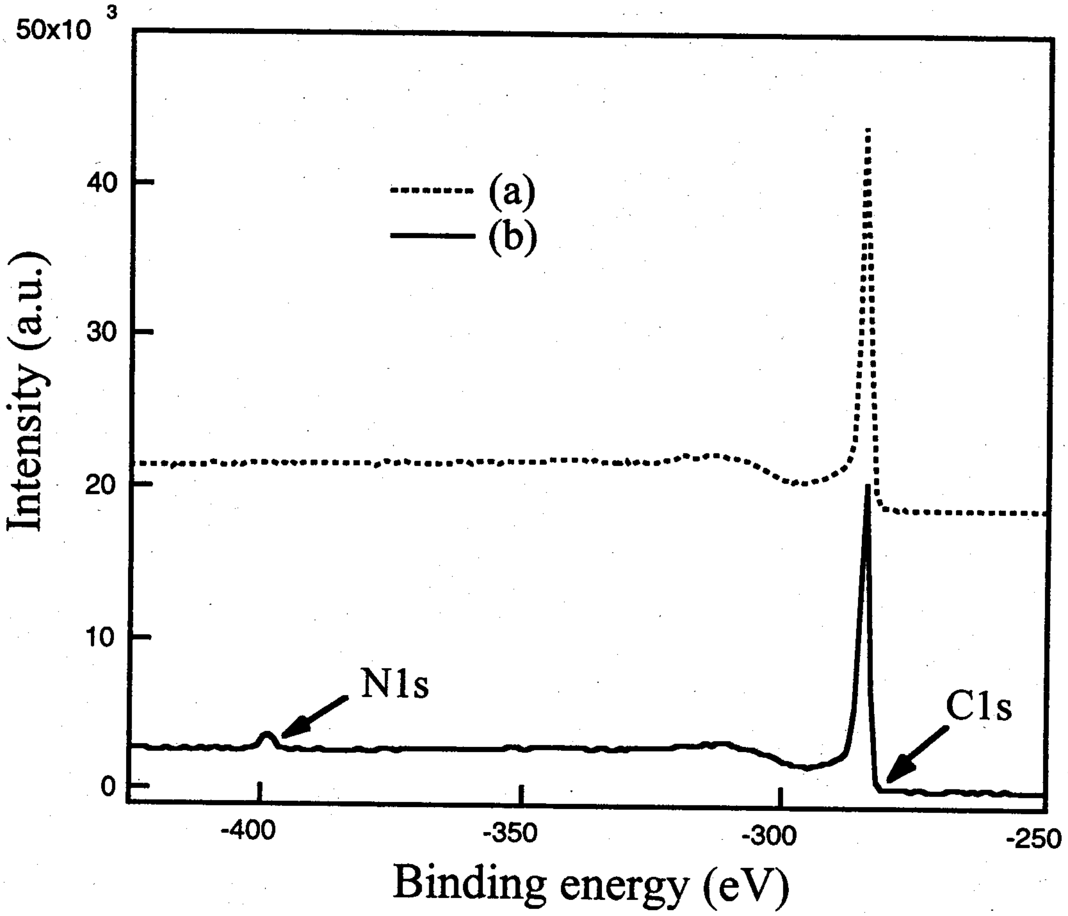

Figure 6.1. Core level X-ray Photoelectron spectra of Carbon (C1s) and nitrogen (N1s) for the a) undoped carbon film and b) the film deposited at 5 × 10^{-1} Torr.

6.3.2 Nitrogen Content in the Film

Figure 6.2 shows the variation of the atomic % of N in the carbon films as a function of nitrogen partial pressure (NPP) in the PLD chamber. N content in the film increased rapidly initially and gradually with NPP till 1 × 10^{-3} Torr. However, for higher NPP the N content is saturated. The N content is about 0.4% for the film deposited at 2.2 × 10^{-4} Torr and increases to about 1.4% for the film deposited at 1×10^{-3} Torr. With further increase of NPP, the N content increases to about 3.5 % for the film deposited at 1×10^{-2} Torr and saturates thereupon at about 3.5% in the film.

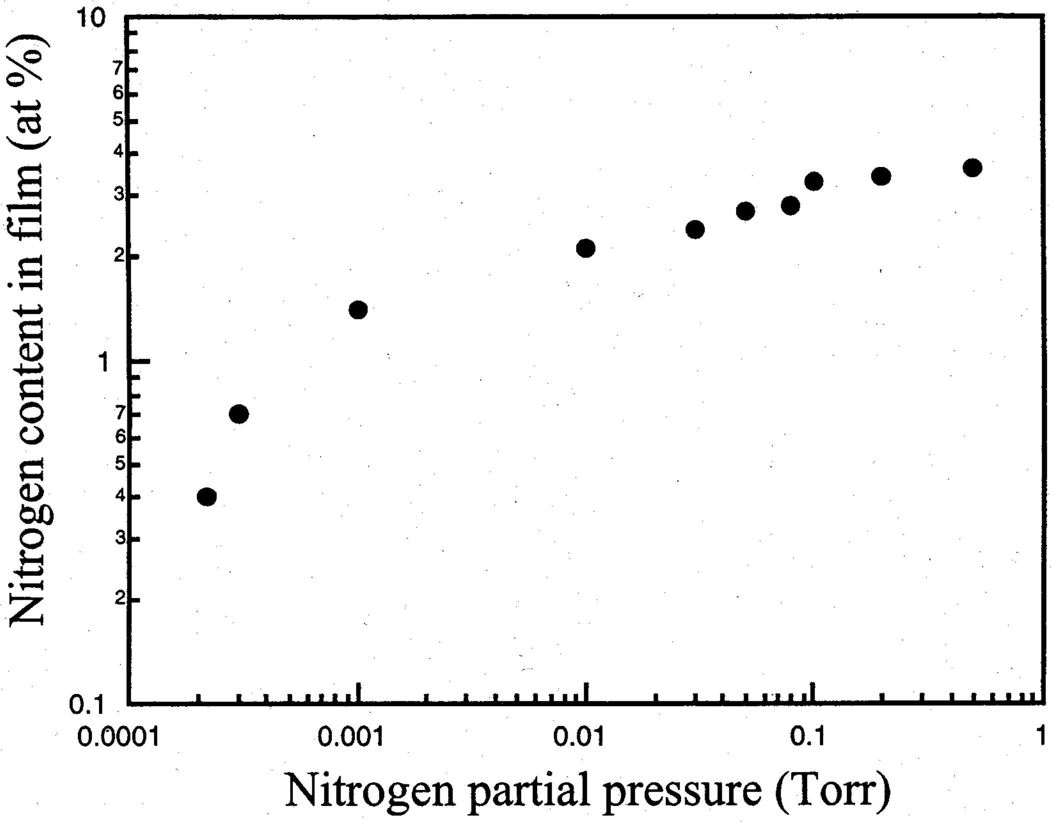

Figure 6.2. Nitrogen content (at %) in the film as a function of nitrogen gas partial pressure in the PLD chamber.

6.3.3 Optical Absorption

6.3.3.1 Transmittance and Reflectance spectroscopy

The optical absorption characteristic is obtained from spectral reflectance and transmittance measurements. Reflectance and transmittance spectra are shown in Fig. 6.3a and 6.3b respectively. The reflectance is decreased while transmittance is observed to increase with NPP. The optical gap is obtained from Tauc relationship [19]. The detailed procedure for the estimation of optical gap is explained in chapter 3 (See Sec. 3.3.1.1).

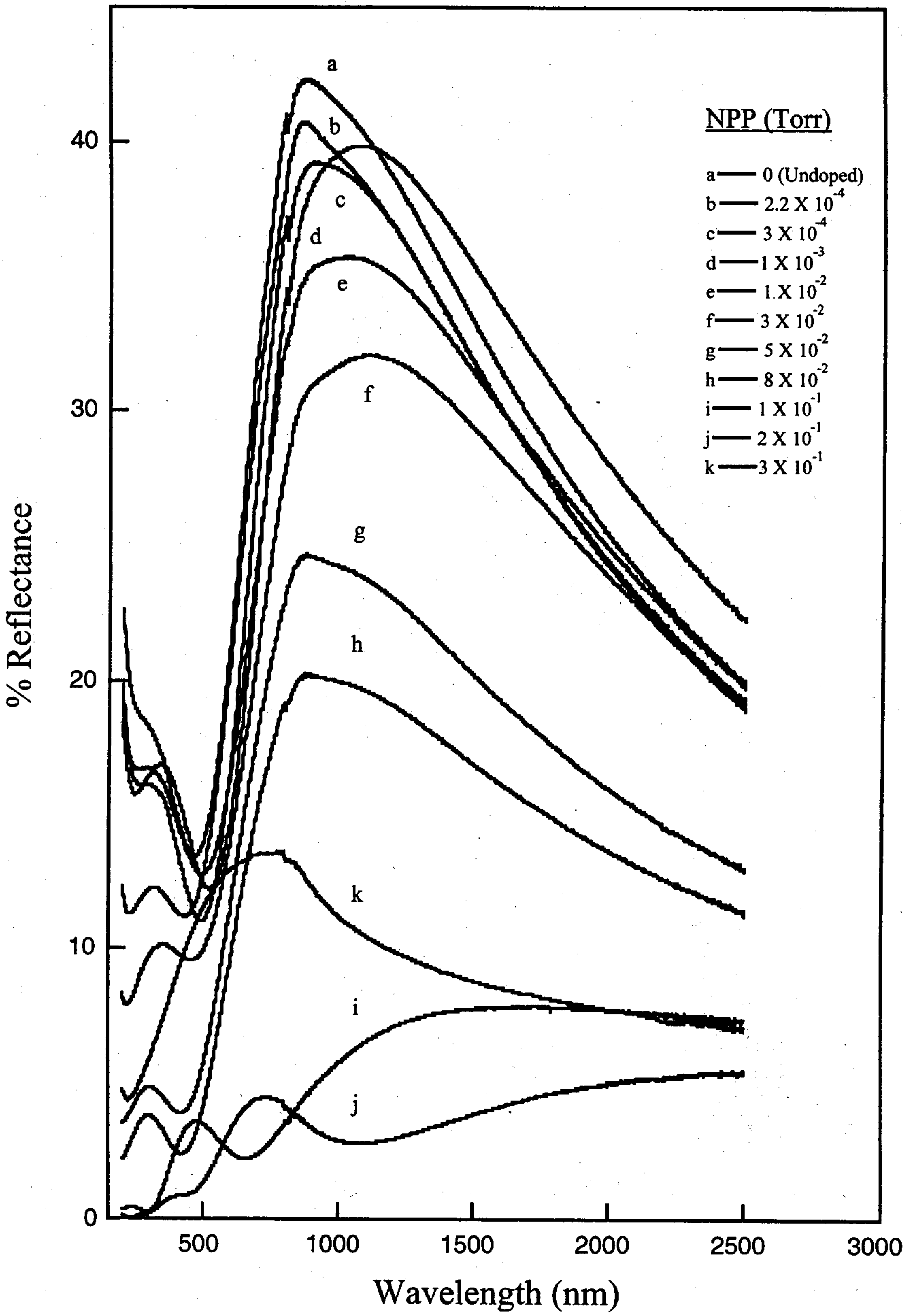

Figure 6.3a. Reflectance spectra of the undoped and N incorporated carbon films.

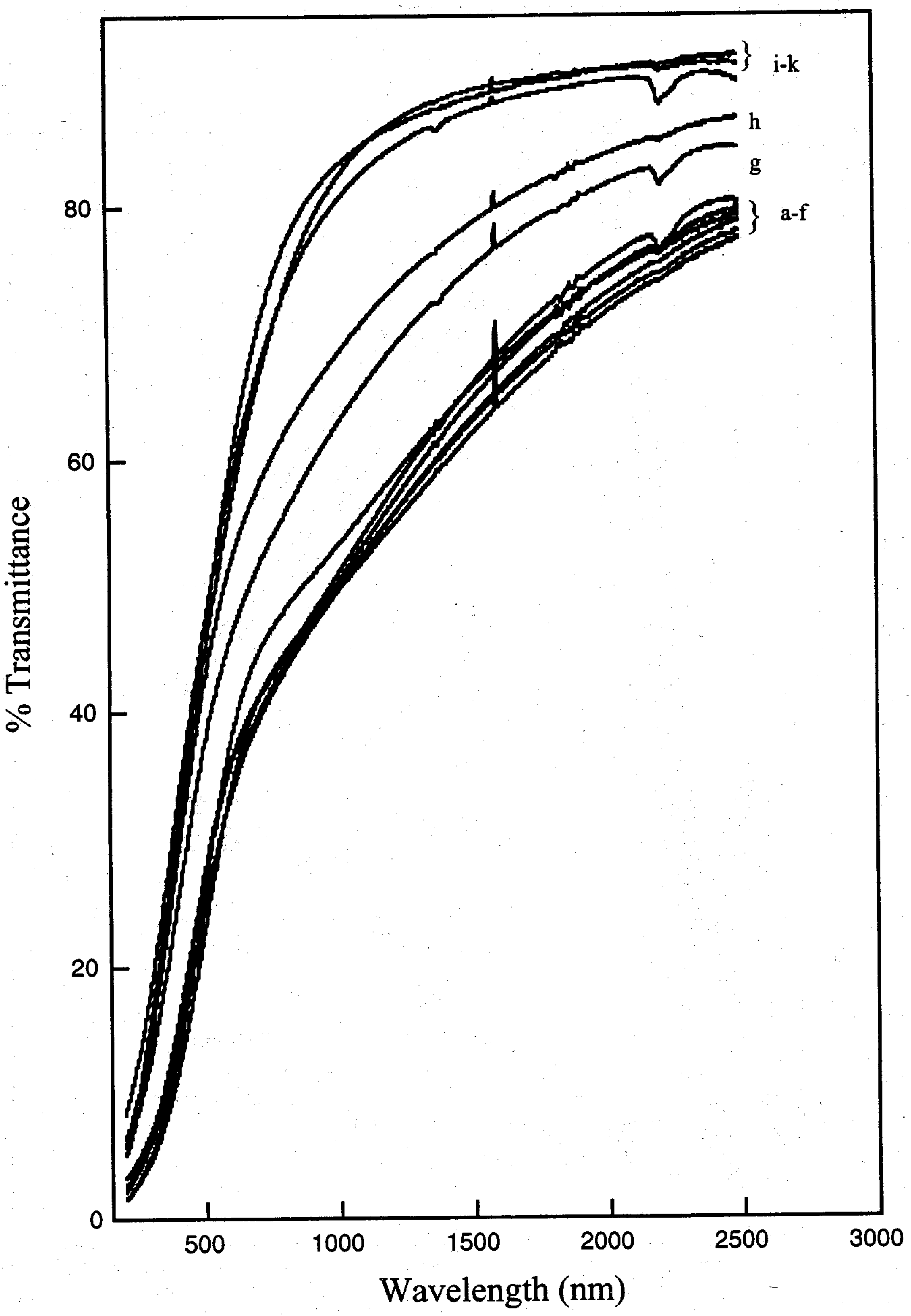

Figure 6.3b. Transmittance spectra of the carbon films in Fig. 6.3a.

6.3.3.2 Tauc Plot and Optical Gap

A plot of $(\alpha h\nu)^{1/2}$ versus hν is shown in Fig. 6.4 for the films deposited without nitrogen and Nitrogen partial pressure at about 3×10^{-4}, 3×10^{-2} and 5×10^{-1} Torr respectively.

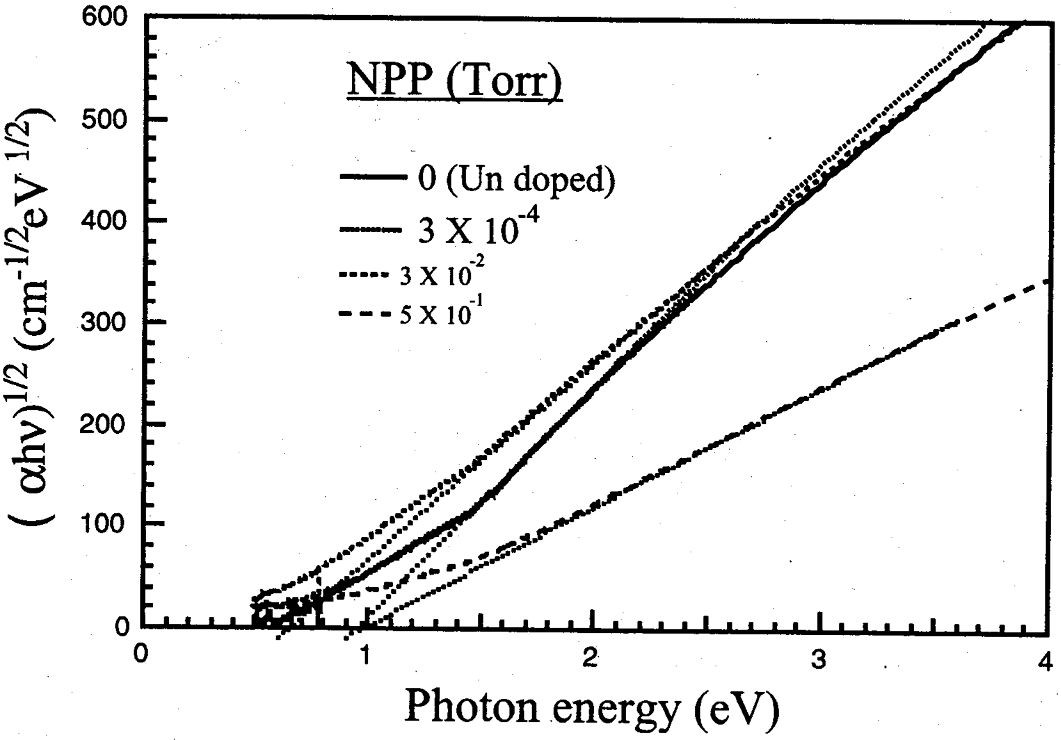

Figure 6.4. $(\alpha h\nu)^{1/2}$ as a function of hν, for the N incorporated carbon films.

Optical gap for the undoped film is about .95 eV. The optical gap remains unchanged for low nitrogen content and decreases to about 0.7 eV (Fig. 6.4c). With higher nitrogen content the optical gap increases. The optical gap for the film having amount of nitrogen in this work is about 1 eV and is higher than that of the undoped film. Fig. 6.5 shows the variation of the optical gap as a function of the NPP.

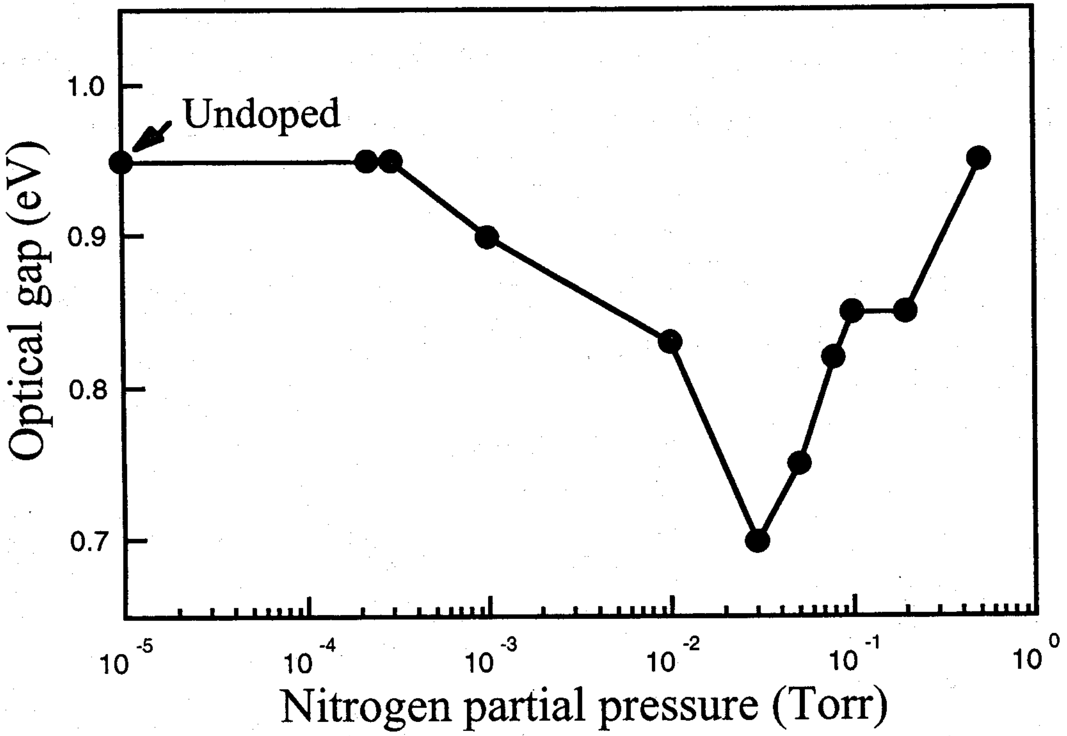

Figure 6.5. Optical (Tauc) gap as a function of nitrogen partial pressure.

6.3.4 Electrical Resistivity

The resistivity of the carbon films are measured by 4 -point probe technique-the usual way for high resistance measurement. The resistivity of the carbon film is observed to increase with N content initially and decreases with higher N content till for the film deposited at 3×10^{-2} Torr. We have observed similar trend for our P-doped films (See chap. 5) . Veerasamy et al [13] observed decrease of resistivity for their P and N doped films. However, the increase of resistivity is observed for the films deposited above 3×10^{-2} Torr nitrogen partial pressure. The variation of resistivity is shown in Fig. 6.6. We can relate this variation of resistivity to doping of nitrogen in our films for low content of nitrogen as the optical gap remain unchanged till for the film deposited at 1×10^{-2} Torr. Since both the optical gap and resistivity are decreased with higher N content, this phenomenaon can be related to graphitization as observed by other researchers [20-21].

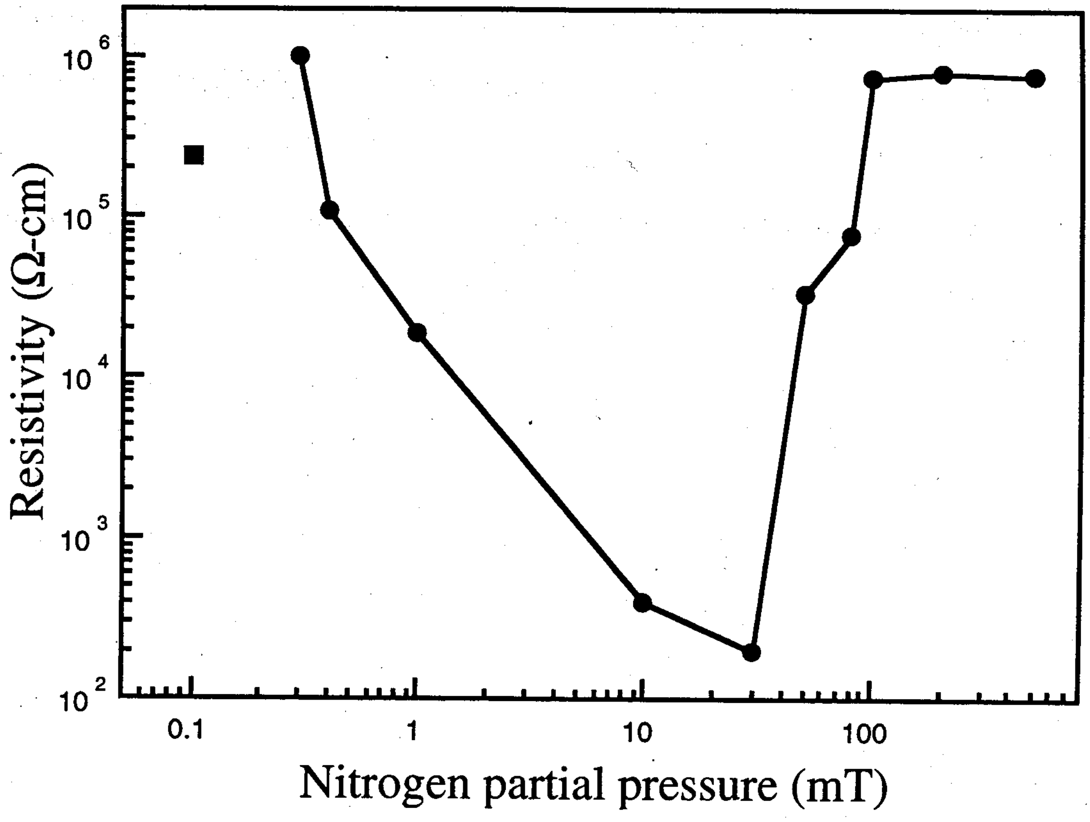

Figure 6.6. Room temperature resistivity as a function of nitrogen partial pressure.

6.3.5 C-N Alloy

However, the increase of optical gap and resistivity with more N content can be related to structural change in carbon film. Usually this kind of behavior is observed for high content of N in the carbon film , i. e. for the CN alloy [22-23]. This film might be promising for optical applications as photoconductivity is reported by Nitta et al for CN alloyed film [12]. More information can be obtained for these N incorporated films from the analysis of Raman and temperature dependence conductivity measurements and is under progress.

6.4 Conclusions

The effect of nitrogen incorporation in camphoric carbon thin film by pulsed laser deposition system is investigated. The variation of optical gap and resistivity show successful doping for the film deposited at low nitrogen partial pressure. With increase of N content in the film, the reduction in resistivity and optical gap is due to the graphitization of carbon film. With further nitrogen addition in carbon films results in increase of the optical gap and resistivity and are attributed to structural modifications through formation of some form of carbon nitrogen alloy (CN_x).

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Chapter 7

Photovoltaic Properties of Phosphorus Doped Camphoric Carbon/Silicon Cells

7.1 Introduction

Carbon-based heterostructures such as, metal insulator semiconductor (MIS) diodes [1], Schottky diodes [2], metal-insulator-semiconductor field effect transistor (MISFET) [3], heterojunction diodes [4-7] on silicon have already been reported and thereby demonstrates the potentiality of carbon materials in electronic devices. At present, silicon and compound semiconductor based devices are dominated in the solar cell technology. However, the cost of these cells is much high to reach for day to day life. The silicon has also drawback of using under illumination due to the degradation which limits its lifetime and stability.

However, undoped amorphous carbon (a-C) / amorphous hydrogenated carbon (a-C:H) are weakly p-type in nature [8] and the complex structure and presence of high density of defects restricts its ability to dope efficiently and is the main barrier for its application in various electronic devices.

Graphite is the most common solid target used in physical vapor deposition methods for the diamond-like carbon (DLC) films. We have been working on semiconducting carbon [9-15], obtained from camphor ($C_{10}H_{16}O$), a natural source. The starting precursor, camphor, has both sp^2 and sp^3 hybridized bonds while graphite has 100% sp^2 bonded structure.

In this chapter, carbonaceous films are deposited on Si (100) substrates by XeCl laser using camphoric carbon (CC) target. Some photovoltaic properties of this heterostructure is studied under illumination at 1 sun AM0 condition. Effect of phosphorus (P) content in the

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carbonaceous layer also investigated. For comparison, we have also deposited film from commercial graphite target and used as a carbonaceous layer in the mentioned heterostructure. In Chapter 5, we have studied some electrical, optical and structural properties of P-incorporated CC films on quartz substrates and observed successful doping of phosphorus in CC films.

7.2 Experimental Details

7.2.1 Device Fabrication

The undoped carbon film is reported to show p-type characteristics [13]. Therefore, we have deposited carbon films obtained from both CC and graphite targets on n-Si substrates. P incorporated carbon films are deposited on p-Si substrates. The resistivity of the substrates are about 5-10 Ω-cm. Gold (Au) electrodes of about 15nm is deposited on carbon film for the top contact and for the back contact, about 100nm antimony-gold electrode on n-Si and gold electrode on p-Si is deposited by conventional electron beam evaporation method. The contacts are shown to be ohmic. The schematic of the structure is shown in Fig.7.1.

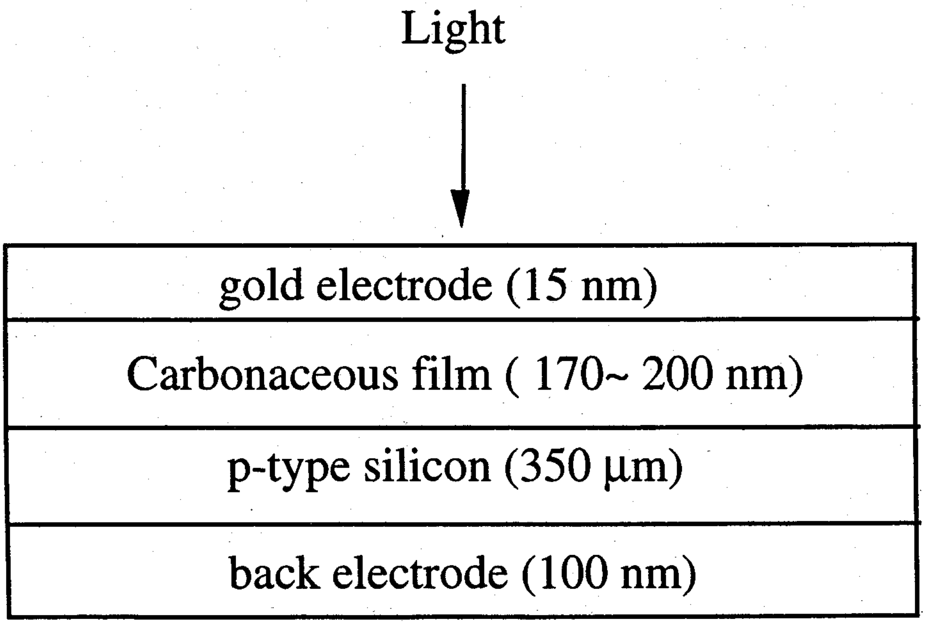

Figure 7.1 Schematic of the carbonaceous/Si photovoltaic cell.

7.2.2 Characterization

Junction Current-voltage (I-V) characteristics of dark and under illumination are measured by HP4145B of Hewlett-Packard. A Solar simulator is used to study photovoltaic properties and Xenon lamp is used as the light source to illuminate the junction at 1 sun AM0 condition.

7.3 Results and Discussions

7.3.1 Dark Current-Voltage Characteristics

Figure 7.2 shows I-V characteristics for the carbon / n-Si junction in the dark. The carbon layer is of undoped graphitic carbon (GC) and undoped camphoric carbon (UCC) in Fig. 7.2a and 7.2b, respectively. The dark I-V relationship can be expressed by Shockley equation [16]

$$I = I_0(e^{\frac{q(V-IR_s)}{nkT}} - 1) \tag{7.1}$$

here I_0 is the reverse saturation current, n is the ideality factor, R_s is the total series resistance in the n and p regions and other symbols have there usual meanings.

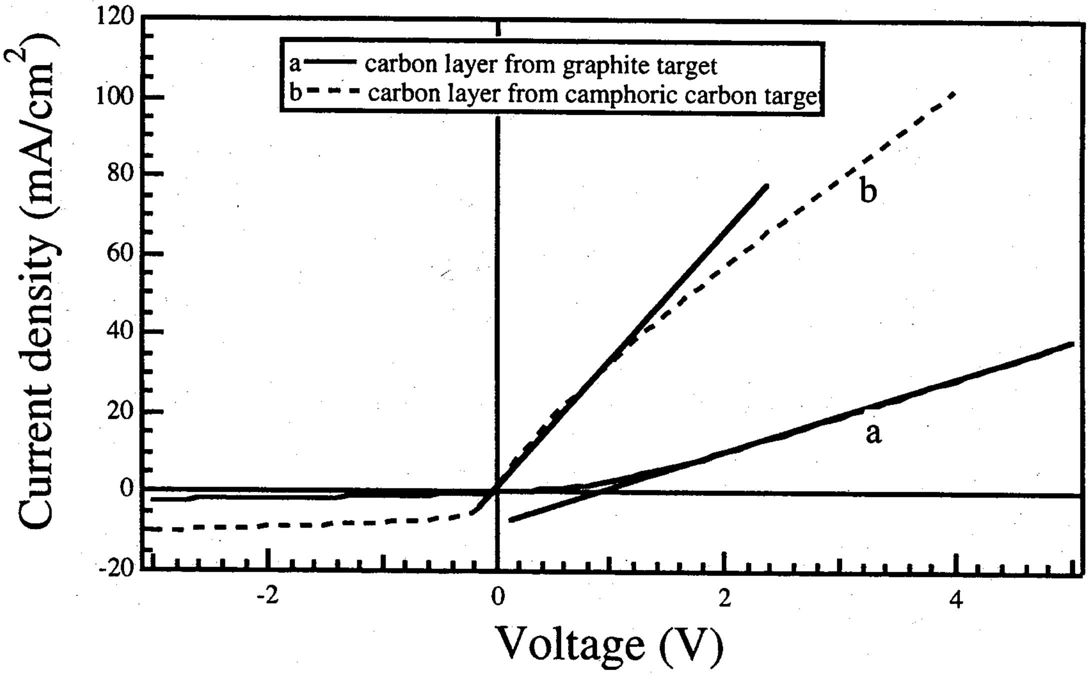

Figure 7.2. I-V characteristics of the C/Si junction with undoped carbon layers.

Both junctions reveal rectifying nature. However, the increase of current in the forward biased condition does not increase exponentially and the slope of the I-V curve indicate better quality of the UCC/n-Si junction. The deviation from the ideal characteristics can be due to comparable voltage drop in the undoped carbon layers. The I-V characteristics are improved remarkably with P incorporation in the carbon films (Fig. 7.3). The slope of the curves (semi-log scale) are increased with the P content in the carbon layer of the C/p-Si junction indicate improvement of the quality factor (n) with P-addition. The decrease of the deviation from the exponential region with P content indicates reduction of the series resistance loss with P content in the film. The reverse saturation current also reduced remarkably with P addition.

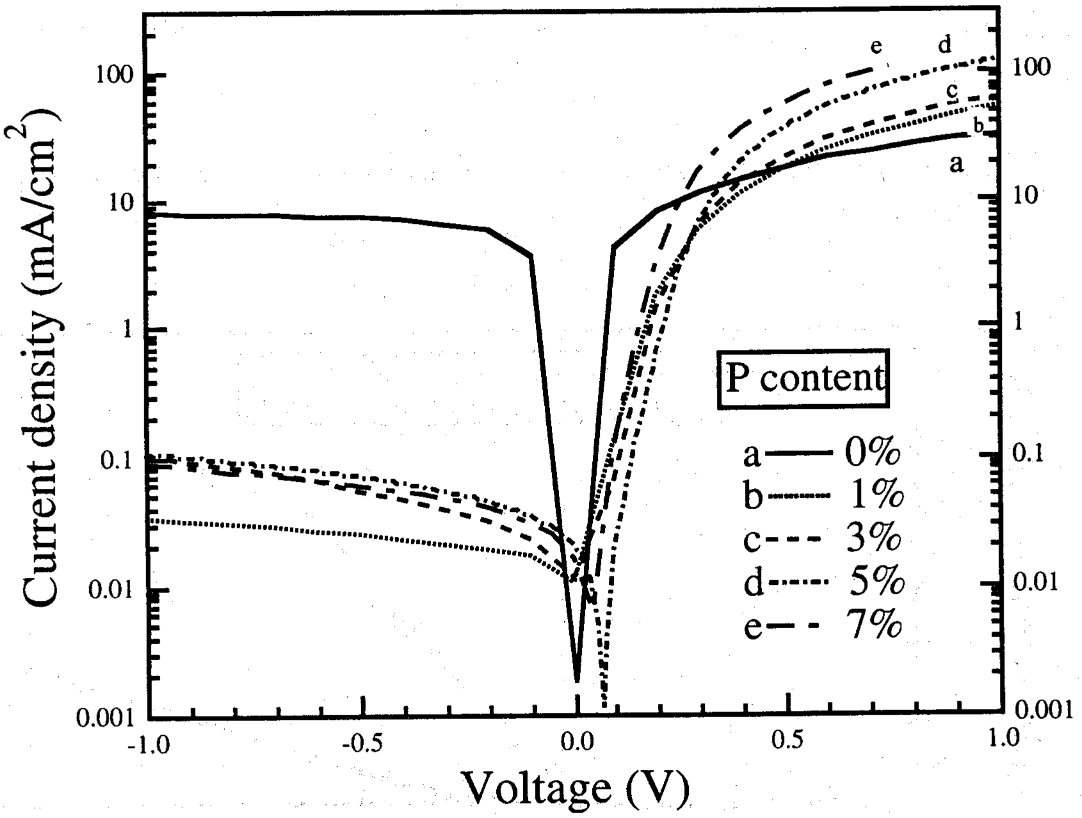

Figure 7.3. I-V characteristics of the C/Si junction with P doped carbon layers.

7.3.2 Photovoltaic Characteristics

The current-voltage characteristics of the cells under illumination at 1 sun AM0 condition is shown in figure 7.4. The photovoltaic characteristics are improved with the amount of P content up to 5% and deteriorate thereupon. The open circuit voltage (V_{oc}) and short circuit current density (J_{sc}) obtained from I-V characteristics are shown in Fig. 7.5. V_{oc} and J_{sc} vary from 0.22 to 0.27 V and 9 to 12 mA/cm^2, respectively. The cell with 5% P-doped shows highest efficiency (η) (1.25%) and fill factor (FF) (0.53%) (Fig. 7.6). In chapter 5, we have seen that with P incorporation, the spin density is decreased (Fig 5.6) and conductivity is increased (Fig 5.5) for the carbon films deposited from the targets containing up to 5% P. The improvement of the device characteristics including photovoltaic properties could be related to the successful doping and decrease of the defects in the gap with P addition. The decrease of efficiency and other photovoltaic characteristics might be due to the creation of gap states and decrease of the optical gap for higher amount of P content (above 5% P). Study of the junction characteristics is under progress.

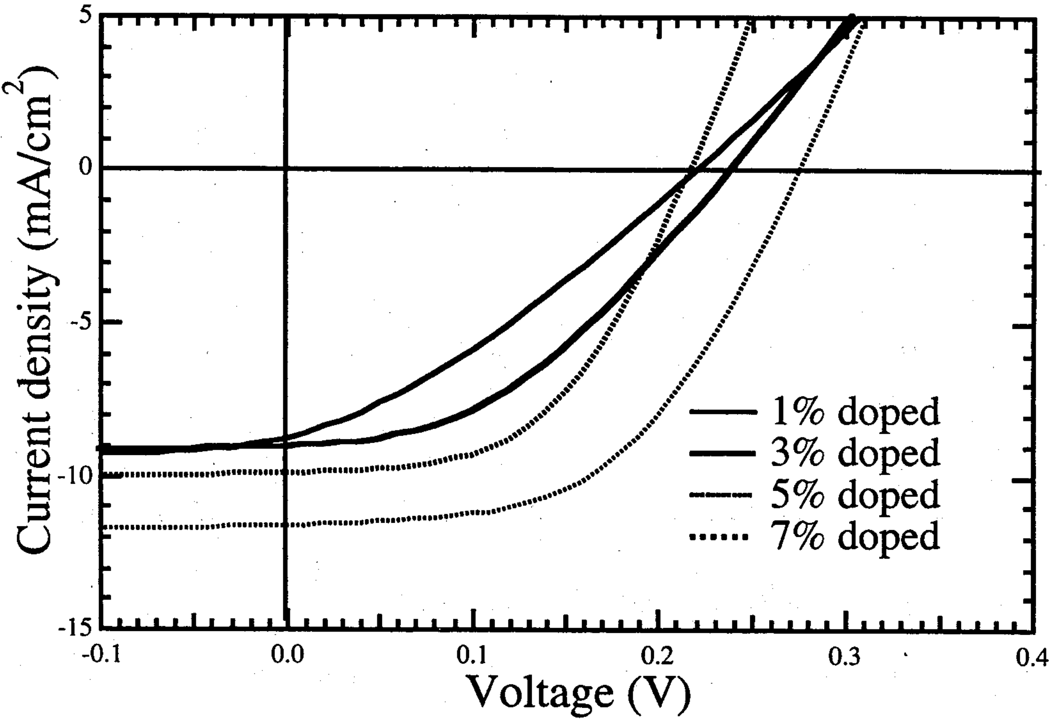

Figure 7.4. I-V characteristics of the C/Si cell under illumination with P doped carbon.

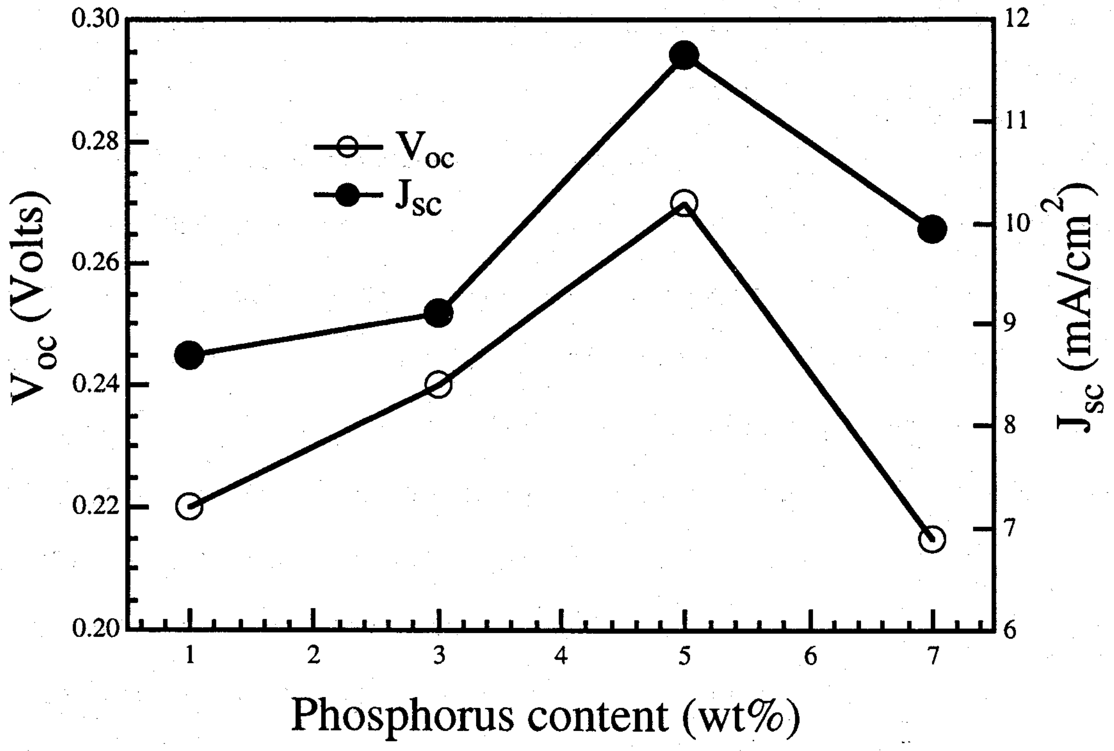

Figure 7.5. V_{oc} and J_{sc} with P content in the carbon layer used in the photovoltaic cell.

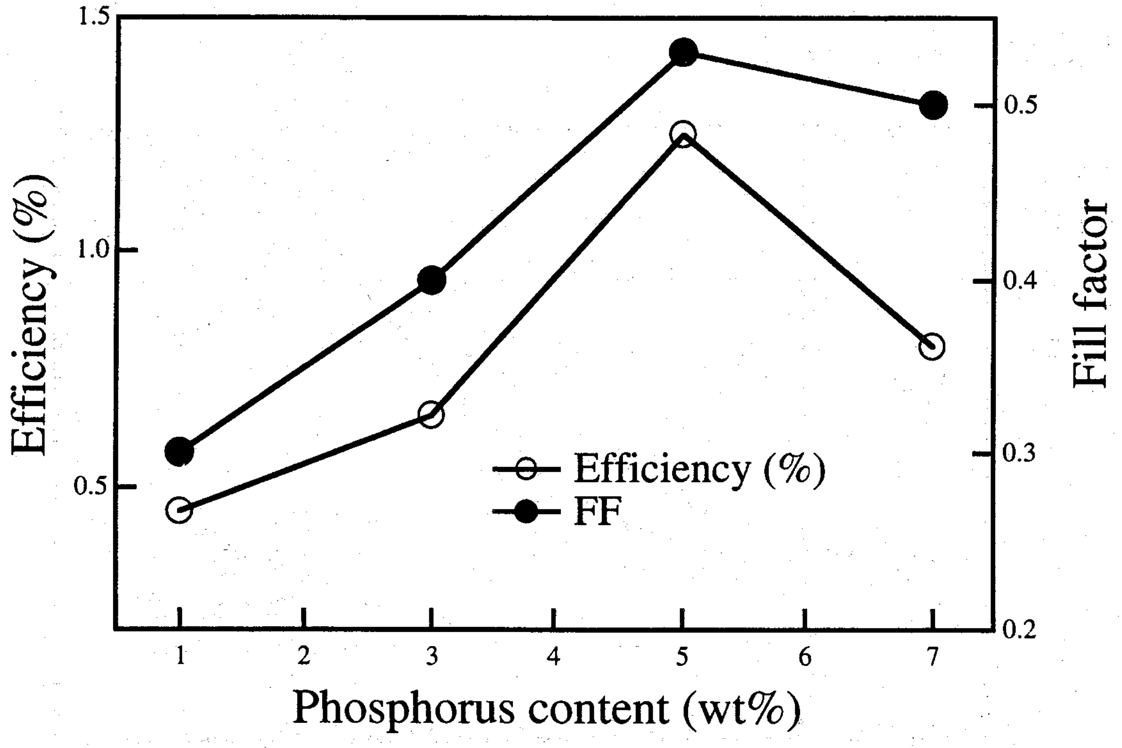

Figure 7.6. η and FF with P content in the carbon layer used in the photovoltaic cell.

7.4 Conclusions

Photovoltaic cell of P-doped CC/ p-Si heterostructure prepared by PLD using a natural precursor, camphor, is presented. The photovoltaic characteristics are improved with the amount of P content up to 5% and deteriorate thereupon. The open circuit voltage and short circuit current density obtained from I-V characteristics varies from 0.22 to 0.27 V and 9 to 12 mA/cm^2, respectively. The cell with 5% P-doped shows highest efficiency (η) (1.25%) and fill factor (FF) (0.53%). The results suggest the possibility of the low cost, non-toxic carbon based solar cells in the near future.

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Chapter 8

Summary and Future Scope

8.1 Summary

There is a long standing need for cheap and clean energy sources as the world population continues to increase with an increasing energy consumption per person. A solution to the problem is to tap the energy from sunlight. However, the solar cells fabricated to date are far too expensive to use on a commercial basis. With respect to present high efficient solar cell materials, carbon is a material of highly stable, cheap, non-toxic which can be obtained from precursors those are sufficiently available in nature. Present work is aimed at preparing and characterizing physical, electrical and optical properties of the thin carbonaceous films obtained from camphor by ion beam sputtering (IBS) and pulsed laser deposition (PLD) methods leading toward developing low cost solar cells based on low cost semiconducting material, Carbon, for common consumer application.

The properties of carbon films strongly depend on the precursor material and the method of their deposition. Graphite is being used almost exclusively as the target material for physical vapor deposition of DLC films systems, such as, sputtering, filtered cathodic vacuum arc (FCVA), PLD etc. Therefore, additional hydrogen gas/ ions are needed to get hydrogenated carbon where hydrogen is used to passivate the dangling bonds in the gap states and also to tailor the optoelectronic properties for a specific application. However, camphoric carbon (CC) soot obtained from burning camphor (($C_{10}H_{16}O$), a cheap, non-toxic and naturally available material might be a new precursor material and has hydrogen abundantly in its structure. Furthermore, the presence of sp^3-hybridized bonds in camphor molecule might play an beneficial role for the deposition of carbon films.

A special set up is made for preparing carbon soot from camphor. Thin films of camphoric carbon (CC) are deposited by ion beam sputtering (IBS) and pulsed laser deposition (PLD) methods. The optical and electrical properties have shown the film to be semiconducting in nature for both IBS and PLD films.

Effect of heat treatment in the range of 200 to 800℃ for the IBS deposited films is studied. As-deposited films have shown amorphous in nature. The optoelectrical and structural properties reveal the carbon films to be stable up to 400℃. The conduction mechanism for the films heat treated up to 400℃ is dominated by thermally activated hopping in the tail states in the high-temperature region ($400\ K \geqq T > 200\ K$), and variable-range hopping near the Fermi level in the low-temperature region ($200\ K \geqq T \geqq 40\ K$).The property of the IBS film is similar to the amorphous hydrogenated carbon (a-C:H) reveals presence of hydrogen in the carbon films. Structural change upon heat treatment by Raman spectra analyses help to understand the physics of the CC films.

In fact, the energy of the carbon species generated by various preparation methods is different and plays an important role in controlling the sp^3/sp^2 ratio. In the past decade, pulsed laser deposition (PLD) process has become popular for its ability to generate highly energetic carbon species which enhances the formation of high percentage of sp^3 bonded carbon atoms at low substrate temperatures, and therefore, deposition of high quality of DLC film can be realized. We have investigated the characteristics of DLC films prepared by PLD from compressed CC soot target and compared to that obtained from conventional graphite targets. The films deposited from CC target are more diamond-like. The optical gap of the carbon film obtained from CC target is about 0.9 eV, close to that of silicon whereas, the optical gap of carbon film obtained from graphite target is about 0.5eV. The ESR studies show lesser defects for the CC film compared to the graphitic carbon (GC) film. The high deposition rate along with the better structural and optoelectronic properties reveal its usefulness as an alternative target to graphite for PLD and demonstrates potentiality of CC film for the application in light conversion devices, such as, solar cells.

Unlike silicon, carbon has both σ and π states while silicon and other group IV semiconductors have only σ states. Since π states are more weakly bonded, they lie closer to the Fermi level (E_F) than the σ states. Therefore, filled π states form valence band edges while empty π^* states form conduction band edges, and these (π and π^*) states control the characteristics of gap states. Microstructure in carbon is complicated due to the presence of both σ and π states. However, undoped carbon is weakly p-type and presence of the high density of intrinsic defects restricts its successful doping which is the main barrier for its application in various electronic devices. Effective doping can modify electronic properties, specially gap states, conductivity, etc. in semiconductor materials. Attempts have been made to dope carbon films using various elements. However, P doping in relatively smaller gap DLC film with optical gap close to that of silicon which has scope in optoelectronic device applications, is not reported so far. P is the widely used n-type impurity in silicon and is a possible alternative to nitrogen (N) in carbon. Electronic doping in carbon films using P target is yet to be realized by PLD using conventional graphite target. Furthermore, our effort to get P doped carbon film by XeCl excimer laser using graphite target is unsuccessful. We have investigated the effect of P incorporation in DLC films using CC soot target by PLD technique. The structural, optical and electrical properties including defect states are studied. Raman G peak is not shifted with P addition in CC film. As sp^3/sp^2 ratio remain same, the bonding structure of the CC film is unaltered with P incorporation. Study of activation energy obtained from temperature dependence conductivity measurements, suggests that the Fermi level of the carbon film is moved from valence band edge to near to the conduction band edge through mid gap. The variation of conductivity is in good agreement with the activation energy analyses. As optical gap remains almost constant while conductivity increased by 2 orders, successful doping is realized for the films deposited from the target containing up to 5% P. The decrease of ESR spin density reveals modifications of the gap states with increase of P content.

Carbon nitride (CN) has already shown considerable interest in the field of protective coating for magnetic and optical materials. Recently Nitta et al. has reported photoconductivity from a-CN_x film and is encouraging for its application in optoelectronic devices. Since N has smaller radius compared to P and is close to that of carbon, the former would be preferred. Further, the N, being gas phase has the advantage of better control of dopant concentration over P in physical deposition systems. The effect of N incorporation in CC film by PLD system is investigated. The variation of optical gap and resistivity show successful doping for the film deposited at low nitrogen partial pressure (NPP). With increase of N content in the film, the reduction in resistivity and optical gap is due to the graphitization of carbon film. With further nitrogen addition in carbon films results in increase of the optical gap and resistivity and are attributed to structural modifications through formation of some form of carbon nitrogen alloy (CN_x).

Photovoltaic cell of P-doped CC/ p-Si heterostructure was prepared by PLD. Some photovoltaic properties of this structure are studied under illumination at 1 sun AM0 condition. Effect of P content in the carbonaceous layer is also investigated. The junction characteristics are improved with P addition up to 5% P in the target and deteriorate thereupon. The open circuit voltage and short circuit current density obtained from I-V characteristics varies from 0.22 to 0.27 V and 9 to 12 mA/cm^2, respectively. The cell with 5% P-doped shows highest efficiency (η) (1.25%) and fill factor (FF) (0.53%). The results are the signature of the low cost, non-toxic carbon based solar cells in the near future.

8.2 Future Scope

We are succeeded in doping camphoric carbon film using phosphorus and nitrogen and obtained n-type carbon. However, electronic boron doping is not realized so far. Other dopants can be tried in order to get good p-type carbon film. Fabrication of n-C/p-C devices from carbon films can be realized with successful p-doping which can be the future scope of this work.