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Structural, Optical and Electrical Properties of Chemical Vapour Deposited Nitrogen Doped Diamond Like Carbon Films

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Authors' contributions

This work was carried out in collaboration between both authors. Author S. Kundoo designed the study, wrote the manuscript. Author S. Kar managed the literature searches and performed Fourier-transform infrared spectroscopy analysis. All other works were done by author S. Kundoo. Both authors read and approved the final manuscript.

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ABSTRACT

Aim: Deposition of undoped and nitrogen doped amorphous diamond like carbon films and investigation of improved electrical, optical, field emission as well as structural properties of the films due to nitrogen doping.
Materials and Methods: Nitrogen doped diamond like carbon (N-DLC) films were deposited on silicon and glass substrates by using direct current plasma enhanced chemical vapour deposition (PECVD) process. Precursor gases used were acetylene and nitrogen. The films were characterized by different techniques such as XRD, FTIR, UV-Vis-NIR spectroscopy. Also electrical conductivities and field emission parameters of the films were measured.
Results and Discussion: XRD spectra showed the amorphous nature of the deposited films. Fourier-transform infrared spectroscopy (FTIR) measurements showed different vibrational modes of tetrahedrally bonded carbon present in the DLC films. With the incorporation of nitrogen into the DLC matrix different CN absorption bands appeared in the FTIR spectra. From FTIR spectra variation of sp^3/sp^2 ratios in the films with nitrogen concentration in the plasma were measured. The

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results showed that sp^3 fraction decreased with increase in nitrogen concentration. N-DLC films showed higher room temperature conductivity and better field emission properties. Tauc gap of the films was decreased with the increase in nitrogen percentage in the plasma, as with the increase of nitrogen content in the DLC films sp^2 carbon content was increased, which is consistent with the FTIR results. Nitrogen doped films showed higher emission currents at lower turn-on fields.

Conclusions: FTIR measurements showed various C–H bonding vibrations and different CN absorption bands present in the spectra of N-DLC films, thus confirmed incorporation of nitrogen into DLC matrix. Highest $sp^3/(sp^2+sp^3)$ ~ 85.65% was achieved in the undoped DLC film. N-DLC films showed two orders of magnitude more room temperature conductivity than undoped ones. The lowest turn-on field achieved was 6.56 volt/μm for current density of 0.5 $\mu A/cm^2$.

Keywords: Diamond like carbon films; nitrogen doping; infrared spectroscopy; electrical conductivity; Tauc gap; field emission.

1. INTRODUCTION

During the last two decades diamond like carbon (DLC) films received much attention due to their high potential in various technological applications, in electronic and photonic devices [1-3] and in biomedical applications [4]. These films have number of useful properties such as high young's modulus, high dielectric strength, chemical stability, high thermal conductivity, optical transparency in visible and infrared region, tunable optical (Tauc) gap by adjusting the sp^3 and sp^2 bonding ratio and high room temperature photoluminescence efficiency. In general the diamond like carbon material consists of tetrahedrally bonded carbon i.e. the sp^3 component and the trigonally bonded carbon i.e. the sp^2 part. The properties of the films depend on the ratio of these two components whose amount further depends on the deposition conditions. Increase in sp^2 component decreases the diamond like properties and the films richer in sp^3 component are hard and suitable for tribological applications. DLC films are in general deposited by a number of different techniques among which plasma enhanced chemical vapour deposition (PECVD) is the most convincing method. Inspite of the most interesting properties of the DLC films, there is some serious problems with them. It is found that DLC films are always formed with high amount of intrinsic stress. The level of intrinsic stress will affect the mechanical resistance and integrity of the deposited materials i.e. brittleness, adherence of the films with the substrates etc. Often it is found that the films peel off from the substrate. This also affects the electrical and optical properties of the films seriously. It is known [5] that introduction of a foreign element (e.g. nitrogen) into the DLC lattice structure may alter its local bonding pattern and may also change the co-ordination number. In DLC nitrogen does not necessarily enter into a four-fold coordinated site. Nitrogen may form terminated site in a-C:N:H network. It is expected that successful introduction of nitrogen in DLC lattice may reduce the stress. Introduction of nitrogen was found effective in reducing the stress with improved adhesion of the film on the substrate [5]. In the recent years diamond like carbon and chemical vapour deposited diamond films have received much interest due to their unusual field emission properties, as they possess negative or low electron affinity [6]. The emission depends on variable factors, such as electron affinity, band gap, surface termination, nitrogen addition, depletion layers and film thickness [7,8]. Researchers have found that nitrogen incorporation enhanced the electron emission from diamond like carbon films at lower turn-on fields [9-11].

In this work diamond like carbon films were deposited by plasma enhanced chemical vapour deposition (PECVD) process. The aim of the work was to reduce the intrinsic stress in the deposited DLC films so that the films could not be delaminated easily from the substrates and to improve optical, electrical properties, to get higher field emission at lower turn-on fields from DLC films by incorporating nitrogen into DLC, so that the material can be used for device applications. The effect of nitrogen incorporation on the sp^3/sp^2 ratio in the film, optical properties, electrical conductivity and electron field emission properties have been studied in detail.

2. MATERIALS AND METHODS

DC plasma CVD system equipped with various vacuum components was used to deposit undoped and nitrogen doped diamond like carbon films. The distance between the anode and the cathode was ~ 2.5 cm. Films were deposited on single crystalline silicon (400) substrates and also on glass substrates (2.5 cm x 2.5 cm). At first the substrates were cleaned thoroughly. For cleaning the Si substrates, they were dipped in HF (20%) solution for 5 minutes and then were cleaned in acetone in an ultrasonic bath. The glass substrates were cleaned at first in mild soap solution, then using deionised water and after that they were placed in boiling water for 15 minutes. Finally they were cleaned in acetone in an ultrasonic cleaner for 15 minutes. In the CVD unit the upper electrode (6 cm diameter) was connected to the negative of a D.C. power supply (0-3 kV) and the substrates were placed on the lower grounded electrode (12.5 cm diameter). Negative bias could be applied to the substrate with the help of a ring electrode placed midway between the anode and cathode electrode and near to the lower electrode. The chamber was evacuated by using standard rotary and diffusion pump arrangement. The base pressure was 10^{-6} Torr. The desired gasses (C_2H_2 and N_2) were introduced and the plasma was ignited. The deposition pressure was 0.3 Torr. The voltage and current maintained were 1.5 kV and 1.5 mA/cm^2. The substrate temperature was measured by a pre-calibrated chromel-alumel thermocouple placed behind the substrate. No intentional heating of the substrate was done and it was found that due to plasma heating the temperature was raised to 50 ^{o}C. The deposition time was normally 1 hour.

The films were characterized by different techniques, such as X-ray diffraction, Fourier transform infrared (FTIR) spectroscopy, ultra violet-visible-near infrared (UV-Vis-NIR) spectrophotometry and electrical properties along with the field emission studies. The films deposited both on glass and Si(400) substrates were characterized by X-ray diffraction using a diffractometer (Bruker, Advance D8) by Cu K_α line operating at 40 kV and 30 mA. For finding various local bonding information, FTIR spectra for both undoped and nitrogen doped DLC films deposited on Si(400) substrates were recorded in a Nicolet Magna – 750 spectrophotometer. The optical properties of the films deposited on glass substrates were studied by recording transmittance spectra using a spectrophotometer (Hitachi - U3410) in the UV-Vis-NIR region for both undoped and N-doped films by subtracting the transmittance of the glass substrates taking as reference. The temperature (T) dependence of electrical conductivity (σ) of the undoped and N-doped DLC films deposited on glass substrates was measured by four-probe technique using a Keithley electrometer (Model – 6514) and a constant current source. The electrical contacts were made by using conducting silver paint with the DLC films. The ohomic nature of the contact with the film was tested at first. Field emission measurements were carried out by using a parallel plate electrode configuration with the sample deposited on Si (400) substrates mounted on the cathode and a stainless steel anode having an area of 0.07 cm^2. The anode was attached to a micropositioner for varying the cathode-anode spacing with an accuracy of ±10 μm. Measurements were performed in a high vacuum of 10^{-7} mbar.

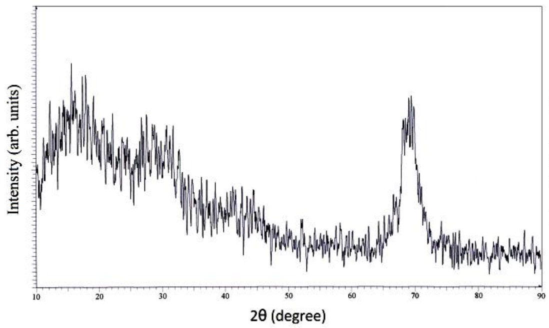
3. RESULTS AND DISCUSSIONS

3.1 Xray Diffraction Studies

Fig. 1(a) shows the XRD spectrum of a representative DLC film deposited on Si(400) substrate. The peaks coming in the spectrum are due to the substrate and no peak from the DLC film was originated. This indicated that the films were amorphous in nature. Fig. 1(b) shows the XRD spectrum of a typical DLC film deposited on glass substrate. It only shows a hump in the 2θ range ~ 23 degree originating from the glass substrate. Again as no other peaks were originated, hence the DLC coating was amorphous.

3.2 Fourier Transform Infrared Spectroscopic (FTIR) Measurements

Fig. 2(a) shows the FTIR spectrum of a typical undoped DLC film deposited on Si substrate. Table 1 shows different vibrational wave numbers appeared in the spectrum and their assignments to various C–H bonding. Fig. 2(b) shows the FTIR spectrum of DLC film deposited with 11 volume percentage of nitrogen in the plasma. It has been found that with the increase of nitrogen content in the plasma, intensity of the N-H absorption band around 3350 cm^{-1} was increased. Similar observation was reported by other workers [12]. Also various CN stretching vibrational bands appeared in the range from 1100 – 1600 cm^{-1} due to incorporation of nitrogen in the DLC films.



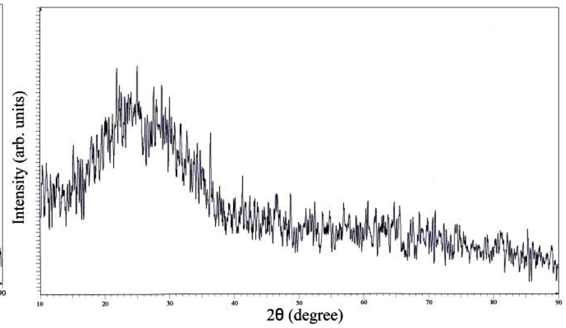
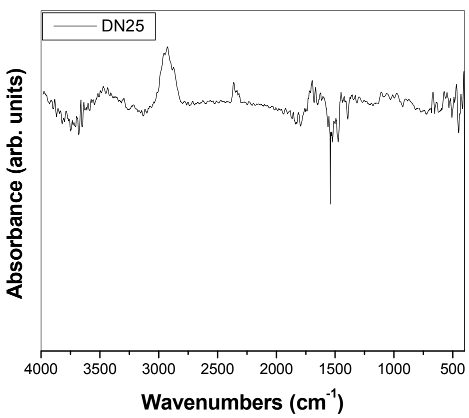


Fig. 1(a) **Fig. 1(b)**

Fig. 1(a). XRD spectrum of a representative DLC film on Si (400) substrate and Fig. 1(b). XRD spectrum of a representative DLC film on glass substrate



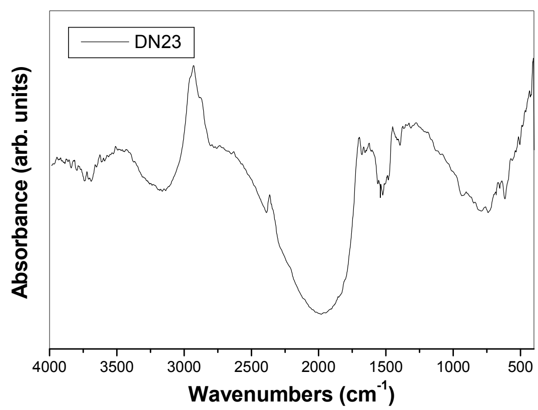


Fig. 2(a). FTIR spectrum of undoped DLC and Fig. 2(b). FTIR spectrum of N-DLC for 11 vol% N_2

Table 1. Assignment of different C – H vibrational modes of a representative DLC film

Wave numbers (cm^{-1})	Vibrational modes
2854	sp^3-CH_2 (symm)
2873	sp^3-CH_3
2902	sp^3-CH stretch
2927	sp^3-CH_2 (asymm)
2957	sp^3-CH_3 (asymm)
2987	sp^3-CH_3
3009	sp^2-CH (symm)
3022	sp^2-CH (asymm)

To find the sp^3/sp^2 ratio in the deposited films, the spectra were deconvoluted into a number of Gaussian peaks by using a computer program. It has been popular to fit C-H bands with Gaussian peaks to derive sp^3 fractions. The bands can be broad and the centers can drift, so the decomposition of the C-H stretching modes into the individual bands is not unique [13]. Fig. 3(a) shows the deconvoluted spectra for the corresponding FTIR spectrum shown in Fig. 2(a), for the undoped film deposited with zero bias. Fig. 3(a) yields the $sp^3/(sp^2+sp^3)$ ~ 85.65 %. But this undoped DLC film was delaminated easily from the substrate. This is because the film possesses large amount of intrinsic stress. After nitrogen incorporation stability of the films was improved. Similarly, sp^3/sp^2 ratios were determined for N-DLC films at different nitrogen percentages. Fig. 3(b) shows the deconvoluted spectra for the corresponding FTIR spectrum shown in Fig. 2(b), for nitrogen doped film. Fig. 4 shows the variation of sp^3/sp^2 ratio with nitrogen concentration in the plasma. It was found that sp^3 fraction was decreased with the increase of nitrogen content in the plasma. This observation is supported by other workers [14] also. This is may be due to the fact that as the nitrogen content in the plasma and in the films was increased, more sp^2 sites were generated. At very high nitrogen content in the plasma graphitization of the deposited films takes place.

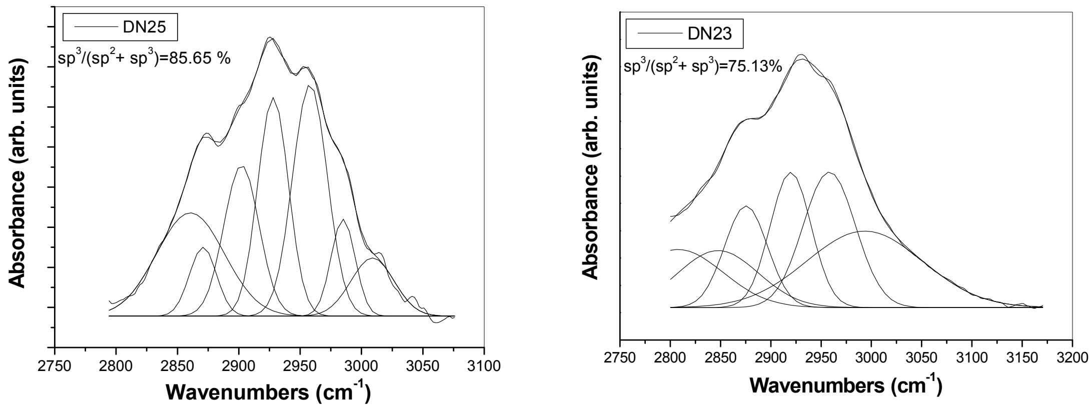


Fig. 3 (a). Deconvoluted C-H bands of undoped DLC and Fig. 3 (b). Deconvoluted C-H bands of N-DLC for 11 vol% N_2

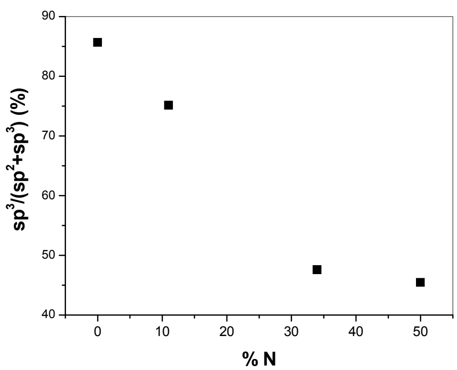


Fig. 4. Variation of sp^3/sp^2 ratio with nitrogen percentage in the plasma

3.3 Optical Properties

Fig. 5 shows the UV-Visible-NIR spectrum of undoped DLC film deposited on glass substrate with zero bias. The optical transmission spectra of the undoped and nitrogen doped films with different volume percentages showed high transparency in visible and NIR region. The spectra can be divided into two distinct regions, one is beyond absorption edge and the other is the region of strong absorption. The thickness values of the films were determined from fringe pattern appearing in the beyond absorption edge of the spectra using Manifacier model [15,16] and also from SEM cross-sectional images (not shown here). The thickness values lie in the range form 0.8 - 1.2 μm. From transmittance data absorption co-efficients (α) have been calculated in the region of strong absorption. As the films were amorphous in nature as evident from XRD spectra, Tauc relation [17] of absorption co-efficients (α) and the incident photon energy ($h\nu$) can be written as follows

$$(\alpha h\nu)^{1/2} = A\,(h\nu - E_g) \qquad (1)$$

where A is a constant, E_g is the Tauc gap of the material.

The $(\alpha h\nu)^{1/2}$ vs. $h\nu$ plots of undoped DLC and N-DLC films are shown in the Fig. 6. Extrapolating the linear portions of the plots to the $h\nu$ axis the Tauc gap of the films were determined for undoped and N-doped DLC films. Fig. 7 shows the variation of Tauc gap with nitrogen percentages in the plasma. It can be inferred that the nitrogen incorporation decreased the Tauc optical gap of DLC films. This decrease in E_g value is due to the increased sp^2 fraction in the films induced by addition of nitrogen. Other workers [18] have also observed the decrease in Tauc gap values with the incorporation of nitrogen.

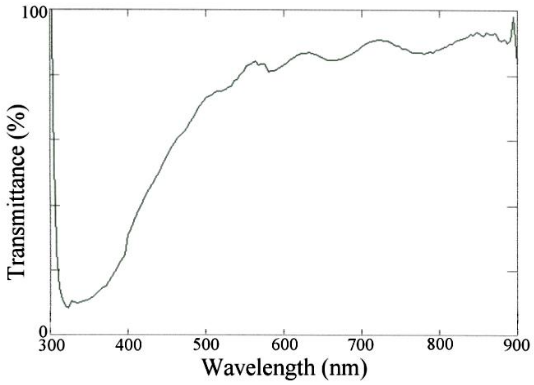


Fig. 5. Transmittance (T) vs. wavelength (λ) DLC plot for undoped DLC film

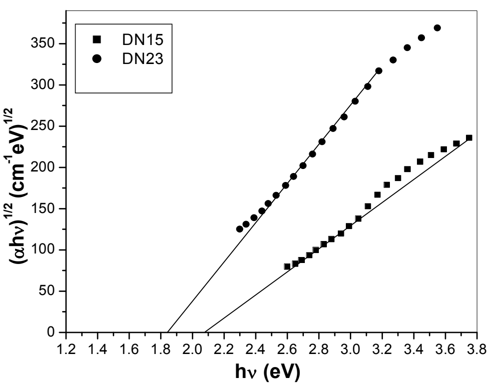


Fig. 6. $(\alpha h\nu)^{1/2}$ vs. hν plots for undoped and N-DLC films (DN15, N_2-0 vol.%; DN23, N_2-11 vol.%)

3.4 Electrical Conductivity Study

Fig. 8 shows the lnσ vs. 1/T plots for undoped DLC film and for the films deposited with various nitrogen contents in the plasma. The plots indicated two linear regions with different slopes. In the lower temperature region (below 150°C) conduction mechanism is attributed to localized band tail states and in the higher temperature region (above 150ºC) thermally activated conduction predominates. With addition of nitrogen the room temperature conductivity increased initially upto 11 vol.% of nitrogen by two orders of magnitude. After that, with further addition of nitrogen in the plasma no noticeable change in the room temperature conductivity was observed. It did not show any monotonic increment with addition of nitrogen in the plasma. Increase in electrical conductivity due to nitrogen doping upto certain amount was observed by other workers also [19]. The increase in conductivity with the addition nitrogen may be correlated with the FTIR data, which indicated decrease of sp^3/sp^2 ratio with the addition of nitrogen in the plasma.

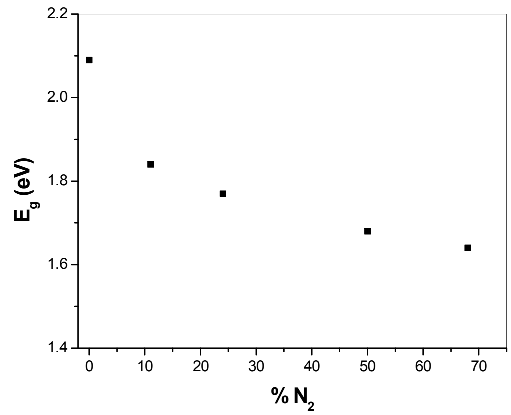


Fig. 7. Variation of Tauc gap (E_g) of DLC films with N_2 volume percentage

3.5 Field Emission Studies

Fig. 9 shows the emission current density (J) plotted against the applied field (E) for undoped and nitrogen doped DLC films. It can be observed that upon incorporation of nitrogen in the plasma during deposition, the turn-on field (E_c) was greatly decreased in the nitrogen doped DLC films as compared with the undoped film. The turn-on field (E_c) was defined as the electric field needed to get a current density of 0.5 μA/cm². The electric field was computed as E_c = applied voltage/cathode-anode spacing. The lowest turn-on field achieved was ~ 6.56 volt/μm for the film with the amount of nitrogen 50 vol.% in the plasma, whereas for the undoped film no turn-on was found in our range of measurement.

Field emission characteristics of the films were studied using the Fowler-Nordheim (F-N) theory [20]. A simplified F-N equation for the local current density J (amp/cm²) at some point on the emitting surface may be written as [21,22]

$$J = S\frac{1.54\times10^{2}F^{2}}{\phi}\exp\left[\frac{-6.83\times10^{3}\phi^{3/2}}{F}\right] \quad (2)$$

Where F is the local field and ϕ is the local work function (expressed in eV), which is called the emission barrier. F is not simply V/d, which is the macroscopic field obtained with an applied voltage of V between two electrodes separated by distance d. However, in most cases, $F = \beta E$, where $E = V/d$ and β is the geometrical enhancement factor, S is the fraction of area emitting electrons. E is in volt/μm.

The F-N plots for undoped and different amount of nitrogen doped DLC films are shown in Fig. 10(a). The linearity of these F-N plots in the high field region (> 4 volt/μm) (Fig. 10(b)) suggests the field emission from these films. Assuming a plane flat emitter with $\beta = 1$, emission barriers (ϕ) were calculated from the slopes of F-N plots. The value of ϕ was reduced from 0.08 eV to 0.04 eV by nitrogen doping. But the true barrier must be larger than these values. Such a low work function obtained may be due to an underestimation of the field enhancement factor β.

The above results obtained by different characterization techniques can be correlated with each other. From FTIR analysis it was found that sp^2 fraction increased in the films with the increase of nitrogen content in the plasma in the present measurement range. As sp^2 fraction increased in the films, the Tauc gap of the material was decreased with the incorporation of nitrogen. For more sp^2 sites generated in the films, electrical conductivity was also increased with the addition of nitrogen. Maximum electrical conductivity was obtained for 50 vol. % of nitrogen in the plasma. For more sp^2 contents higher emission currents were also obtained from nitrogen doped DLC films at lower turn-on fields. Lowest turn-on field was achieved with the amount of nitrogen 50 vol. % in the plasma. Therefore it can be concluded that in this work optimum nitrogen percentage was 50 vol. % to get better quality films deposited by PECVD method.

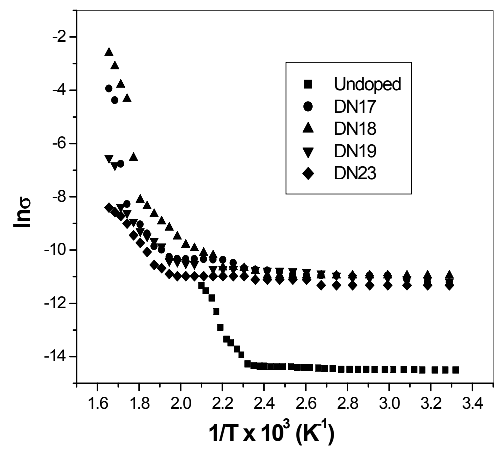


Fig. 8. ln σ vs. 1/T plots for undoped and different nitrogen doped DLC films (DN17, N_2-68%; DN18, N_2-50 %; DN 19, N_2-34 %; DN23, N_2-11%)

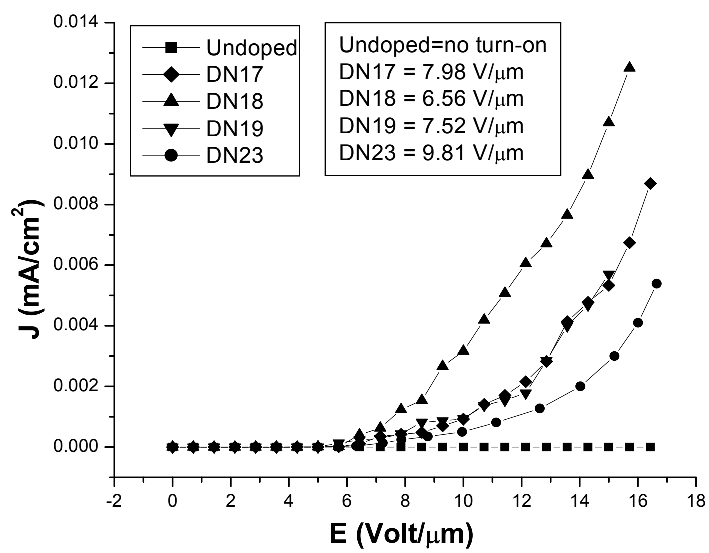


Fig. 9. Emission current density (J) vs. electric field (E) plots for undoped and N-doped DLC films (DN17, N_2-68 vol.%; DN18, N_2-50 vol.%; DN 19, N_2-34 vol.%; DN 23, N_2-11 vol.%)

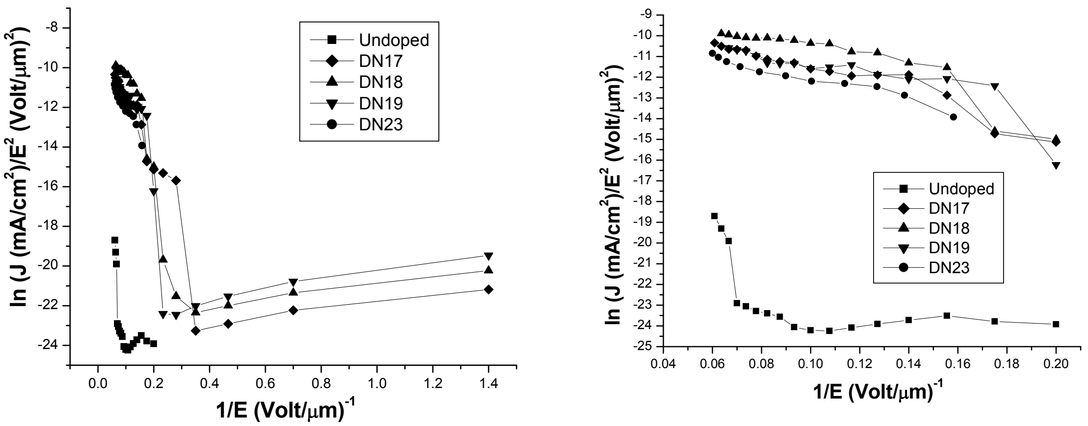


Fig. 10(a). F-N Plots for undoped and N-doped DLC films (DN17, N_2-68vol.%; DN18, N_2-50 vol.%; DN 19, N_2-34 vol.%; DN 23, N_2-11 vol.%) and Fig. 10(b) Redrawn F-N plots in the high field region

4. CONCLUSION

Diamond like carbon films (DLC) were synthesized on single crystalline Si (400) wafer and on glass substrates by DC plasma enhanced chemical vapour deposition method using acetylene as precursor gas. Nitrogen doping was done in the DLC films by introducing nitrogen gas at various volume percentages. The deposited films were amorphous, as evident from XRD spectra. FTIR measurements showed various C–H bonding vibrations present in the DLC films. With the incorporation of nitrogen into the DLC matrix different CN absorption bands appeared in the FTIR spectra. Highest $sp^3/(sp^2+sp^3)$ ~ 85.65% was achieved in the undoped DLC film, as calculated from deconvoluted C–H absorption bands. Nitrogen incorporation increased the room temperature conductivity of the DLC films by two orders of magnitude. Tauc gap of the films was decreased with the increase in nitrogen volume percentage in the plasma. Electron field emission properties were also improved with the incorporation of nitrogen in the DLC films. Lowest turn-on field (for current density of 0.5 $\mu A/cm^2$) achieved was 6.56 volt/μm. Effective emission barrier was reduced from 0.08 eV for undoped DLC film to 0.04 eV by nitrogen doping. Thus this material with optimum nitrogen doping can be used in field-emitter devices. By adjusting nitrogen doping percentage the optical gap of the material can be tuned and can be used in photovoltaic devices.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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