



Effect of electron irradiation on morphological, compositional and electrical properties of nanocluster carbon thin films grown using room temperature based cathodic arc process for large area microelectronics



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ABSTRACT

The influence of 8 MeV electron beam bombardment on room temperature grown nanocluster carbon using cathodic arc process has been studied here. Atomic force microscopy (AFM) study shows that surface roughness varies with varying electron doses. High doses of electrons could cause thermal induced graphitization and morphological changes in the films. Raman spectroscopy analysis reveals that G-peak vary from 1555 cm^{-1} to 1570 cm^{-1} and D-peak varying from 1361 cm^{-1} to 1365 cm^{-1} indicating the disorderliness and presence of both graphitic and diamond-like phases. Room temperature conductivity changes by two to three orders in magnitude. The conductivity in the films could be due to conduction of charge carriers through neighboring islands of conductive chains. Defect states calculated using the differential technique varies from $8 \times 10^{17}\text{ cm}^{-3}\text{ eV}^{-1}$ to $1.5 \times 10^{19}\text{ cm}^{-3}\text{ eV}^{-1}$. Irradiation of nanocluster carbon thin films could be helpful to tune the electrical properties and defect densities of the nanocluster carbon films for various large area, flexible electronic and nano electronic applications.

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1. Introduction

Carbon films by virtue of its versatility have been found possessing extraordinary properties to be used in optical windows, magnetic storage disks, car parts, biomedical coating, field-effect transistors, antifuses, low dielectric constant material, field emission devices and as micro-mechanical devices (MEMS) [1–5]. All these devices have been fabricated using different chemical and physical techniques. Famous physical techniques involve bombarding the films surface by radiation of ions, photons and electrons [6–8]. Physical and structural properties of the films can be changed due to electron irradiation of carbon films. Young's modulus, maximum hardness and elastic hardness, of disordered carbon films are the function of electron dose [8]. Due to electron irradiations, carbon films form various structures like nano-columns [8],

nano-sized tubules [9], carbon onions [10], nano-crystallites of diamond [11], carbon nanotubes, fullerene like structures [12], sp^2 structures from sp^3 [13].

Irradiation of disordered carbon films has been always a subject of active interest as it often results in change of morphological, electrical and optical properties of the films. Energetic ion falling on regular hexagonal structure in graphitic like material breaks the structure which results in decrement of conductivity. Similarly, for insulating polymeric film irradiation creates micro-crystals along the path of ion bombardment. This results in increase in conductivity. Disordered carbon films have been subjected to various sources of irradiation like Pulsed UV laser annealing of hydrogenated amorphous silicon [14] and in textured carbon films [15], pulsed laser UV irradiation of amorphous carbon films [16], irradiation of tetrahedral amorphous carbon films by energetic (2 MeV) Au^+ beam [17] etc.

Nanocluster carbon thin film (NC) is one form of disordered carbon film containing clusters of various dimensions [18].

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Nanocluster carbon thin films have been grown using the room based cathodic arc process [18–21]. The room temperature cathodic arc process offers unique opportunity to grow various forms of nanocarbon thin films, starting from predominantly sp^3 (diamond like) to predominantly sp^2 (graphite like) films. Cathodic arc process is a high energetic process and variation in ion energy leads to growth of films of different morphology and composition varying from atomic smooth films to clusters with varying dimension and fibrous films [18]. These films have also been reported to exhibit good electron emission properties, conducting channel for Thin-Film Transistor (TFT) and resonant tunneling behavior [22–24]. So, effect of irradiation on mixed phase nanocluster carbon thin film to be used as an insulator or semiconductor has been explored here. Radiation by 8 MeV variable energy Microtron source energetic electrons could induce structural and electrical changes in NC thin films synthesized at room temperature by means of the cathodic arc process. Our primary aim is to study the morphological, room temperature (RT) electrical conductivity and defect density of states (DOS) of films with various doses of radiations. At last, compare the same with non-irradiated film which could be suitable for nano/microelectronics, optoelectronic, flexible electronics and large area electronics applications.

2. Experimental methods

Nanocluster carbon (NC) thin films have been grown using vacuum cathodic arc process [18]. In this deposition process, a carbon striker electrode touches the graphite cathode with high energetic plasma of C^+ ions and having high the degree of ionizations. A pure graphite rod has been used as the cathode for deposition process. Deposition chamber has been maintained to a vacuum of $\sim 10^{-7}$ Torr, before initiating the deposition process. The C^+ ions have been guided through a magnetic field and made to deposit on silicon. In this process, hydrogen and helium gases have been used as a background gas to influence the morphology of the films. The helium environment is used to control the energy of the ions and to facilitate clustering process. Film thickness of 100 nm has been grown on the silicon substrates. Thickness profiler has been used to measure the thickness of the films.

Four samples have been irradiated with 8 MeV electrons from variable energy microtron equipment. The unit of dose is Gray (Gy). The irradiation work has been carried out at Microtron Centre, Mangalore University, India [25]. The films have been irradiated with doses of 500 Gy, 1000 Gy and 2000 Gy. Here, the dose of 1000 Gy is equal to 2.5×10^{17} electrons/cm². The indigenously developed variable energy microtron provides 8 MeV electrons, bremsstrahlung radiation of peak energy 8 MeV and neutrons of reasonably high flux for variety of research activities in interdisciplinary areas of science and technology. The microtron is currently being operated with a radiofrequency cavity type I and directly heated LaB₆ single crystal (cylindrical) cathode of dimension 3 mm × 3 mm. Bremsstrahlung radiation of peak energy 8 MeV is generated by making 8 MeV electrons from the microtron to fall on a high Z material, tantalum. The bremsstrahlung radiation is also used for neutron production through (γ, n) reaction. Neutrons of yield 10^9 neutrons/s can be generated using the facility with a suitable neutron converter fabricated using beryllium discs. A brief feature of Microtron is given in Table 1.

The surface morphology of the films has been examined using Nanosurf® EasyScan 2 atomic force microscopy (AFM) under room temperature. Atomic force microscopy (AFM) measurements have been carried out to extract surface roughness of the films. The lateral scan area is 27 μm × 27 μm for all the films. Each measurement contains 256 × 256 data points. The scan rate is 3 Hz. The root mean square (RMS) surface roughness and other statistical

Table 1
Important features of Microtron Source [25].

Parameters	Values
Beam energy	8/12 MeV (variable)
Pulse current	50 mA (max)
No. of electron orbits	14
Beam size	3 mm × 5 mm
Pulse duration	2.5 ms
Pulse repetition rate	250 Hz (max)
Average beam power	375 W (max)
Magnet field strength	1927.5 G
Magnetron power	2 MW
Operation frequency	2998 MHz

distribution of grain size of AFM images have been carried out using Gwyddion software [26]. The Raman measurements have been carried out using a Renishaw micro Raman system and an excitation wavelength of 514.5 nm. It has been used to obtain information regarding the structural changes within the irradiated areas. Raman spectra have been deconvoluted using Briet–Wigner–Fano (BWF) lineshape for G-peak and Lorentzian lineshape for D-peak.

Current–Voltage (I–V) measurements have been carried out with Al/NC/c-Si device structures. It is fabricated on highly doped (100) n++ silicon wafers of resistivity 0.001–0.004 $\Omega\text{-cm}$. Aluminium contacts have been fabricated by thermal evaporation technique under the vacuum of 10^{-6} Torr. The contacts are ohmic in nature. All measurements have been carried out at room temperature at constant voltage of 2V using Keithley 6517 electrometer. The data have been acquired using IEEE-488 bus and programmed using MATLAB.

3. Results and discussion

Surface morphology, compositional, electrical resistivity and defect density of electron irradiated NC thin films have been studied.

3.1. Morphological study of irradiated NC thin films using AFM

Surface morphology of non-irradiated and irradiated NC thin films samples using AFM are shown in Fig. 1. Atomic force microscopy (AFM) measurement yields information on roughness, three-dimensional topography and fractal analysis of the films. Roughness values extracted from AFM measurement show that the surface roughness increases as the electron dose increases and then decreases. Rrms values of films for 0, 500, 1000 and 2000 Gy electron doses are found to be 20.3, 20.8, 22.9 and 20.4 nm, respectively.

Generally, thermal annealing is the common technique to improve electrical and optoelectronic properties of these films [27]. Here, irradiation source has been used to find the morphological changes in the films. Surface roughness increases as electron doses increases from 0 to 1000 Gy and then decreases at 2000 Gy. The transition from the smooth surface to the rough surface and then back to smooth surface is evident from Table 3. This could be possible due to the phase transformation of sp^3 bonded material to sp^2 bonded material due to use of energetic electrons. Surface has been modified by bombardment of the energetic electrons. According to sub-plantation model [28], films are transforming due to breaking of bond i.e. from sp^3 to sp^2 bonds. Radiation is enhancing diffusion phenomena (atomic displacement) in a substantial amount by which the surface roughness is going to increase with an increase in sp^2 (graphitic bonding) and decrement in sp^3 (diamond-like bonding).

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