SYNOPSIS OF

CHARACTERISATION OF NANOCARBON THIN FILMS
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CHARACTERIZATION OF NANOCARBON THIN FILMS

1. INTRODUCTION

*Nature* and its unique designs make economic use of materials by optimizing the design of the entire structure or system to meet various needs. The designs found in nature are the result of millions of years. The transfer of technology from nature to engineering is referred as Biomimetics [1-5]. The advances in science and technology leading to knowledge and capabilities have brought the ability to create technology that is far beyond the simple mimicking of nature. Today, the science and technology has better tools to understand and to implement nature’s principles. It focuses on characterizing the structure and mechanics of natural materials into new, high-performance engineering materials. Biomimetics is a biologically inspired technology [1-3]. For example mimicking of a heart resulted in the design of sophisticated heart pumps [4], and mimicking of sensory organs like eye resulted in bionic eye [5].

Nature employs many inorganic materials together with organic matter to create family of organisms [2]. It is very difficult for us to mimic the nature materials or systems, because of incomplete understanding of natural biochemical or biosynthesis processes. The nature materials are highly ordered, self-generating, hierarchical, multifunctional, adaptive, self-repairing and biodegradable structures. The possibilities exist for the synthesis of materials by mimicking biological systems [1-5]. The innovations in the material science has led to the evolution of a series of carbon allotropes having diverse characteristics, which could be extremely useful for *engineering a functional systems at the molecular scale* [6, 7].

The advances in computing power, materials modeling, coupled with significant advances in characterization tools, which have provided additional factors that have enabled the design and development of self-aligned nanomaterials for specific applications [8]. The surface to volume ratio is an important factor in case of dimension reduction [9]. The characteristic of material drastically changes with change in the surface to volume ratio. Thus the scientists focus, from microelectronics has turned towards nanoelectronics on one side and large area & flexible electronics on the other sides [7, 8]. This trend is useful to focus on increasing the performance by other means than just scaling and it is referred as “More-than-Moore” (MtM) Technology [7], which allows the non-digital functionalities (e.g., RF communication, sensors, actuators) to migrate from the system board-level into the package (SiP) or onto the
chip (SoC) [7, 8]. As per ITRS, the nanostructures are expected to be the building blocks of future nanoelectronics or biomedical systems, where self-aligned, ordered, self-generating, multifunctional, adaptive, self-repairing, biocompatible and biodegradable nano-structures plays a major role [6-11].

Carbon as a material has shown more diversity compared to other nanomaterials. The carbon atoms bond differently leading to the evolution of materials like diamond, graphite, fullerenes, carbon nanotubes, diamond like carbon and tetrahedral amorphous carbon, carbon nanoclusters and others [8,10-17]. However the interesting characteristics, assured functionalities, and promising results motivated most of world’s researcher’s to focus on the study of a family of nanocarbons, and its characterization.

2. LITERATURE REVIEW

Carbon based nanotechnology began with the discovery of fullerenes (1985) [18], the carbon nanotubes (1991) [10] and graphene (2004)[12]. Nanocarbons in its different manifestation, exhibit many interesting physical, structural and compositional properties [19, 20]. The unique properties of these nanocarbons include low electron affinity, high mechanical strength, chemical inertness, high conductivity, high temperature stability and hydrogen storage capability [8, 13-15, 17]. The carbon nanotube has high tensile strength (hundred times that of steel), better thermal conductivity and the electrical conductivity similar to copper, but with capability of withstanding higher current densities [13].

Graphene is an allotrope of carbon [12, 21] and its structure is one-atom-thick planar sheets of sp²-bonded carbon atoms that are densely packed in a honeycomb crystal lattice. It is a two dimensional (2-D) with a high crystal and electronic quality. It can be wrapped up into 0-D buckyballs, rolled into 1-D nanotubes or stacked into 3-D graphite. Graphene is considered as the 2D building block for carbon allotropes of every other dimensionality. Graphene may be extracted from a piece of graphite using top-down approach or may be grown on SiC with the bottom up approach [12].

One of the hardest and strongest, Diamond like Carbon (DLC) is known as tetrahedral amorphous carbon, or ta-C. The tetrahedral amorphous carbon may have majority of sp³ bonded carbon atoms [21]. Thus we have the unique of opportunity of tailoring various novel materials by using the diverse properties of carbon based materials.
The nanocarbons morphological details may be studied with SEM [22] or AFM [23]. The compositional details such as sp² or sp³ bonding are generally studied with Raman spectroscopy [24] or with EELS [25]. Raman spectroscopy is a powerful system for the fundamental study of molecular structure, dynamics and is also a nondestructive and instantaneous tool for nanomaterial characterization [26].

Nanocarbon has wide ranges of possible applications which include biomedical engineering [27], life science, biotechnology [28], energy systems, and communication systems [29-35]. The design of miniaturized X-ray source is a promising application, and is based on vacuum electronics concept. Vacuum nanoelectronic devices use the property of field assisted electron emission under vacuum [27, 36, 37]. These emitted electrons from nano or micro sized electron sources found used even to develop sensor.

However there are other applications based on electron emitters which include: field emission displays, large area electron beam lithography, electron and ion guns, a wide variety of sensors, electron microscopes and microprobes, low & medium power microwave sources, micro and pico satellite propulsion systems, high power devices and Tera Hz communication devices [29-35]. For example the electronic properties of CNTs are sensitive to the adsorptions of certain type of gas molecules, which make CNTs a good sensor [27, 29, 33].

2.1. Thin film process technology

Nanocarbons are grown with different process techniques. They may have different size, structure and properties. The nanodiamond films are grown using Hot Filament Chemical Vapour Deposition (HFCVD) [38]. The carbon nanowalls were grown using DC plasma Chemical Vapor Deposition [39], carbon nanotubes grown using Thermal CVD[40] and the cluster assembled carbon films grown using Pulsed Laser Assisted cluster beam assembly source [41] and nanocluster carbon films grown using the Cathodic Arc process [19,42]. Cathodic arc deposition is a plasma based technology for the fabrication of nanocluster carbon thin films. The process can be carried out either at high vacuum or in a low pressure gaseous environment using continuous operation (DC) cathodic vacuum arcs (CVA) or using pulsed cathodic arc system. Along with carbon thin films, grown other films like metallic or ceramic, semiconductors, super conductors, and more [43,44].
The nanocarbon films are grown using different processes at various deposition conditions may have variations in morphological, dimensional and compositional properties [19]. The most of the reported nanocarbon films have been grown at relatively higher temperatures (700 – 1000°C) [19, 45]. But cathodic arc system has proven to be a suitable technology for electronic materials [36]. Its compatibility with existing semiconductor technology, made it so attractive for large area or flexible microelectronic applications.

### 2.2 Characterization of nanocarbons

To use nanocarbons effectively it is important that it is fully characterized, with respect to its diverse physical properties including morphological, dimensional, structural, compositional, electrical, electronics and optoelectronic. Generally the morphological or dimensional details of nanocarbons are studied with the material probing tools like Scanning Electron Microscopy (SEM), Atomic Force Microscope (AFM). The structural details are studied with high resolution transmission electron microscopy (TEM) and x-ray diffraction (XRD). The bonding nature of carbon atom such as \( sp^2/sp^3 \) bonding ratio is studied using EELS [25] or Raman spectroscopy [46-49]. However these characterization techniques are not well established especially while looking for nanocarbon nature or composition analysis. Among many techniques Raman spectroscopy offers an indirect way to probe the material characteristics instantaneously [46-55]. This indirect method has been widely used as tool for research and industrial applications. In the case of materials like amorphous carbon [46,48,50,55], Diamond Like Carbon (DLC)[35], tetrahedral amorphous carbon (ta-C)[19,56] and carbon nanotubes[57] there is a reasonable amount of literature. Even here the analysis is not well established.

In case of Raman studies of nanocarbons, the visible Raman spectrum shows one or two prominent features and other minor modulations. The prominent features are G (Graphite) Peak and D (Disorder) Peak [46]. Raman measurements are used to study the bonding and compositional nature of nanocarbon films. Recent reports show that similar to our effort there are preliminary reports on a qualitative analysis and correlation with SEM and Raman responses of CNTs to understand the properties and use it to try automating the production of nanostructures [47]. Among many nanocarbons, the mixed phase low temperature grown nanocluster carbon is one of the most promising material [36, 58-60]. Here Raman spectroscopy may be a useful technique for characterizing \( sp^2 \) and \( sp^3 \) hybridized carbon atoms [62]. Raman spectroscopy is one of the most sensitive techniques to characterize...
disorder in sp\textsuperscript{2} bonding nature of carbon materials [52, 53]. The Raman signatures such as the intensity [I(D)/I(G)] ratio, G peak position and FWHM (full width half maximum) were found to change gradually with the deposition parameters or with the carbon structure [46]. The amorphous carbon films were analyzed by laser Raman spectroscopy (LRS) at 514.5nm, along with X-ray photoelectron spectroscopy (XPS) [51]. Curve fitting numerical techniques are generally used for analyzing the Raman data [46, 48, 52, 53].

The Raman response of DLC has two peaks called D and G peaks respectively. DLC structure has higher sp\textsuperscript{3} bond compared to sp\textsuperscript{2} and here the D peak intensity is higher [63]. However Raman spectroscopy is already considered as good tool for nanocarbons study. For example, Raman spectroscopy may be employed to differentiate between metallic and semi-conducting nanotubes [53].

Nanocarbons may be used along with biomedical implantable device or system, only by understanding its nature, characteristics, and biocompatibility [64]. The combined studies of diversified nanocarbons with varying process parameters are important for future nanocarbon based developments.

3. NEED AND MOTIVATION FOR THE RESEARCH WORK

Carbon in various form have been studied extensively. However, these studies do not seem to look at leveraging the properties in an integrated way. To make use of benefits of the varied form of the carbon, it is important to understand the morphological, dimensional, compositional, structural similarities or dissimilarities between them. Hence, the motivation is to study the nanocarbons and to understand a correlation between morphological, dimensional, compositional, and structural properties. Further, most of the nanocarbon may be grown with high temperature process. Generally, the nanocarbon process technology is not compatible with the conventional semiconductor technology. Hence, there is a need to look at compatible technology alternatively and preferably at low temperature or at room temperature. It is always desirable to have a low temperature process as this enables deposition on substrates such as glass and plastic. There is a possibility to use this nanocluster carbon in the large area microelectronics, printed electronics, and in flexible or stretchable electronics. Thus, there is a huge potential to study these mixed phase low temperature grown nanocluster carbon.
4. OBJECTIVE

To study wide variety of nanocarbons and to look at their morphology, dimension, structural and compositional properties along with the room temperature grown nanocluster carbon for consideration in the area of nanotechnology, flexible electronics, large area microelectronics, vacuum nanoelectronics and biomedical engineering.

Given below are the key objectives:

- Proposed to study wide range of nanocarbons, along with a low temperature grown nanocluster carbon (grown using cathodic arc system).
- To use SEM (Scanning Electron Microscope) data for studying the morphological and dimensional properties.
- To use Raman spectroscopy to understand the sp\(^2\)-sp\(^3\) bonding (indirectly), a possible correlation to size and possible grouping of nanocarbons.
- To study and understand if there is correlation, between process conditions or growth conditions and a) Morphology, b) Dimension, c) Composition, d) Structure and e) Electrical and Electronic properties.
- Finally, to look at feasibility of usage of these nanocarbons for device or sensor based application.

5. METHODOLOGY

Typically diamond (sp\(^3\) bonded) and graphite (sp\(^2\) bonded) are considered as naturally available allotropes of carbon. Compiled data from the literature including graphitic like (sp\(^2\) rich bonding), diamond like (sp\(^3\) rich bonding), and correlated their morphological or compositional details. I have studied nanocarbons grown at higher and lower temperature process, which includes: nanodiamond, nanopillar, t-aC, nanowalls, and graphene. The nanocluster carbon is grown at low temperature using cathodic arc approach. In this research work, I have considered SEM and Raman data of nanocarbons for morphological study and classifications of the nanocarbons based on indirect sp\(^2\)/sp\(^3\) bonding information.

I have studied various nanocarbons from diamond (structure has sp\(^3\) bonding) to graphite (structure has sp\(^2\) bonding), to that nanocluster carbon (sp\(^2\) bond rich) films. To understand the process of growth of novel room temperature grown nanocluster carbon, studied the...
functioning of cathodic arc system and various deposition parameters associated with the growth of nanocluster carbon. The morphological or surface details of the carbon samples are visualized using Scanning Electron Microscope (SEM) and developed a dimension analysis program to enhance the capability of in-situ surface analysis. The compositional property of nanocarbons are studied indirectly with the Raman response. With the help of numerical technique (Lorentzian peak fit) analyzed the Raman response and extracted parameters such as peak position, peak intensity, peak intensity ratio, full width half maximum (FWHM) of the G peak or D peak are considered as good signatures of the nanocarbon. Raman study is nondestructive and are used for evaluating the sp²/sp³ bonding nature of nanocarbon. Reported here a protocol to classify nanocarbons into groups such as CNT, nanodiamond and nanocluster carbon.

To identify the nanocluster carbon whether it is metallic or semiconductor or insulator, established electrical experimental facility. The nature of the nanocluster carbon was studied with the with the conductivity measurement.

To understand and demonstrate the feasibility of the nanocarbon for application an experimental set up for field assisted electron emission was established. The field emission measurements were carried out with a nanocarbon as cathode and phosphor coated on transparent conducting glass as anode. Thus possibility of usage of vacuum nanoelectronics application is studied.

5.1 Cathodic arc system

Cathodic arc system is evolving as an interesting process technology for growing wide range of metals, semiconductors, oxides at relatively low temperature [19, 65] at high vacuum (better than 10⁻⁵ Torr). The system has linear or bend (90degree/ S shaped) duct and a magnetic filter to minimize macro-particle deposition. The nanocluster carbon are grown on the glass and silicon substrate using a pure graphite disc (used as cathode). The arc system is triggered with mechanical trigger to initiate the plasma. The vacuum arc produces a highly ionized plasma stream with energetic ions. The controls of these ions by varying its energy or by facilitating its interaction with other molecules, nanocarbons with varying structure composition and physical properties can be grown. A broad range of process parameters used to grow nanocluster carbon are given below in the table 5.1. This growth process study
could enable us to understand the feasibility of growing nanocarbons with varying morphology, composition and physical properties.

Table 5.1: List of deposition conditions for low temperature grown nanocluster carbon

<table>
<thead>
<tr>
<th>Sample No</th>
<th>base vac. (Torr)</th>
<th>N2 flow (SCCM)</th>
<th>He flow (SCCM)</th>
<th>Total pressure (Torr)</th>
<th>Dep. time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>5.13E-07</td>
<td>90</td>
<td>425</td>
<td>4.86E-01</td>
<td>120</td>
</tr>
<tr>
<td>S2</td>
<td>5.00E-07</td>
<td>70</td>
<td>500</td>
<td>3.65E-01</td>
<td>120</td>
</tr>
<tr>
<td>S3</td>
<td>6.50E-07</td>
<td>50</td>
<td>500</td>
<td>3.52E-01</td>
<td>120</td>
</tr>
<tr>
<td>S4</td>
<td>4.24E-07</td>
<td>10</td>
<td>200</td>
<td>2.21E-01</td>
<td>110</td>
</tr>
<tr>
<td>S5</td>
<td>3.94E-07</td>
<td>30</td>
<td>200</td>
<td>2.47E-01</td>
<td>110</td>
</tr>
</tbody>
</table>

5.2 Morphological and Dimension Analysis

The nanocarbon samples are imaged with scanning electron microscope (SEM) for the visual details. Then established a methodology for estimation of cluster dimension/ size and distribution. The developed program for analyzing the SEM image enhances the cluster regions, using watershed algorithm [66]. The cluster dimension or size is represented with histogram. The histogram with lesser width indicates whether the film has uniform sized cluster distribution or not. This quantitative approach for thin film analysis helps in indirect qualitative analysis of the thin film because surface to volume ratio is critical.

5.3 Raman Response study

To understand the nanocarbon compositional details or to group it under a pre-labeled classes of nanocarbon, studied nanocarbons with the measurements obtained from Reinshaw Raman spectroscopy (with the source as 514.5nm). These nanocarbon responses are studied as it is and with preprocessing (smoothing, filtering, interpolating and normalizing). Considered nanocarbons grown at various process or at various deposition conditions and looked their signatures (such as peak position). Raman response of each nanocarbon (sp² bond rich, sp³ bond rich or mixed phase samples) has a unique signature of that material, for example diamond has a sharp peak at near 1332 cm⁻¹. The lorentzian curve fitting technique help us to identify the two curves (D and G). The parameters obtained from Raman response G / D curve such as peak position or width of the peak, are considered as features or parameters of
Raman are then correlated with the morphological, dimensional and with compositional details.

5.4 Electrical /Electronic Characterization

To ascertain the nature of nanocluster carbon whether it is metallic or semiconducting, established electrical measurement is set up. The electrical measurement is carried out with the conductivity measurement. The samples grown on the glass are used for the coplanar configuration measurement. The current is measured for nanocluster carbon thin films from room temperature to 180°C with an interval of 5°C, with the voltage source kept constant (20V). Then the conductivity is calculated as follows:

Conductivity= \( \frac{\text{length} \times \text{current}}{\text{voltage} \times \text{width of channel} \times \text{temp}} = \frac{L \times I}{V \times W \times T} \)

Typical values of length of the channel (L)=7.5mm; width (W)=0.6mm; thickness of the film=120nm; Temperature in kelvin.

To check if there is a mark difference between vacuum and ambient conditions, the experiment for measurement of conductivity is repeated under vacuum. The experiment was conducted with a chamber base pressure of \(10^{-5}\) Torr.

5.5 Field emission measurement set up

For understanding the field assisted electron emission characteristics of the nanocarbon and to look at its feasibility of usage for vacuum nanoelectronics application, established field assisted electron emission experiment is set up. Reported the field assisted electron emission measurements of various nanocarbons includes nanocluster carbon, nanowall, nanodiamond and carbon nanotube. The vacuum diode experimental configuration has an anode (patterned phosphorous screen) and a cathode (field emitting electrode) arrangement in a vacuum chamber at 1.2mm distance using a glass spacer. The voltage source applied across the anode and the cathode is varied during the field assisted electron emission experiment. The nanocarbon thin film is arranged inside the vacuum chamber, with the help of high quality spacer glass. Chosen corning 7059 glass as spacer, it has smooth, flat surface, with a near-zero Alkali, and non-ionic properties. The Zinc sulfate (green color phosphor) patterned over the glass surface is considered as the screen. The diode configuration with an anode consisting of patterned phosphorous screen and cathode being a nanocarbon film, are
arranged in a vacuum chamber. The experiment was carried out with a chamber vacuum of about $10^{-6}$ Torr.

6. RESULTS AND DISCUSSIONS

A series of nanocarbons, grown at various process conditions including CNT (carbon nanotube), nanodiamond, nanocluster carbon, Nanowalls and Nanopillars are studied for understanding their morphology, dimension, composition, electrical and electronic properties. These nanocarbons were grown with various processes under different deposition conditions, at various temperature levels. The reported nanodiamond films are grown using Hot Filament Chemical Vapor Deposition (HFCVD) at around $800^0$ C, the carbon nanowalls are grown using DC plasma CVD at $900^0$C. The carbon nanotubes are grown using Thermal CVD at $750^0$C. The nanocluster carbon films with varying $sp^2/sp^3$ bonding are grown at room temperature using the cathodic arc process.

6.1 Morphological characterization

The morphology of the grown nanocarbon defines the surface details of nanocarbon. The surface to volume ratio of nanocarbon with varying dimension or size is key to the material characteristics. These characteristics are the key for design of novel biocompatible surface, devices, sensors, biomimetic microelectronic systems (BMES).

The morphological details of nanocarbons were studied using SEM micrographs. Shown in figure 6.1 are various nanocarbons such as a) Diamond films, (b) Nanostructured Graphitic sheets (nanowalls), (c) Nanotubes, (d) Nanocluster carbon films, (e) Nanopillars and (f) taC. These images have varying morphology either as clusters or tubes or pillars or walls. These varying sizes of structures has varying shapes, varying levels of surface roughness and protrusions. No two materials are seen to be same. The protrusions vary from a few nanometers to over a few microns depending on the material and the process of growth. The surface morphological feature has great influence on the electron emission [37] and also important for surface based sensor designs. The sharp protrusion regions are of interest for field emitter arrays and sharp regions act as field enhanced active emission sites. The SEM shown in figure 6.1(a-d) has scale of 1cm=1micro meter (Please consider the scale of 1cm=1micro meter for the SEM without scale). The morphology study of nanodiamond film surface indicates availability of clusters. The sizes of these clusters are about 300nm and are
shown in figure 6.1 (a). The distributions of these clusters over the substrate are more or less uniform. In figure 6.1 (b) are the nanowalls and they show 5-10nm thicker vertical walls of carbon. The carbon nanotubes having diameter of about 10-15nm, are shown in the figure 6.1 (c). These nanotubes are available in a bundle, consists few cluster growth attached to it as shown in figure 6.1(c). The figure 6.1 (d) shows vertical protrusions referred as pillars and are about 15-20nm of perimeter.

Fig. 6.1 SEM images of nanocarbon films grown using different process techniques: (a) Diamond films, (b) Nanostructured graphitic sheets (nanowalls), (c) Carbon nano tubes, (d) Nano cluster carbon films, (e) Nanopillars, and (f) taC.

6.1.1 Influence of the process parameter’s on the nanodiamond growth

The morphology of the nanocarbons is found to vary widely based on the process parameters. In case of the nanodiamond grown with the HFCVD process, the morphology change is studied with methane concentration and the temperature variation. The samples studied have shown increase in the nucleation density and a corresponding increase in surface coverage of the substrate when the methane concentration was increased. The methane concentration of 1% (in hydrogen), resulted in clusters of 200nm size. The increase of methane concentration from 1% to 3% increased the size of clusters nearly double. The study of nanocluster carbons with increased temperature from 775°C to 925°C, resulted in the growth of nanoclusters of size varying between 500nm-700nm. The nanodiamond grown with temperature 775°C has rough cluster surface. Whereas increase in temperature the nanocluster surface are modified from rougher to smoother (within the sample set considered) [19].
6.1.2 Influence of process parameter’s on nanocluster carbon

The morphology of nanoclusters grown at various deposition conditions are studied using SEM / AFM image. The morphological changes are observed with the change of concentrations of helium and nitrogen along with the other process parameters such as set distance or substrate bias. The increase in nitrogen partial pressure has shown small clusters in taC at nano dimension of 5-10nm [25]. Too much incorporation of nitrogen results in poor quality of nanocarbon films. At lower partial pressure of nitrogen typically the film observed to be smooth. The nanocluster carbon samples grown at nitrogen partial pressure $10^{-3}$ Torr and $10^{-4}$ Torr have shown clusters from 100-600nm. The addition of helium along with nitrogen improves the growth of cluster, and as a result morphology changes.

The morphology of the films is varying smooth surface to surface with clusters, and a few samples have fibers. The SEM micrographs of nanocluster carbon films shown in figure 6.2 (a) has surface details such as a) smooth film (No helium), (b) nanocluster film (0.05 Torr), and (c) fibrous film (50 Torr). The SEM micrographs of nanocluster carbon films grown with a nitrogen partial pressure of $10^{-3}$ Torr, with helium partial pressures around 0.05Torr have shown very good clusters surface. Morphological changes may severely affect surface smoothness or characteristics of the film. He=0.05Torr to He < 5Torr may be recognized as the optimum range of helium to be maintained during the process for a better, cluster formation and its uniform distribution.

![SEM micrographs](image)

Fig. 6.2 (a) Room temperature grown nanocluster carbon with varying morphology: a) atomic smooth film with specs of macro particles (No helium), (b) Nanocluster film (He partial pressure of 0.05 Torr), and (c) Fibrous film (He partial pressure of 50 Torr)

The nanocluster carbons grown using cathodic arc deposition system with a base vacuum of $10^{-7}$ torr range using different arc types shows morphological changes. The surface details of films are influenced by arc type [19, 39]. The nanocluster carbon samples are deposited under varying helium partial pressure [$10^{-4}$ Torr to 10 Torr] conditions, for a fixed nitrogen partial
pressure $[10^{-3}\text{Torr}]$ using a continuous cathodic arc process has shown 100-600nm clusters [19]. Shown in figure 6.2 (b) is the SEM image of nanocluster carbon grown using pulsed cathodic arc system has clusters of size between 50-100nm.

![SEM image of nanocluster carbon grown using pulsed cathodic arc system](image)

Fig. 6.2 (b) The SEM of nanocluster carbon ($\text{sp}^3$) grown from the pulsed cathodic arc system.

The clusters are seen distributed on the surface of the substrate with overlapped in nature, but compared to continuous arc deposition these clusters have smaller size. Continuous arc process generates more macro particles; to minimize it needs magnetic filtering which increases the system cost and bulkiness. It was observed that the pulsed cathodic arc process generates relatively lower macro particles even when no filtering process was used. The comparative study of nanoclusters carbon films shows growth of moderate uniform and good clusters in the pulsed arc process. The results indicate that with further optimization, it should be possible to grow more controlled, low field electron emitting nanoclusters using the pulsed cathodic arc process.

Shown in figure 6.3 are the nanocluster carbon films grown using cathodic arc system. The deposited films have smooth surface and distributed clusters of size varying between 300-500nm.

![SEM images of nanocluster carbon films](image)

Fig. 6.3 Low temperature grown nanocluster carbon films deposited on separate wafer shows similar morphology.

The details of the deposition parameters such as nitrogen concentration, bias, substrate deposition time are listed in the table 6.1. The samples deposited on different wafers under similar deposition conditions indicated similar cluster growth and distribution.
Table 6.1 Nanocluster carbon deposition conditions

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Base vacuum (Torr)</th>
<th>N2 (SCCM)</th>
<th>Chamber</th>
<th>DC bias Voltage</th>
<th>Amperes</th>
<th>Set position</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>7.93E-07</td>
<td>1.2</td>
<td>2.15E-05</td>
<td>-40</td>
<td>0.5</td>
<td>190</td>
</tr>
<tr>
<td>B</td>
<td>8.60E-07</td>
<td>1.2</td>
<td>1.10E-05</td>
<td>-40</td>
<td>0.5</td>
<td>190</td>
</tr>
</tbody>
</table>

Similarly the morphology of the low temperature nanocluster carbon films are studied using AFM. The AFM images of smooth nanocluster carbon thin films is shown in figure 6.4 has topographical variation of 2-3nm.

![AFM images of nanocluster carbon with smooth surface morphology.](image)

**6.1.3 Cluster dimension Analysis**

The image processing approach for nanocarbons, finds the size, distribution of the clusters, and helps in the evaluation of quality of the nanocarbon surface. Consider an unprocessed nanodiamond SEM image as shown in figure 6.5(a). It can be seen that the clusters are distributed on the surface non-uniformly along the substrate, even with various cluster dimension or size. The original image is preprocessed and then segmented using Watershed segmentation technique as shown in figure 6.5(b). Shown in figure 6.5(c), is the histogram plot drawn estimating the size of cluster, increase in the peak indicates increase in the cluster densities or cluster distribution. The sharper the histogram indicates more uniform cluster size (dimensions) along the film. The plot with wider width indicates availability of non-uniform cluster size. The histogram plot for nanodiamond shows 95% of the clusters have area less than 0.2 micro-meters square.
6.2 Raman Response Analysis

The Raman response is an extensively used approach for identifying compositional properties of nanocarbons. So nanocarbon compositional details are studied with the Raman measurements. In case of nanocarbons the characterization techniques are not well established, especially while looking for composition. Raman approach is an indirect method, but it has been widely used as tool for research and industrial applications.

Naturally occurring diamond is almost always found in the crystalline form with a purely cubic orientation of sp$^3$ bonded carbon atoms. The Raman response of diamond has a well-defined peak at 1350cm$^{-1}$. Shown in the figure 6.5, is a typical sp$^3$ rich bonding, diamond response with a sharp peak. The sharp peak positioned near 1325cm$^{-1}$ indicate possibility of well-structured sp$^3$ material. The graphitic like sp$^2$ rich bonding carbon film response is also shown in the figure 6.5 and it has peak intensity is around 1582cm$^{-1}$.

This sp$^2$-sp$^3$ mixed phase material has two peaks one at near 1350cm$^{-1}$ and other one near 1582cm$^{-1}$. The broad intensity curves indicate that the material is more amorphous. But in the case of materials like amorphous carbon, Diamond like Carbon (DLC), tetrahedral amorphous carbon (ta-C) and carbon nanotubes there is a reasonable amount of understanding. In case of Raman studies of nanocluster carbon, the visible Raman spectrum
shows one or two prominent features and minor modulations. The prominent features are G (Graphite) Peak and D (Disorder) Peak.

Fig. 6.5 Raman response of typical diamond film (sp³ bond rich) and nanostructured carbon (sp² bond rich).

Nanocarbons having different sp²/sp³ bonding are looked to understand the commonality or diversity within the wide spectrum. Shown in figure 6.6(a), is the Raman response of the nanocarbon samples that includes: nanowall, CNT, nanocluster carbon nanodiamond, and nanopillar. It may be seen that the responses are quite different. Raman spectroscopy is seen as an effective tool to characterize nanomaterials varying from conductors to semiconductors to insulators and also polymeric clusters. It can also be used for both qualitative and quantitative analysis of the materials. In the case of carbon, Raman can be used to identify if the material is amorphous, nanocrystalline or crystalline, the nature of bonding: if it is graphite like (sp² bonding or π bonding) or diamond like (sp³ bonding or σ bonds) and the ratio between the two (sp²/sp³) etc. It may be seen from the figure 6.6(b), that the spectrum has two regions of response.
Fig. 6.6 (a) Raman response of nanocarbon includes: a) Nanowall, b) CNT, c) Nanocluster carbon, d) Nanopillar, and e) Nanodiamond.

The G peak provides information regarding graphite cluster distribution and D is due to defects and deformations of graphite cluster. The G and D are due to sp2 sites. The G peak is due to the bond stretching of all pairs of sp2 atoms in the rings and chains. The D peak is due to the breathing modes of sp2 atoms in the rings [67].

The Raman signature of nanodiamond has sharp peak around 1335 cm$^{-1}$ the typical diamond peak and a broad peak around 1580 cm$^{-1}$ (G peak) showing the presence of graphite like and amorphous phase materials around the grain boundaries. In the case of nanowalls relatively
the narrowest G peak is around 1580cm\(^{-1}\). The very low and narrow peak at 1350cm\(^{-1}\) (D peak), and a sharp peak at 1580cm\(^{-1}\) (G peak), shows that this higher temperature grown nanowall, has relatively the least amorphous phase or disordered cluster phase material. The carbon nanotubes show both the G and D peaks like the nanowalls, but area broader. In the case of room temperature grown nanocluster carbon, it may be observed that G and D peak are even broader, than nanotubes. This indicates the presence of clusters with vastly varying dimensions and also amorphous phase nature. However the data shows the possibility of establishing a correlation between the electrical or electronic properties with Raman measurement based structural and bonding information.

The numerical approach such as lorentzian peak fit is used for extracting various hidden parameters from Raman response. The considered Raman response of five different groups of nanocarbons such as nanodiamond, CNT, nanocluster carbon, nanowall and nanopillar, are fitted with lorentzian peak fit. The two point curve fitting resulted in two important curves called as D-curve and G-curve. Here G peak is more prominent, indicates that the material is more sp\(^2\) like. Extracted various signatures from the Raman includes: G-peak position, D-peak position, D or G peak shift, G or D peak Area, Id/Ig ratio, mean of intensity of response etc. Among the nanocarbons studied the well-structured material like CNT or nanowalls has lesser G peak-Full Width Half Maximum (FWHM), compared to the amorphous materials like taC, nanoacluster carbon films.

6.2.1 Nanodiamond Raman analysis

The nanodiamond films grown with various deposition conditions are having different Raman signature. The considered samples are grown with different methane to hydrogen (CH4/ H2) ratio ranging from 0.5% to 3% (under constant temperature) and at different temperature varying between 775-925°C (under constant CH4/H2) [25, 83, 89].

Raman analysis is carried out with the curve fitting as shown in 6.7(a), has generally two peaks. The peak information such as intensity ratio (Id/Ig) ratio and peak position are correlated with the process parameters such as CH\(_4\)/H\(_2\) ratio, temperature and the results are shown in figure 6.7(b-c). Increase of CH\(_4\)/H\(_2\) concentration during the process, results in higher dimension clusters, and also resulted in higher Id/Ig ratio. The higher value of Id/Ig indicates possibility of lesser sp\(^2\)/sp\(^3\) ratio, or the material is expected to be more diamond like. The increase in temperature resulted in lesser value of Id/Ig indicates, possibility of
having materials with slightly higher value of \( \text{sp}^2/\text{sp}^3 \) ratio when compared with the earlier samples. Seen \( \text{Id}/\text{Ig} \) ratio is nearly equal or greater than one for the nanodiamond. The figure 6.7(a) indicates that, for nanodiamond D peak is more prominent.

![Graph showing Raman response](image1)

![Graph showing changes in deposition parameters](image2)

![Graph showing variation of D peak position](image3)

**Fig. 6.7(a)** Raman response of nanodiamond with curve fitting, **b)** Change in the deposition parameters (Temperature and \( \text{CH}_4/\text{H}_2 \)), **c)** Variation of D peak position with the deposition parameters includes temperature and \( \text{CH}_4/\text{H}_2 \).

Also the increase in \( \text{CH}_4/\text{H}_2 \) and Temperature, shifts the peak to right, so with increase of these parameters, the nanodiamond are expected to be more towards \( \text{sp}^3 \) bond type material. Among the selected nanodiamond samples, the one grown at 3% \( \text{CH}_4/\text{H}_2 \) concentration, has the highest value of D peak, indicates that the sample is more diamond like. This analysis is within the limited range of process parameters. These are essentially to highlight the possible correlation of Raman spectrum with the process parameters or composition.

### 6.2.2 Nanocluster carbon Raman response analysis

Shown in figure 6.8, is a typical Raman response of nanocluster carbon thin film grown at low temperature. This Raman response has a G peak and a shoulder indicated as the D peak.
due to the presence of mixed phase material with predominantly sp$^2$ bonding and has mixture of sp$^2$ and sp$^3$ bonded material. The key challenge is to correlate between composition using a simple and nondestructive means like Raman response.

The Raman response is analyzed at about 1582 cm$^{-1}$ and near about 1350 cm$^{-1}$ for the sp$^2$/sp$^3$ bonding details. The extracted two important curves from the Raman measurement using Lorentzian curve fitting approach provides important parameters like Id/Ig ratio, FWHM etc. The two point curve fitted plot is shown in figure 6.8(b) has two important curves named as D-curve and G-curve.

Fig. 6.8 a) A typical Raman response of room temperature grown nanocluster carbon, b) Raman response of nanocluster carbon after peak fit consisting two important curves called as G and D curve.

The various signatures extracted from the curve fitted Raman includes: G-peak position, D-peak position, D/ G peak shift, G/D peak Area, Id/Ig ratio, mean of intensity of response etc. It is seen that the shift of the G peak from 1580 cm$^{-1}$, influences on the clustering. Further correlated Raman signatures with process parameters, dimension/size of the nanocarbon in an intension of developing new instantaneous, nondestructive tool, further it could help in the process automation when it is standardized.

**Influence of deposition parameters on room temperature grown nanocluster carbon**

Generally cluster formation is influenced by various process conditions or agents inside the system. In case of nanocluster carbon, cluster formation there is a great influence of helium partial pressure and nitrogen concentration. Shown in figure 6.9 are the Raman response for nanocluster carbon thin films grown at various conditions of helium and nitrogen. Each has
varying signature and many Raman embedded features. The Raman response shown in figure 6.9 has more predominant D peak (shoulder). This indicates the influence of helium during the film growth. The change of helium rate predominantly influences D peak and the resulting material expected to be more mixed phase compared to the films which are grown without helium. The G peak shift towards right or left indicates the variation of cluster dimension as well its properties. The extracted parameters include: the ratio of the intensity of the D peak ($I_D$) to that of the intensity of G peak($I_G$) as $I_D/I_G$ ratio, Full Width Half Maximum (FWHM), FWHM ratio, area of G peak, area of D peak G peak position and D peak position. For more detailed information from the Raman response the G peak area were analyzed.

Fig. 6.9 Raman spectra of room temperature grown nanocluster carbon grown with varying helium concentration (ranging $5 \times 10^{-4}$ Torr to 50 Torr) under constant nitrogen partial pressure of ($10^{-3}/10^{-4}$ Torr).

The nitrogen partial pressure $10^{-4}$ Torr has greater influence on Full Width Half Maximum (FWHM) and is shown in figure 6.10(a). The peaks with broadened FWHM are higher disordered samples and there the crystallite size decreases. The small variation in intensity ratio ($I_D/I_G$ ratio) in figure 6.10 (b), indicates that most of the samples are having the composition that is predominantly sp$^2$ bond type or the clusters are highly graphitized in nature. Also correlated helium concentration changes with dimensional variation as seen from the SEM images. Under similar conditions of the process there is better cluster observed at $10^{-3}$Torr when compared to $10^{-4}$Torr. The small variation in $I_D/I_G$ ratio in the above range of study indicates that the composition those nanocluster carbons are more or less similar, but they are sp$^2$ like with disordered or the clusters are highly graphitic like in nature.
The G peak position shift at varying conditions of helium partial pressure for a nitrogen partial pressure of $10^{-3}$ Torr. This indicates that sp$^2$ composition does not change much in this range of process parameters study. However the SEM images show a greater range of change in the morphology and dimensions of the clusters.

The nanocluster carbon grown at low temperature is influenced from process parameters such as substrate biasing, substrate position, arc type (continuous or pulsed), and substrate temperature and time deposition. on the Raman signature. For example in case of samples with varying deposition time, an increase in the FWHM is seen (shown in figure 6.10(c)), indicates increase of the cluster dimension. Increase in the deposition time has inverse influence on the Id/Ig ratio indicates increased value of crystallite size (shown in figure...
6.10(d)). It is because crystallite size is inversely proportional to the Id/Ig ratio. Looking at Raman response of the samples where the set position is varying introduces G peak shoulder level change inversely (indicates possibility of lesser sp² likeness). But by arranging substrate too close to the source results with the films with macro particles. However most of the samples studied have the modulated responses and they indicate likeliness of nanocarbon as mixed phase sp²-sp³ material, with higher value of G peak, indicates possibility of more sp² like material.

6.3 Classification of sp² / sp³ mixed phase nanocarbons

The statistical way of characterization of nanocarbon’s for its classification or evaluation is carried out with the scatter plot. Calculated the mean intensity value the Raman measurement, intensity ratio (Id/Ig) from Raman response and represented nanocarbons on the sample space. The scatter plot shown in figure 6.11, represents distribution of samples on 2D space. This approach helps in nanocarbon sp² or sp³ richness based classification.

Fig. 6.11 Nanocarbons represented in the sample space, plotted using mean of Raman spectra intensity and Id/Ig ratio.

Clustering using Principle Component Analysis: Nanocarbons are classified as CNT, nanocluster carbon and nanodiamond using Principle Component Analysis (PCA) approach. PCA is the simplest of the true eigenvector-based multivariate analysis. Principal Component Analysis is a dimension-reduction tool that can be used advantageously in this situation.
Principal component analysis aims at reducing a large set of variables to a small set that still contains most of the information in the large set. PCA analysis is done with 30 nanocarbons samples which include: nanocluster carbon, CNT and nanodiamond.

The responses are preprocessed (Fourier smoothing, baseline correction, normalizing) and then analyzed with the PLS PLUS/IQ software [68]. Reported here the distribution of nanocarbon samples in the sample space. Shown in figure 6.12(a) is a simple plot, indicates distribution of samples along one of the principle component represented as Principle Component3 (Score3). Shown in figure 6.12(b), is the 2D feature space plot for nanocarbons, with the principle components (components such as PC1 & PC2). All nanocarbons here are represented as dot are seen distributed around from the reference diamond (denoted as DD1, is sp³ rich) and graphitic (sp² rich, denoted as GG1) (the nanocarbons are distributed considering around 86 % of variance). The nanocarbons are distributed in the 3D feature space consisting PC1, PC2 & PC3 as reference. This 3D representation as shown in figure 6.12 (C) has plotted considering variance of 95.2%. The nanocarbon samples with sp²/sp³ bonding nature are grouped into three groups (class or clusters). The nanocarbon classes are labeled as nanocluster carbon, CNT and nanodiamond.

![Graph showing distribution of samples](image)

Fig. 6.12 (a) Nanocarbons represented as a dot using single principle component indicates spread of nanocarbons using 1D plot, (b) Nanocarbons represented as a dot in the 2 dimensional (2D) sample space.

6.4 sp²-Sp³ model

As we pass from ordered graphite to nanocrystalline graphite (0%sp³), to amorphous carbon (~ 20% sp³) and finally to sp³ bonded ta-C (~85% sp³). The ordered structure changes then topologically disordered and finally change from ring to chain configurations. The nanocluster comes here in between NC-graphite and the a-C. The ternary diagram represented with sp² bonded graphite and sp³ bonded diamond at two corners of sp²-sp³-H.
diagram [67] indicates the nanocarbon spread in the carbon model. The nanocluster carbon studied is represented in the ternary phase diagram of carbon alloys (shown in figure 6.13).

Fig. 6.12 (c) Nanocarbons shown in 3D feature space represented using principle component approach

Fig. 6.13 Room temperature grown nanocluster carbon is located in the \( \text{sp}^2-\text{sp}^3-\text{H} \) ternary diagram.

The nanocluster carbon is located in the ternary diagram space with the help of PCA plot and the earlier understanding [67] of hybridization type. This model is developed based on the understanding of nanocluster carbon compared to other nanocarbons as well the supporting
classification plot. Here the nanocluster carbon is occupying a region between sp$^2$ and sp$^3$ corners, but predominantly towards sp$^2$ corner.

6.5 Correlation study

Correlation of Raman parameter with dimension: The variation of mean value of the Raman Response is comparable with the Peak values of the Raman response, which is around the G peak (shown in figure 6.14). Variation of nitrogen concentration of order one results in increased Full Width Half Max. (FWHM) and the G peak shifted to left, indicates it has influence on morphology and composition. The left shift of G peak position may indicate the samples as more graphitic. Nitrogen partial pressure has greater influence on bonding ratio (sp$^2$/sp$^3$ ratio). The increase in helium concentration resulted with decrease of FWHM under (nitrogen partial pressure 10$^{-3}$Torr) and also inverse correlation with dimensional variation is observed initially. Nitrogen partial pressure 10$^{-3}$Torr is comparably better for uniform size of clusters. The FWHM (Raman parameter) is inversely proportional to the helium concentration (one of the process parameter) is shown in figure 6.14. The morphological changes of the nanocluster carbon surface property, is influenced by Helium and Nitrogen concentration along with the other deposition parameters. For example shown in figure 6.15, are the SEM images of nanocluster carbon grown at different helium condition shows clusters of different size.

![Fig. 6.14 Change of helium partial pressure and variation of FWHM of Raman and G-peak position of Raman.](image1)

![Fig. 6.15 SEM of nanocluster carbon deposited with helium partial pressure (0.5 and 0.1 Torr respectively) under constant nitrogen.](image2)
The cluster size does not increase linearly. After a particular cluster size, with increasing helium partial pressure it looks like the clusters do not coalesce. Rather the constant bombardment of the ions and neutrals leads to quicker condensation and smaller cluster or even fibrous structures at 50 torr.

The Raman response of these samples has a D peak shoulder. This indicates that the Helium rate change predominantly influences D peak and resulting material expected to be more mixed phase compared to the films which are grown without helium.

6.6 Carbon nanotube diameter estimation

The Raman spectrum of a single-walled carbon nanotube (SWNT) has RBM (Radial Breathing Mode), D-band and G-band peak as follows: a) RBM (100 to 300 cm\(^{-1}\)), vibration at which the nanotube diameter contracts and expands, b) D band (vicinity of 1350 cm\(^{-1}\)) defect-derived peak, c) G-band (vicinity of 1590 cm\(^{-1}\)), in-plane vibration of graphite. The RBM is the one which shows sensitive to the nanotube diameter (D). The diameter of SWNT was calculated from the RBM wavenumber [47]. Its diameter is estimated using following formula: \( D (\text{nm}) = \frac{238}{\omega} \); where D is the diameter of the single wall nanotube; \( \omega = \text{RBM} \). In case of a SWNT the estimated diameter is \( \frac{238}{184} = 1.3 \, (\text{nm}) \).

The discussion on Raman response of nanocarbons are promising, and indicates the Raman spectroscopy as a tool for identifying the nanocarbon (as CNT, nanodiamond or nanocluster carbon) or its properties.

6.7 Nanocluster carbon based device

The electrical, electronic behavior of the nanocluster carbon is studied with conductivity measurement and its suitability for devices and sensor application are studied.

6.7.1 Nanocluster carbon as semiconductor

The conductivity of room temperature grown nanocluster carbon on glass is was measured with coplanar configuration. The ohmic nature was tested with I-V measurement before conductivity measurement. The conductivity measurement is carried from room temperature to 200°C with a fixed voltage (10V), with an interval of 5°C. The conductivity is plotted and it is observed that the conductivity is increasing with the increase in the temperature and
shows semiconducting behavior. All nanocluster carbon films grown at low temperature typically exhibited the semiconducting behavior. Shown in the figure 6.16 is the conductivity result obtained from nanocluster carbon films studied at atmospheric condition (without vacuum condition) and under vacuum.

Fig. 6.16 Conductivity plot of the nanocluster carbon samples studied at ambient condition and at vacuum.

6.7.2 Nanocluster carbon as vacuum nanoelectronic device

Nanocluster carbon grown at room temperature is studied with field emission measurement for understanding its vacuum nanoelectronics behavior. Demonstrated field emission diode configuration experiment set up with anode consisting of patterned phosphorous coated ITO as screen and cathode being a nanocarbon film. The experiment was carried out under a vacuum of about $10^{-6}$ Torr. Shown in figure 6.17(a, b) are the images obtained during the field emission experiment indicates glowing characters (MIT50) at around 2.3 V/µm.

Fig. 6.17 a) Nanocarbon in vacuum diode configuration, (b) Glowing Field emitting display.
CONCLUSION

Studied morphology, dimension and compositional details of a series of nanocarbons include nanodiamond, carbon nanotubes, nanocluster carbon, nanowall, and nanopillar. Reported here is one the first study of room temperature grown nanocluster carbon, based on its morphological, dimensional, and compositional details. The room temperature grown nanocarbon could be of interest for flexible electronics or electronics on glass or plastic. But the nanocarbon film’s structural, compositional and morphological properties are highly dependent on the process parameter such as temperature, pressure, compositional gas ratios, ion energy, type of arc (continuous arc or pulsed arc), arc current and arc voltage.

The morphology, dimensional, compositional and electrical/electronic properties of nanocluster carbon grown at different growth conditions with different growth parameters is reported. The designed image processing algorithm helps in the estimation of cluster diameter, size/dimension and is helpful in the in-situ process technology. Where estimating the dimension, understanding the distribution evaluation of films may be carried out (the details of the distribution of cluster decides the quality of the film as well quantify the number of clusters having the same dimension).

Raman spectroscopy is non-destructive and ideal for investigating physical properties of nanocarbons. This instantaneous method for nanocarbon analysis, is used to study the bonding and compositional nature. The correlation study of SEM and Raman responses helps in automating the production of nanostructures or helps in the evaluation of the nanocarbons.

The conductivity study of nanocluster carbon indicates that they are all semiconductors in nature. Proposed classification of material based on the feature extracted from the Raman response. The PCA based grouping of nanocarbons using Raman response provides three groups such as CNT, nanocluster carbon and nanodiamond. The reported morphological, dimensional and compositional based study would support the in situ molecular level engineering of nanocarbons.

The room temperature grown nanocluster carbon film shown as semiconductor once optimized may be used different applications in large scale electronics, or vacuum nanoelectronics. Once these materials are tested with biocompatibility, then the proposed nanocarbons may be used in the developments of invasive sensors or along with the implants.
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INTERNATIONAL JOURNALS


**INDEXED INTERNATIONAL PUBLICATION**

1. **Niranjana S., B S Satyanarayana , U C Nirjan and Shounak De, “ Quantitative and indirect Qualitative analysis approach for Nanodiamond using SEM images and Raman Response,” IFBME (Springer), Editors: Lim, Chwee Teck; Goh, James C. H., Volume 23, 782-785, ISSN 1680-0737, ISBN 978-3-540-92840-9 (Print) 978-3-540-92841-6 (Online) , 782-785 (Springer link),2009.**

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Title and Organization of the Thesis

Title: Characterization of nanocarbon thin films

The thesis titled “Characterization of Nanocarbon thin films” is organized with the following chapters. The first chapter of the thesis has a brief introduction to carbon as a material for nanotechnology, its importance, motivation to molecular level engineering and its applications are discussed. Chapter two is an overview on series of nanocarbon includes fullerene, carbon nanotubes, grapheme, amorphous carbon, nanocluster carbon, and nanodiamond. These series may have 1-dimensional, 2-dimensional and 3-dimensional nanocarbons having different morphology, composition and dimension. The discussion provides a comprehensive understanding of nanocarbons, its process technologies. The principles of Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM) and Raman Spectroscopy are also reported. The chapter three focuses on the discussions of morphological study and dimension analysis of various nanocarbons. Chapter four is on Raman spectra of various nanocarbons. Discussed Raman response as unique signature and discussed possible correlations of Raman spectra with various process parameters. Also reported principle component analysis (PCA) based classification of nanocarbons including three classes of carbon. The chapter five discusses the electrical/electronic characterization, discovering the nature of nanocluster carbon as semiconductor. The chapter also discusses low temperature grown nanocluster carbon as vacuum nanoelectronic device. The final chapter provides the thesis conclusion.