Effect of Acetylene Rates and Temperature Variations of Iron Nanoparticles in Carbon Nanotubes

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Abstract: In this paper, low pressure chemical vapor deposition device is used to synthesize carbon nanotubes (CNTs) of iron nanoclusters. The physico-chemistry properties of iron nanoclusters obtained of carbon nanotubes with diameter sized between 2~3 nm. The effect of temperature variation was between 650 to 950°C. The gas flow rates of (Argon, 100 standard cubic centimeters per minute (sccm), Hydrogen 50 sccm and (Acetylene, 10, 20, 30 sccm) were applied respectively, for each 20 minutes of the samples. The best obtained result of the CNTs affected by acetylene rates and temperature variations. It was exactly obtained at 650°C and Acetylene rate 20 sccm. Finally; implications of the scanning electron microscopic SEM nanotubes results and their possible applications were discussed using fractal methods.

Keywords: Acetylene rate; temperature variations; carbon nanotubes, Fractal methods.

1. INTRODUCTION

The carbon nanotubes are currently the focus of intensive research due to their unique properties and potential to impact broad research of science and technology [1]. Our study investigate the effect properties of CNTs using fractal methods by classify surface analysis. The most common parameter derived from the fractal analysis is the fractal dimension $D_4$ that is direct geometric implication in relation to other material parameters found in various applications [2]. Materials with modified nanostructures surface have attracted great attention in the last decade because of their numerous applications such as medicine micro channel plate’s technology (MCP) [3], filtration, and hydrogen sensors [4]. There are six basic methods of measuring fractal properties the box counting method, adsorption studies, chord-length measurements, and correlation function measurements, small angle scattering and spectral methods [5]. Such that; the fractal dimension is a measure the roughness of a surface [6]. In previous study was used the change of Pd/AAO membrane structure using fractal approach [7]. For an image with embedded fractal objects (namely the Pd aggregates), the number of covering boxes scales as $N(l) \sim l^{-D}$, where $D$ is fractal dimension [8]. Here, a direct visualization of these microstructures can be done using the concept of surface roughness [9]. Thus, our main contribution in this study is the application of two-parameter generalized based on temperatures changes and gas flow rates Acetylene. The formation process introduced for iron nanoparticles deposit on the Silicon Wafer substrates on Low Pressure Chemical Vapor Deposition System (LPCVD).

2. MATERIALS AND METHODS

In this study the iron nanoparticles (Fe NPs) size were 2~3nm. The purity of (Fe NPs) was 99.99%. The weight of iron and sample holder was 0.108g. The weight of iron was 0.6074g. The iron samples and sample holder were supported by ceramic and putted in LPCVD device, model (CV-6SLX). Experiment conditions for each samples were annealed at 650, 750, 850, 950°C respectively for 20 minutes in a flow of 100 sccm of Argon and 50 sccm of Hydrogen and then LPCVD growth was carried out by the addition of 10~30 sccm of acetylene for constant timing of 20 minutes. Hereby, the surface
morphology for each samples of the carbon nanotubes were studied by scanning electron microscope (SEM, model: JSM–6460 LV).

3. RESULTS AND DISCUSSION

Figure 1 shows the SEM morphologies for carbon nanotubes. During the annealing, the sample temperature was changed with fixed pressure 30torr. The spots increased in nanotubes growth per each sample subjected to time and heating treatments as shown in Figure 1.

![SEM microstructure images result of Fe CNTs surface with sub-particle sizes/A°](image)

Figure 1: SEM microstructure images result of Fe CNTs surface with sub-particle sizes/A°

3.1. Fractal Analysis Method

In this work we used the box counting methos for fractal analysis and The Fe CNTs microstructure images bellowed were analyzed using program harmonic and fractal image analyzer (HarFA 5.1). A digital image \( a [x, y] \) is a box counting described in a 2D discrete space defined by the x-axis representing the possible gray values and the y-axis representing the number of pixels for each gray value. The value is assigned to the integer coordinates \([x, y] \) with \( x = 0, 1, 2, x-1 \) and \( y = 0, 1, 2, y-1 \). So we can get various fractal dimensions from one image [9]. Therefore, In this work each of the SEM image was subdivided roughly into five digital images of [250 x 250] pixels, to cover the sample completely, which gave a raw image space so that the Fe CNTs surface distribution of the sample could be observed in the flat as shown in Figure 3. The Iron Fe nanoparticles with different sizes were deposited using a sequential electroless deposition technique on the pore walls of nonporous Silicon Si wafer Substrate Figure 4.

The parameter estimation for Fe CNTs microstructures images can be carried out by analyzing the spatial data at two different regimes, namely small scales (high frequencies) and large scales (low frequencies) using different techniques of Box counting methods. As mentioned earlier, from the small scales behaviors one can deduce the locally self-similar scaling exponent \( d \) or the fractal dimension \( D_f \) through \( D=3−d/2 \).
Figure 2: Fractal dimension and Box counting Analysis A, B, C, and D

Figure 2 shows the binary images, which contain two gray levels marked by black and white colors; the black represents the Fe nanoparticles (object) and the white the background Si wafer substrate. Following Figure 5 there is a sufficient number of data points of iron nanoparticles, and then we perform a linear regression of the dataset of object (Particle size distributions) and determine the fractal dimension of the Fe CNTs surface \( D_d \) by using the [10] equation :

\[
D_d = \frac{\log \text{ (number of self-similar) (NSP)}}{\log \text{ (magnification factor) (MF)}} \tag{1}
\]

And thus from equation above plotting log number of self-similar Iron CNTs surface (NSP) versus log magnification factor of the self-similar iron CNTs (MF) will produce a straight line with a slope that gives the fractal dimension of iron CNTs \( D_d \). As bellow see Figure 3.
Figure 3: (A, B, C and D) Binarization Steps for each treated Fe CNTs surface microstructure images into Gray Scales with sub-particle sizes /Å" respectively
3.2. Fractal Dimension of Fe CNTs surface calculations

The fractal dimensions of the pores were estimated by Equation 1 using the Image J1.29x analysis program. The Figures 5 plots the logarithm of the number of self-similar iron CNTs nanoparticles (log NSP) versus the logarithm of the magnification factor of self-similar pores (log MF). The relationships are linear with their slopes giving the fractal dimensions (Dd) of the iron CNTs. The values of Dd have been varied.

![Fractal Dimension of Fe CNTs surface](image)

Figure 4: Subdivided samples of Fe CNTs surface 250*250 pixles

3.3. The fractal dimensions for each sample A, B, C, and D

Fractal dimensions of each subdivided samples of microstructure Image iron CNTs surface sub-particle sizes/A° shown as:

![Fractal Dimension Analysis](image)
Figures 5: A, B, C and D Fractal dimensions of each subdivided samples of microstructure Image iron CNTs surface sub-particle sizes/A".

Table 1: Summarized measurements of Fractal dimensions of treated Fe CNTs microstructure surface, subdivided into five samples (sample A.1 to D.1) (Fe CNTs sub-particle sizes/A"

<table>
<thead>
<tr>
<th>Iron CNTs Samples</th>
<th>Fractal Graphic Band Widths BW (n)</th>
<th>Regressions Equations of each Sample</th>
<th>Regressions Equation Values of each Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample A.1</td>
<td>-0.46 7</td>
<td>y = 0.9724X + 7.536</td>
<td>-0.46 7.089</td>
</tr>
<tr>
<td>Sample B.1</td>
<td>-0.42 10</td>
<td>y = 1.4925X + 11.612</td>
<td>-0.42 10.99</td>
</tr>
<tr>
<td>Sample C.1</td>
<td>-0.42 7</td>
<td>y = 1.0403X + 7.822</td>
<td>-0.42 7.39</td>
</tr>
<tr>
<td>Sample D.1</td>
<td>-0.42 6.6</td>
<td>y = 1.3011X + 8.155</td>
<td>-0.42 7.61</td>
</tr>
</tbody>
</table>

Table 2: Analysis the fractal dimension (Dd) of Iron CNTs results of graphic Samples on table 1 of box counting self similar value numbers In(S) and the nanoparticle Dimension Axises values In(n)

<table>
<thead>
<tr>
<th>In(S)</th>
<th>In(n)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-0.46</td>
<td>7.089</td>
</tr>
<tr>
<td>-0.42</td>
<td>10.98</td>
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<tr>
<td>-0.42</td>
<td>7.385</td>
</tr>
<tr>
<td>-0.42</td>
<td>7.609</td>
</tr>
</tbody>
</table>

Figure 6: Iron CNTs Analysis fractal Dimension (Dd) Results of treated graphic samples shown above as a Box counting Self similar numbers In(S) functioned on nanoparticle dimension axises numbers In(n)
3.4. Surface roughness of treated iron CNTs microstructure Samples

The surface roughness of treated iron CNTs microstructure for each Sample a, b, c, and d shown in Figure 7. It is determined the variations of carbon nanotube of formation density and changable parameters of heating and gases flow rates.

Figure 7: Surface Roughness of iron CNTs treated microstructure Sub-particle sizes/Å

4. CONCLUSION

To date, fractal concepts have provided powerful tools for describing different physical properties for nanomaterials. Our analysis revealed from above fig. 5 results as follow a highly multi-walled iron CNTs obtained as a result of Fe Nanoparticles random distribution on Silicon Si Wafer substrate was appeared associated with different particle sizes distributions. The surface roughness of iron CNTs increased with increasing iron sub-particle sizes. There is a Non-linear (Steady) relationships was observed between fractal dimension of iron CNTs with its particle sizes /Å” and fractal analysis on gray-level images of Fe Carbon Nanotubes CNTs Surface has been carried out based on two related concepts, namely self-similarity and long-range dependence. As a result of various temperatures T°C formations affects at CNTs production processes both characteristics of self-similarity and long-range can be deduced from the presence of power scaling laws, but at two opposite extremes of scales. A generic model that couples these two ubiquitous properties is the well-known on bellow graphic, where the scaling behavior is observed over the whole range of scales proportionally.

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REFERENCES


