



A NOVEL FABRICATION OF HIGH GRADE SWCNTS FROM GR (1mm) FOR NANO PROCESSOR IN NANO SUPER COMPUTER, NEMS AND RAM MEMORY DEVICES - A NEW FEASIBILITY STUDY

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ABSTRACT

CNTs act as “future silicon” to bring down the costs of nano, semiconductor, memory devices and their fabrication facility. CNT Field effect transistors, diodes, logic gates, nano mechanical single electron transistor (SET) on off switches in nano sensors (which are faster and more energy efficient), CNT nano processor (central microprocessor), CNT nano supercomputer in molecular computing are prominent applications among them. Also in nanotechnology CNT acts as promising material for CNT ICs, micro chips, NEMS in nano electronics and Non-volatile RAM memory devices. A novel modified AC method – VSA methodology (with KRS or NTFDS theory) was adopted in this present work for the preparation of CNTs from natural organics i.e., GR (1 mm) graphite rods (under specialized materials category). Structural, Compositional, Surface Morphological and Nano structural Characterizations were carried out on harvested products. The effects of optimizations parameters like pH of the various dipping solutions (acidic, basic and neutral), volume of dipping solutions, various types and parts of the materials, various dipping timings, number of annealing and dipping, various annealing temperature, various time of annealing and various dipping solution temperatures on structural, compositional, surface morphological, nano-structural characterizations of materials and on high grade SWCNTs growth with high yield were studied intensively. Inferences from characterizations were derived. Correlation studies between these characterization inferences (such as grain size, purity) and above optimization parameters were carried out with a high light on yield of high grade SWCNTs. Beyond all of these, we have carried out a new feasibility study at first time, which comprises the possible usage of precursor organic carbon sources i.e., GR (1 mm) graphite rods (under specialized materials category) for high grade SWCNTs (with inner diameter approximately 1.6 Angstrom unit) with high yield via a low cost technique and methodology as value in commercial efforts.

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INTRODUCTION

The present work showed that SWCNTs (with inner diameter approximately 1.6 Angstrom unit) obtained by open air annealing and cooling of GR (1 mm) graphite rods (under specialized materials category). The present work made a novel break through via plays an alternative method to conventional, traditional methods such as arc discharge, laser ablation, metal catalysts, pyrolysis, and varieties of CVD methods and also removes the usage of synthetic chemicals. The modified AC method [Zhenhui Kang *et al*, 33] – VSA methodology (with KRS or NTFDS theory) was adopted in this present work for the preparation of CNTs from natural organics. This work based on the fabrication of SWCNTs via a novel modified methodology from already existed AC method for MWCNTs (Zhenhui Kang *et al*, 5).

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The entire steps such as Precursor materials selection with cleaning, Annealing, Sudden cooling, Interaction between red hot natural organic carbon resource materials and Dipping solutions (DS) [including Nano Thermo Fluid Dynamics (NTFDS) and Nano-drilling process] involved in this process were explained in detail. The characterizations performed on outcome products were internationally accepted, standard methods as followed by field specialists through out the world [1-33].

Experimental Details

Materials i.e., leaves of GR (1 mm) graphite rods (under specialized materials category) collected from nature, cleaned in water and dried in open air. These materials were used without further purification. Then the materials were air annealed (upto red hot) in a muffle furnace at various temperatures viz., 600^oC and 800^oC for various time of heating viz., 1minute - 5 minutes. After that they were immediately

dipped into various types of solutions viz., Sodium hydroxide (NaOH), Hydrogen peroxide (H₂O₂), Nitric Acid (HNO₃), Sulphuric Acid (H₂SO₄), Hydrochloric Acid (HCl), Mineral Water (MW), Salt Water (SW), Double Distilled Water (D2W), Ice Water (Ice W), Hot Water (HW), Pure Water (PW), Ice water mixed double distilled water (IceW+D2W) and Hot water mixed double distilled water (HW+D2W) solution for various solution temperature ranging from 0°C to 100°C, for various time of dipping viz., 30 seconds, 45 seconds, 60 seconds, 75 seconds, 90 seconds and 120 seconds. The final samples were dried in open air at room temperature for 5 hours and then packed for characterization with mentioning synthesis conditions.

The above process was optimized with 11 Physical parameters viz., 1. Nature (pH) of the dipping solutions (acidic, basic and neutral), and 2. Volume of Dipping Solution ranges from 0.5 ml, 1.0 ml, 1.5 ml, 2.0 ml and 2.5 ml, 3. Various types of carbon source materials: Conventional: varieties of plants, trees: Specialized: GR (1 mm) graphite rods (under specialized materials category), 4. Various Parts of the materials: leaves, stems., 5. Various Dipping solutions: Sodium hydroxide (NaOH), Hydrogen peroxide (H₂O₂), Nitric Acid (HNO₃), Sulphuric Acid (H₂SO₄), Hydrochloric Acid (HCl), Mineral Water (MW), Salt Water (SW), Double Distilled Water (D2W), Ice Water (Ice W), Hot Water (HW), Pure Water (PW), Ice water mixed double distilled water (IceW+D2W) and Hot water mixed double distilled water (HW+D2W), 6. Various Dipping Timings: 30 seconds, 45 seconds, 60 seconds, 75 seconds, 90 seconds and 120 seconds, 7. Number of Dipping: 1, 8. Various Annealing Temperatures: 600°C and 800°C, 9. Various Time of Annealing: 1 minute to 5 minutes., 10. Various Dipping Solution temperatures: 0°C to 100°C., 11. Number of Annealing: 1

RESULTS AND DISCUSSION

Characterization of SWCNTs

Structural Characterization

GR (1 mm) graphite rods

Fig. [3.1.1] explained the XRD studies on GR (1 mm) annealed at 800°C (2 mints) dipped in Distilled water (volume: 2 ml) for 120 seconds. The first order [C (002)] and second order [C (004)] diffraction peaks showed the single crystalline nature of the material. Actually during GR (1 mm) graphite rods preparation process, a strong covalent bonding formation takes place inside the carbon matrix, which finally leads to lattice formation of single crystalline nature of the material.

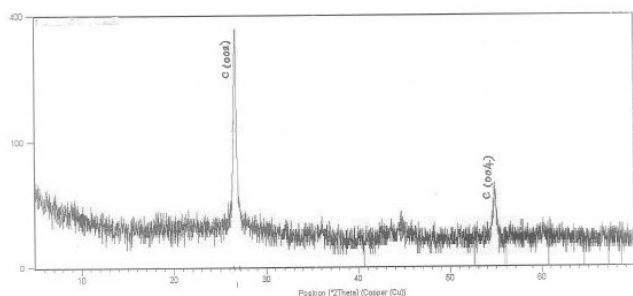


Fig [3.1.1] XRD studies on GR (1 mm) graphite rods (under specialized materials category) annealed at 800°C (2 mints) dipped in Distilled water (volume: 2 ml) for 120 seconds. The

first order [C (002)] and second order [C (004)] diffraction peaks shows the single crystalline nature of the material.

Compositional Characterization

GR (1 mm) graphite rods

Fig [3.2.1] EDAX studies on GR (1 mm) annealed at 800°C (2 mints) dipped in Sulphuric acid (H₂SO₄) (volume: 2.5 ml) for 60 seconds. The EDAX spectrums showed compositional elements present in the GR (1 mm) graphite rods. Presence of rich Carbon atoms (1st prominent peak in EDAX spectrum) confirmed that the formed tubes are made up of carbon atoms. Which was authentically shows the formation of CNTs. Due to high temperature annealing, dehydration takes place, H₂ atom was removed. Which was evidentially shown from the EDAX spectrum (i.e., H₂ not found in the EDAX spectrum) Presence of O₂ atom in the EDAX spectrum evidentially shows that oxygen was injected / feed up during annealing. Presence of (Silicon) Si, (Oxygen) O shows that the organic source (from which the graphite rods were derived) was enriched with them. Se, Na, Cl, K, Ca and Fe proved that the organic source (from which the graphite rods were derived) was enriched with them

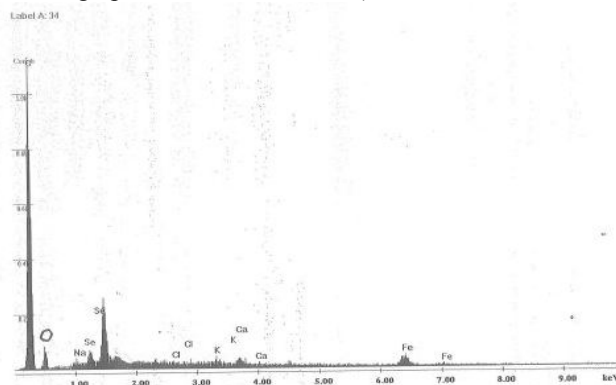


Fig.[3.2.1]. EDAX studies on GR (1 mm) graphite rods (under specialized materials category) annealed at 800°C (2 mints) dipped in Sulphuric acid (H₂SO₄) (volume: 2.5 ml) for 60 seconds.

Surface Morphological Characterization

SEM studies were carried out with a JEOL JSM-840 operated at 20 KV.

GR (1 mm) graphite rods

In Fig [3.3.1], Surface morphology (SEM) of 600°C (3 minutes) annealed red-hot GR (1 mm) dipped in sodium hydroxide (NaOH) solution (volume: 1.5 ml) for 75 seconds shows in-homogeneous scattering of hexagonal carbon matrices due to sudden cooling.

In Fig. [3.3.2], Surface morphology (SEM) of 800°C (3 minutes) annealed red hot GR (1 mm) dipped in Double Distilled Water (volume: 1.5 ml) for 60 seconds shows clear picture of in-homogeneous origination, scattering and coagulation of hexagonal carbon matrices (close up view) from core species due to sudden cooling finally leading to the budding / initial formation of tubular SWCNTs.

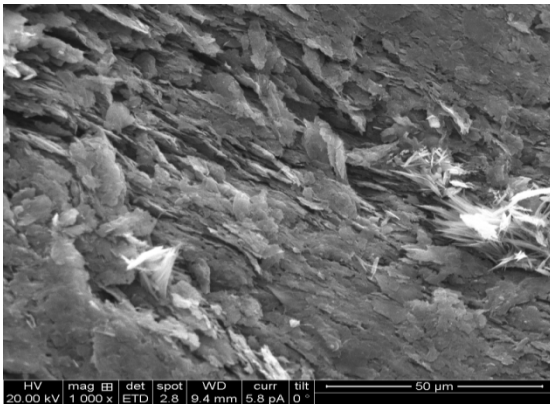


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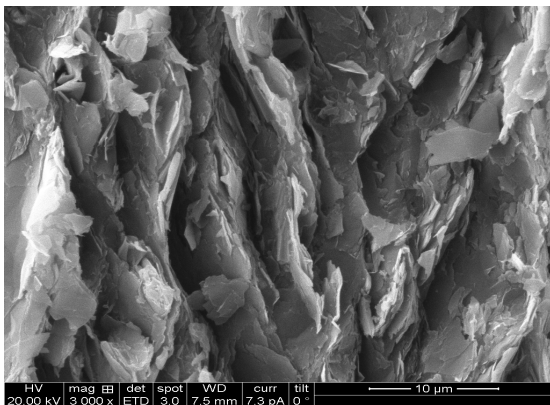


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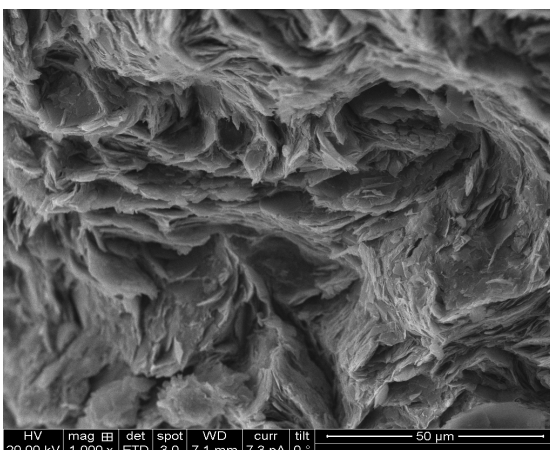


Fig. [3.3.3]. Surface morphology (SEM) of 600°C (3 minutes) annealed red hot GR (1 mm) dipped in Double Distilled Water (volume: 1.5 ml) for 75 seconds shows clear picture of homogeneous distribution of hexagonal carbon matrices from core species due to vortex motion involved in the conduction (heat transfer flow) and convection (heat and mass transfer

flow) process in allied with a novel thermodynamic (CO₂ evolution and it's flow of thrust) process called NFTDS (Nano Fluid Thermo Dynamics). All these were happened because of sudden cooling of red hot GR (1 mm) in Double Distilled water, [i.e., a dipping solution of comparatively low temperature with pH=7],, finally leading to the budding / initial formation of tubular SWCNTs.

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In Fig. [3.3.4], Surface morphology (SEM) of 800°C (1 minute) annealed red hot GR (1 mm) dipped in double distilled water (volume: 0.5 ml) for 120 seconds, emphasized the clear evidence for the initial growth stage of super matured horizontal SWCNTs – tubular structure (as rolled graphene layer which composed of hexagonal carbon matrix networks) (longitudinal sectional view) as individual fibres exhibit a middle hallow empty space (an eagle insight view). In Fig. [3.3.5], Surface morphology (SEM) of 800°C (3 minutes) annealed red hot GR (1 mm) dipped in double distilled water (volume: 1.5 ml) for 60 seconds, emphasized the clear evidence for the intermediate growth stage of super matured vertical and horizontal SWCNTs – tubular structure (as rolled graphene layer which composed of hexagonal carbon matrix networks) (longitudinal and cross sectional view) as individual fibres exhibit a middle hallow empty space (an eagle insight view).

In Fig. [3.3.6], Surface morphology (SEM) of 800°C (4 minutes) annealed red hot GR (1 mm) dipped in double distilled water (volume: 2.0 ml) for 45 seconds, emphasized the clear evidence for the intermediate growth stage of super matured inclined horizontal SWCNTs – tubular structure (as rolled graphene layer which composed of hexagonal carbon matrix networks) (longitudinal and cross sectional view) as individual fibres exhibit a middle hallow empty space (an eagle insight view). In Fig. [3.3.7], Surface morphology (SEM) of 800°C (5 minutes) annealed red hot GR (1 mm) dipped in double distilled water (volume: 2.5 ml) for 30 seconds, emphasized the clear evidence for the final stage of super matured vertical and horizontal SWCNTs – tubular structure (as rolled graphene layer which composed of hexagonal carbon matrix networks) (longitudinal and cross sectional view) as individual fibres exhibit a

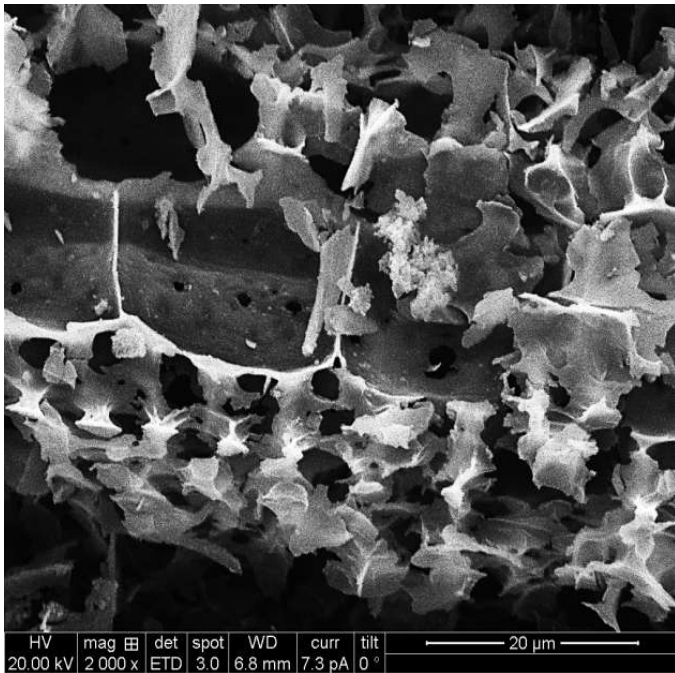


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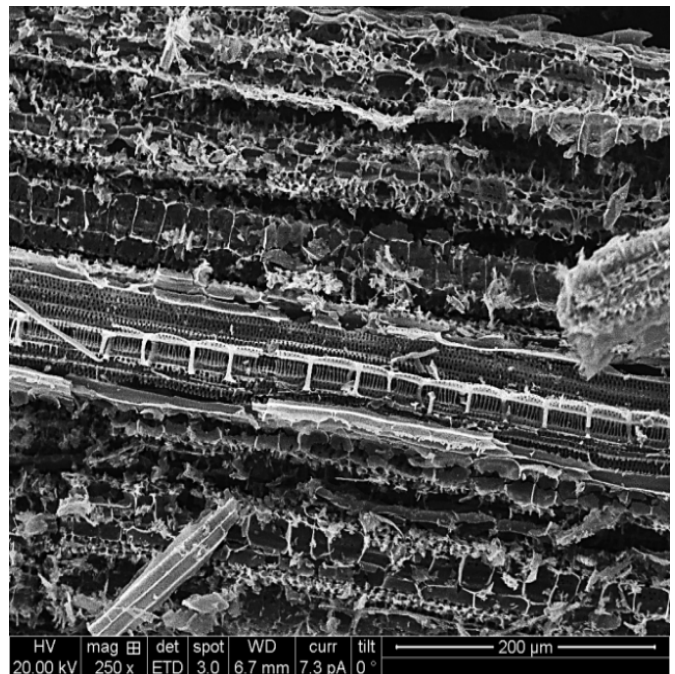


Fig. [3.3.5]. Surface morphology (SEM) of 800°C (3 minutes) annealed red hot GR (1 mm) dipped in double distilled water (volume: 1.5 ml) for 60 seconds, emphasized the clear evidence for the intermediate growth stage of super matured vertical and horizontal SWCNTs – tubular structure (as rolled graphene layer which composed of hexagonal carbon matrix networks) (longitudinal and cross sectional view) as individual fibres exhibit a middle hallow empty space (an eagle insight view).

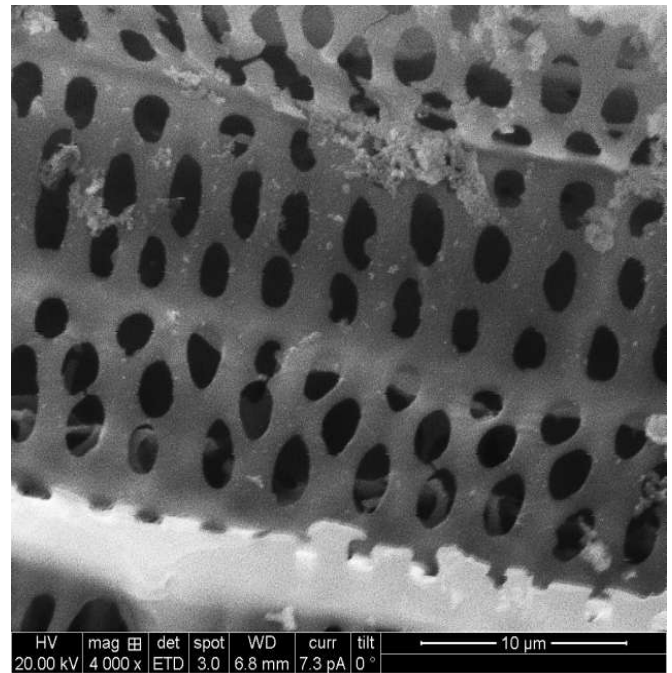


Fig. [3.3.6]. Surface morphology (SEM) of 800°C (4 minutes) annealed red hot GR (1 mm) dipped in double distilled water (volume: 2.0 ml) for 45 seconds, emphasized the clear evidence for the intermediate growth stage of super matured inclined horizontal SWCNTs – tubular structure (as rolled graphene layer which composed of hexagonal carbon matrix networks) (longitudinal and cross sectional view) as individual fibres exhibit a middle hallow empty space (an eagle insight view).

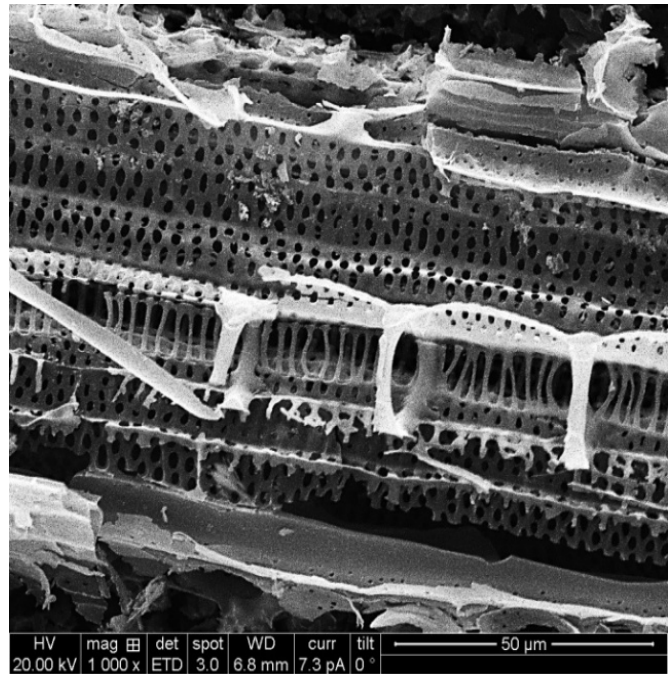


Fig. [3.3.7]. Surface morphology (SEM) of 800°C (5 minutes) annealed red hot GR (1 mm) dipped in double distilled water (volume: 2.5 ml) for 30 seconds, emphasized the clear evidence for the final stage of super matured vertical and horizontal SWCNTs – tubular structure (as rolled graphene layer which composed of hexagonal carbon matrix networks) (longitudinal and cross sectional view) as individual fibres exhibit a middle hallow empty space (an eagle insight view).

middle hallow empty space (an eagle insight view). In Fig. [3.3.8], Surface morphology (SEM) of 600°C (4 minutes) annealed red hot GR (1 mm) dipped in Hydrochloric (Hcl) acid (volume: 1.0 ml) for 75 seconds shows close up view of the above image which explains intermediate stage of in-homogeneous distribution of hexagonal carbon matrices from core species.

In Fig. [3.3.9], Surface morphology (SEM) of 800°C (5 minutes) annealed red hot GR (1 mm) dipped in Hydrochloric (Hcl) acid (volume: 2.5 ml) for 30 seconds shows close up view of the super matured final pattern of perfectly aligned SWCNTs (i.e., honey comb structure composed of straws) as an evidential proof of a novel thermodynamic (CO₂ evolution and it's flow of thrust) process called NFTDS (Nano Fluid Thermo Dynamics). All these were happened because of sudden cooling of red hot GR (1 mm) in Hydrochloric (Hcl) acid, [i.e., a dipping solution of comparatively low temperature with pH < 7], finally leading to the formation of tubular SWCNTs. In Fig. [3.3.10], Surface morphology (SEM) of 800°C (4 minutes) annealed red hot GR (1 mm) dipped in Hydrochloric (Hcl) acid (volume: 2.5 ml) for 45 seconds, shows semi-matured stage of [eagle view] of both vertical and horizontal SW-CNTs. (cross-sectional view as well as longitudinal view).

In Fig. [3.3.11], Surface morphology (SEM) of 800°C (4 minutes) annealed red hot GR (1 mm) dipped in Hydrochloric (Hcl) acid (volume: 2.5 ml) for 45 seconds, shows a damaged portion of the semi-matured stage of [eagle view] of both vertical and horizontal SW-CNTs. (cross-sectional view as well as longitudinal view). In Fig. [3.3.12], Surface morphology (SEM) of 800°C (3 minutes) annealed red hot GR (1 mm) dipped in Hydrochloric (Hcl) acid (volume: 1.5 ml) for 60 seconds, shows a damaged portion of the semi-matured stage of [eagle view] of both vertical and horizontal SW-CNTs. (cross-sectional view as well as longitudinal view). In Fig. [3.3.13], Surface morphology (SEM) of 800°C (5 minutes) annealed red hot GR (1 mm) dipped in Hydrochloric (Hcl) acid (volume: 2.5 ml) for 60 seconds, shows a perfectly grown damaged portion of the semi-matured stage of [eagle view] of both vertical and horizontal (closed type) SW-CNTs. (cross-sectional view as well as longitudinal view).

In Fig. [3.3.14], Surface morphology (SEM) of 800°C (4 minutes) annealed red hot GR (1 mm) dipped in sulphuric acid (H₂SO₄) solution (volume: 2.0 ml) for 45 seconds, shows inclined horizontal SWCNTs – tubular structure as closed individual fibres exhibit a middle hallow empty space (longitudinal view).

(volume: 1.0 ml) for 75 seconds shows close up view of the above image which explains intermediate stage of in-homogeneous distribution of hexagonal carbon matrices from core species.

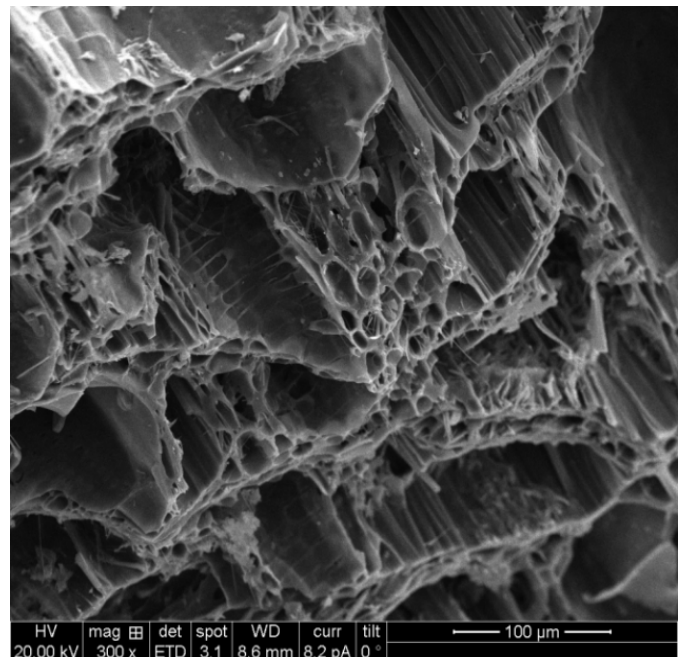


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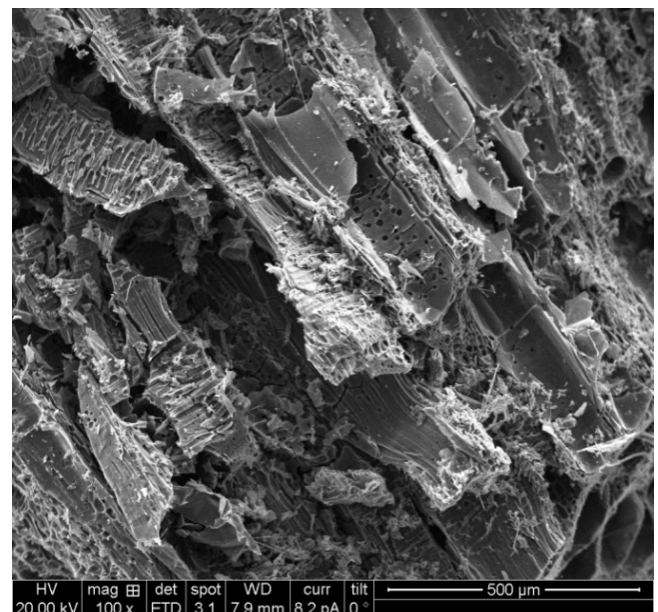


Fig. [3.3.10]. Surface morphology (SEM) of 800°C (4 minutes) annealed red hot GR (1 mm) dipped in Hydrochloric (Hcl) acid (volume: 2.5 ml) for 45 seconds, shows semi-matured stage of

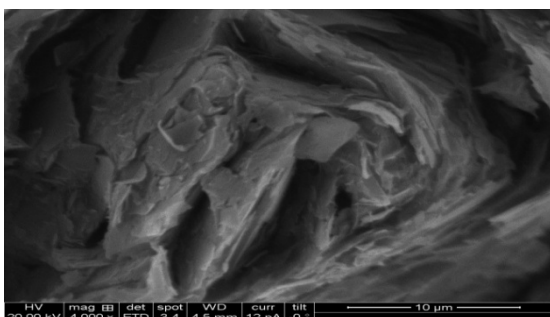


Fig. [3.3.8]. Surface morphology (SEM) of 600°C (4 minutes) annealed red hot GR (1 mm) dipped in Hydrochloric (Hcl) acid

[eagle view] of both vertical and horizontal SW-CNTs. (cross-sectional view as well as longitudinal view).

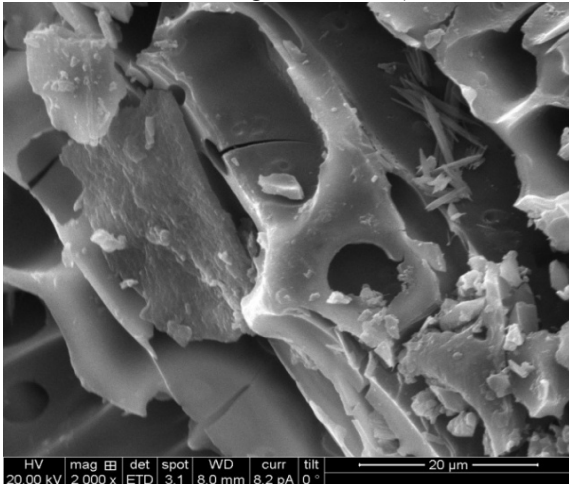


Fig. [3.3.11]. Surface morphology (SEM) of 800°C (4 minutes) annealed red hot GR (1 mm) dipped in Hydrochloric (HCl) acid (volume: 2.5 ml) for 45 seconds, shows a damaged portion of the semi-matured stage of [eagle view] of both vertical and horizontal SW-CNTs. (cross-sectional view as well as longitudinal view).

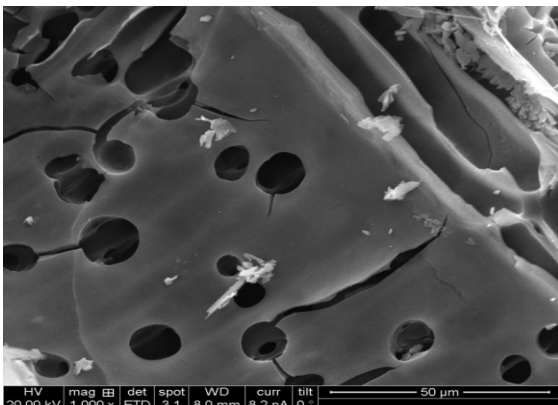


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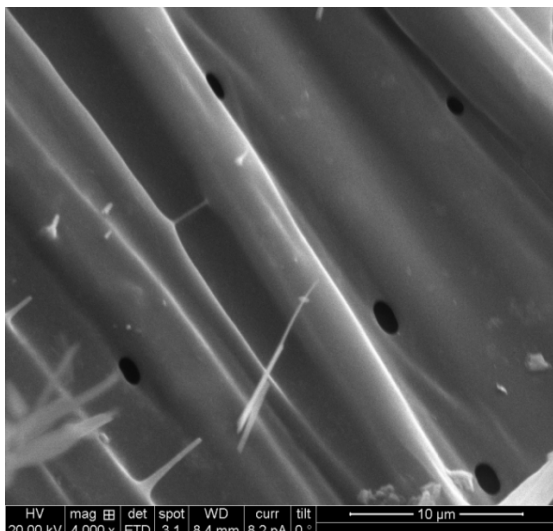


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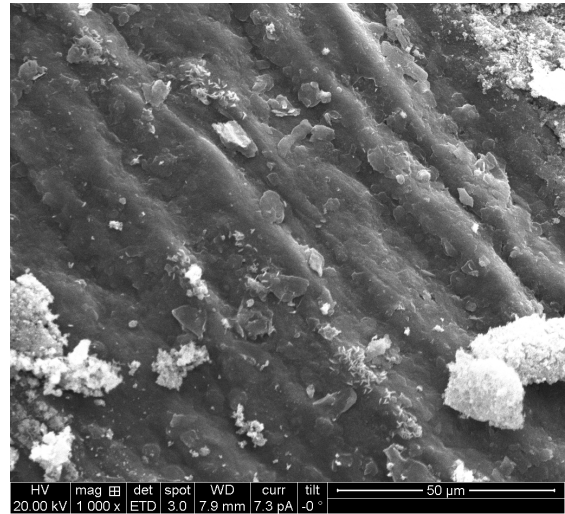


Fig. [3.3.14]. Surface morphology (SEM) of 800°C (4 minutes) annealed red hot GR (1 mm) dipped in sulphuric acid (H₂SO₄) solution (volume: 2.0 ml) for 45 seconds, shows inclined horizontal SWCNTs – tubular structure as closed individual fibres exhibit a middle hallow empty space (longitudinal view).

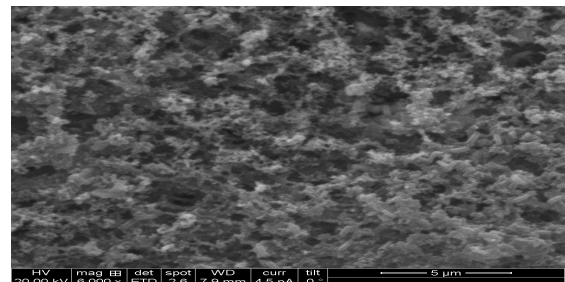


Fig. [3.3.15]. Surface morphology (SEM) of 800°C (3 minutes) annealed red hot GR (1 mm) dipped in sulphuric acid (H₂SO₄) solution (volume: 2.0 ml) for 60 minutes, shows homogeneous distribution, scattering and coagulation of hexagonal carbon matrices (basic building blocks of graphene layer). Due to heavy explosion during sudden cooling, patterns of sea algae (composed of sugar crystals like grains) were obtained.

In Fig. [3.3.15], Surface morphology (SEM) of 800°C (3 minutes) annealed red hot GR (1 mm) dipped in sulphuric acid (H₂SO₄) solution (volume: 2.0 ml) for 60 minutes, shows homogeneous distribution, scattering and coagulation of hexagonal carbon matrices (basic building blocks of graphene layer). Due to heavy explosion during sudden cooling, patterns of sea algae (composed of sugar crystals like grains) were obtained.

Nanostructural Characterization

HRTEM studies were carried out with a JEOL JSM-840 operated at 20 KV.

GR (1 mm) graphite rods

In Fig. [3.4.1], HRTEM image shows eagle view of super matured, perfectly aligned SWCNTs (with inner diameter

approximately 1.6 Angstrom unit) grown from nano scattering of hexagonal carbon matrix (building blocks) when 800⁰C red-hot GR (1 mm) when dipping in Double Distilled Water for 120 Sec. In Fig. [3.4.2], HRTEM image shows eagle view of super matured, perfectly aligned SWCNTs grown from nano scattering of hexagonal carbon matrix (building blocks) when 800⁰C red-hot GR (1 mm) when dipping in Double Distilled Water for 90 Sec. In Fig. [3.4.3], HRTEM image shows eagle view of super matured, perfectly aligned SWCNTs (with inner diameter approximately 1.6 Angstrom unit) grown from nano scattering of hexagonal carbon matrix (building blocks) when 800⁰C red-hot GR (1 mm) when dipping in Double Distilled Water for 75 Sec. In Fig. [3.4.4], HRTEM image shows eagle view of super matured, perfectly aligned SWCNTs grown from nano scattering of hexagonal carbon matrix (building blocks) when 800⁰C red-hot GR (1 mm) when dipping in Double Distilled Water for 60 Sec.

In Fig. [3.4.5], HRTEM image shows eagle view of super matured, perfectly aligned SWCNTs grown from nano scattering of hexagonal carbon matrix (building blocks) when 800⁰C red-hot GR (1 mm) when dipping in Double Distilled Water for 30 Sec.

The effects of optimizations parameters like pH of the various dipping solutions (acidic, basic and neutral), volume of dipping solutions, various types and parts of the materials, various dipping timings, number of annealing and dipping, various annealing temperature, various time of annealing and various dipping solution temperatures on structural, compositional, surface morphological, nano-structural characterizations of materials and on high grade SWCNTs (with inner diameter approximately 1.6 Angstrom unit) growth with high yield were studied intensively. Parameters, as inferences from above characterizations were calculated, tabulated and graphically emphasized. Correlation studies between these characterization inferences (such as grain size, purity) and optimization parameters were carried out with a high light on yield of high grade SWCNTs. Beyond all of these, we have carried out a new feasibility study at first time, which comprises the possible usage of precursor organic carbon source i.e., GR (1 mm) graphite rods for high grade SWCNTs with high yield via a low cost technique and methodology as value in commercial efforts.

CONCLUSION

In this over all study, Graphite Rod i.e., GR (1 mm) plays as a most optimum material for high grade SWCNTs growth. Generally, we may understand that specialized materials play as most optimum materials than fibrous and conventional materials. Also any Dipping Solution (of Volume: 2.5 ml) having pH=7, i.e., neutral solution (normally maintained at room.temp.,RT) (with 30 Seconds as optimum time of dipping) act as an optimum medium which provides suitable environment for high grade, large quantity SWCNTs growth. Similarly 800⁰C (having annealing time: 3 minutes) provides suitable background thermal history for high grade, large quantity SWCNTs growth based on single time annealing and dipping process. High purity precursor material yield high grade SWCNTs.

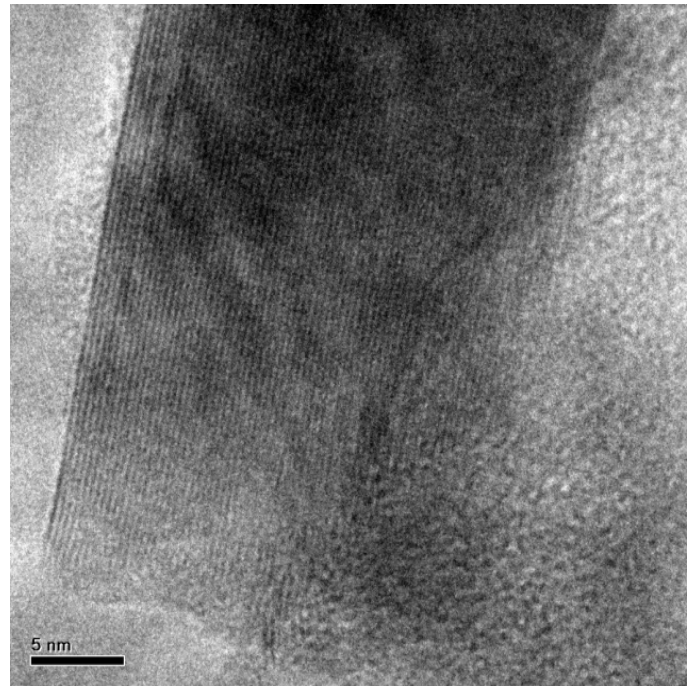


Fig. [3.4.1] HRTEM image shows eagle view of super matured, perfectly aligned SWCNTs (with inner diameter approximately 1.6 Angstrom unit) grown from nano scattering of hexagonal carbon matrix (building blocks) when 800⁰C red-hot GR (1 mm) when dipping in Double Distilled Water for 120 Sec.

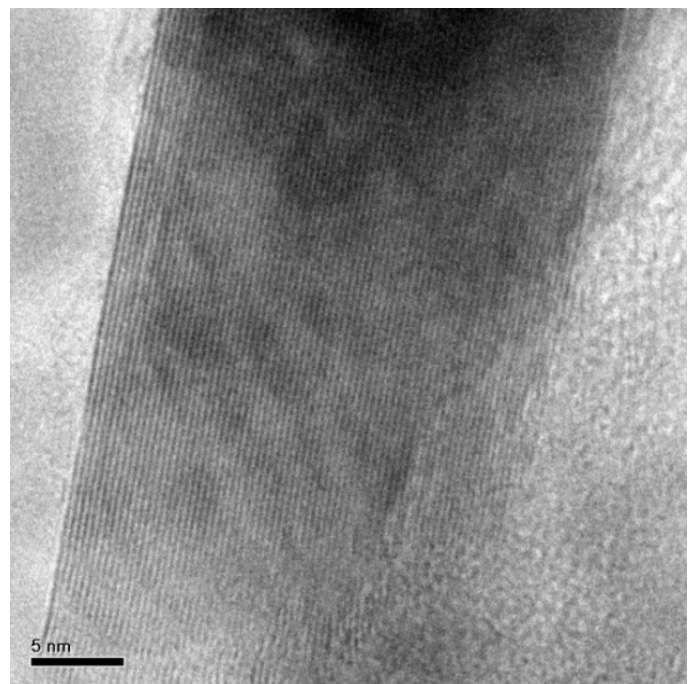


Fig. [3.4.2] HRTEM image shows eagle view of super matured, perfectly aligned SWCNTs (with inner diameter approximately 1.6 Angstrom unit) grown from nano scattering of hexagonal carbon matrix (building blocks) when 800⁰C red-hot GR (1 mm) when dipping in Double Distilled Water for 90 Sec.

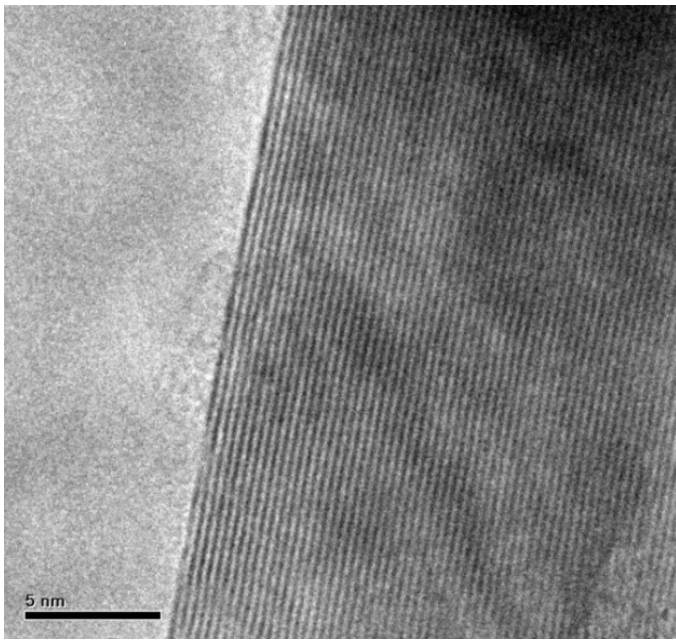


Fig. [3.4.3] HRTEM image shows eagle view of super matured, perfectly aligned SWCNTs (with inner diameter approximately 1.6 Angstrom unit) grown from nano scattering of hexagonal carbon matrix (building blocks) when 800⁰C red-hot GR (1 mm) when dipping in Double Distilled Water for 75 Sec.

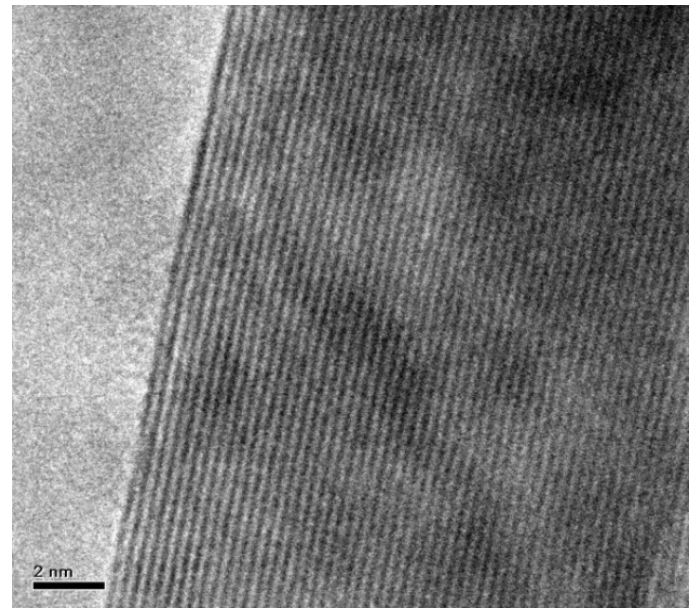


Fig. [3.4.5] HRTEM image shows eagle view of super matured, perfectly aligned SWCNTs (with inner diameter approximately 1.6 Angstrom unit) grown from nano scattering of hexagonal carbon matrix (building blocks) when 800⁰C red-hot GR (1 mm) when dipping in Double Distilled Water for 30 Sec.

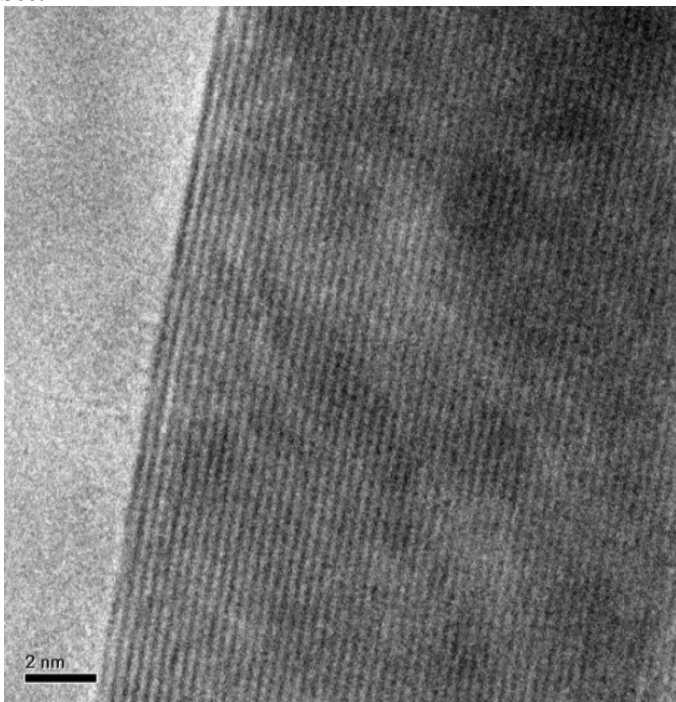


Fig. [3.4.4] HRTEM image shows eagle view of super matured, perfectly aligned SWCNTs (with inner diameter approximately 1.6 Angstrom unit) grown from nano scattering of hexagonal carbon matrix (building blocks) when 800⁰C red-hot GR (1 mm) when dipping in Double Distilled Water for 60 Sec.

GR (1 mm) graphite rods

Fig [4.1] confirmed the Variation of Quantity of SWCNTs formation (%) with respect to dipping solution with a high light on Quality grades of Synthesised SWCNTs (with inner diameter approximately 1.6 Angstrom unit) from GR (1mm) graphite rods. Also explained that neutral solution (normally maintained at room.temp. RT) act as an optimum medium which provides suitable environment for high grade, large quantity SWCNTs growth in GR (1 mm) graphite rods. All of these works have value in nanotechnology, nano-materials processing and device fabrication efforts either as a technical or scientific basis, also as a contribution to the present day state of the art of nano-electronics : nano processor, nano supercomputer, nano ICs, NEMS and Non-volatile RAM memory devices.

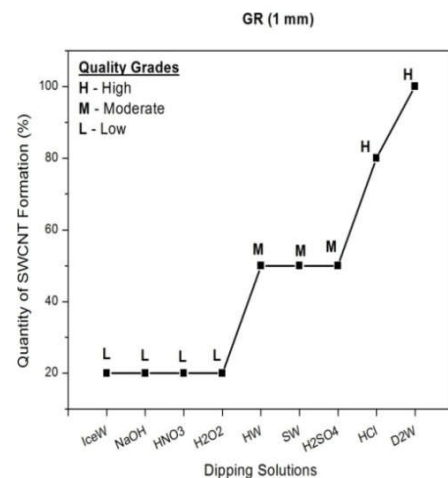


Fig. [4.1] Variation of Quantity of SWCNTs formation (%) with respect to Dipping solution with a high light on Quality grades of Synthesised SWCNTs from GR (1mm).

This work based on the fabrication of SWCNTs via a novel modified methodology from already existed AC method for MWCNTs (Zhenhui Kang *et al*, 5). Also to the best of our knowledge this may be the first time that we got natural SWCNTs (with inner diameter approximately 1.6 Angstrom unit) for nDDS.

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