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APPENDICES

APPENDIX A

The application of carbon nanoparticles

A. Carbon nanotubes (CNTs)

Carbon nanotubes can be considered as elongated fullerene. It could be noted that there are 6 pentagons in each end of tube so that a hexagonal graphene sheet is able to close. Two types of CNTs can be categorized if considering the types of CNTs wall. One is single-walled carbon nanotubes (SWCNTs). Another is multi-walled carbon nanotubes (MWCNTs). Interestingly, these nested tubes exhibited interlayer spacing of 3.4 A°, a value that is slightly greater than that of graphite (3.35 A°).

Many exotic structures of fullerenes exist: regular spheres, cones, tubes and also more complicated and strange shapes. Here we will describe some of the most important and best-known structures. Single Walled Carbon nanotubes (SWNT) can be considered as long wrapped graphene sheets. As stated before, nanotubes generally have a length to diameter ratio of about 1000 so they can be considered as nearly one-dimensional structures.

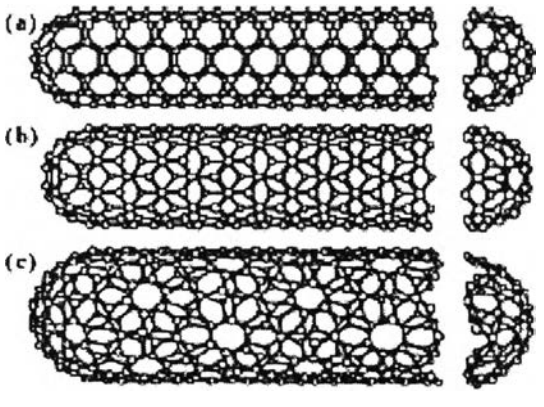


Figure A.1 Some SWNTs with different chiralities. The difference in structure is easily show at the open end of the tubes. a) armchair structure b) zigzag structure c) chiral structure

More detailed, a SWNT consists of two separate regions with different physical and chemical properties. The first is the sidewall of the tube and the second is the end cap of the tube. The end cap structure is similar to or derived from a smaller

fullerene, such as C_{60}. C-atoms placed in hexagons and pentagons form the end cap structures. It can be easily derived from Euler's theorem that twelve pentagons are needed in order to obtain a closed cage structure which consists of only pentagons and hexagons. The combination of a pentagon and five surrounding hexagons results in the desired curvature of the surface to enclose a volume.

A second rule is the isolated pentagon rule that states that the distance between pentagons on the fullerene shell is maximized in order to obtain a minimal local curvature and surface stress, resulting in a more stable structure. The smallest stable structure that can be made this way is C_{60} the one just larger is C_{70} and so on.

Another property is that all fullerenes are composed of an even number of C-atoms because adding one hexagon to an existing structure means adding two C-atoms. The other structure of which a SWNT is composed is a cylinder. It is generated when a graphene sheet of a certain size that is wrapped in a certain direction. As the result is cylinder symmetric we can only roll in a discreet set of directions in order to form a closed cylinder (Figure A.2).

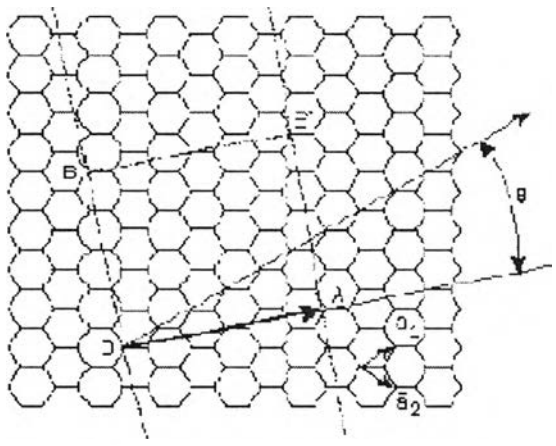


Figure A.2 Vector $\underline{OA}$ is called the chiral vector. It can be defined by the vector $\underline{C_h} = n\underline{a_1} + m\underline{a_2}$ and the chiral angle with the zigzag axis. Vectors a_1 and a_2 are the lattice vectors.

Two atoms in the graphene sheet are chosen, one of which servers the role as origin. The sheet is rolled until the two atoms coincide. The vector pointing from the first atom towards the other is called the chiral vector and its length is equal to the circumference of the nanotube. (Figure A.1) The direction of the nanotube axis is perpendicular to the chiral vector. SWNTs with different chiral vectors have dissimilar properties such as optical activity, mechanical strength and electrical conductivity.

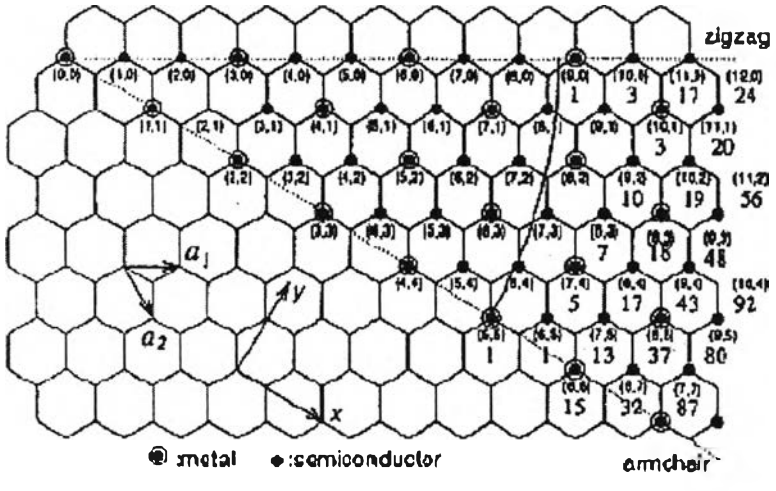


Figure A.3 All possible structures of SWNTs can be formed from chiral vectors lying in the range given by this figure. (n,m) with n,m integer and $m \leq n$ or $\theta < 30°$.

After ideal structures without flaws, we discuss the possible desirable or undesirable defects. Deformations, such as bends and nanotube junctions, are introduced by replacing a hexagon with a heptagon or pentagon. Deformations can be inward or outward and, among others, electrical properties are seriously changed by these deformations. Another class of defects is caused by impurities that are built in during or after the nanotube growth process; Compounds that can be incorporated into the structure are for example catalyst particles. Multi Walled Nanotubes (MWNT) can

be considered as a collection of concentric SWNTs with different diameters. The length and diameter of these structures differ a lot from those of SWNTs and, of course, their properties are also very different. (Figure 3.2.4)

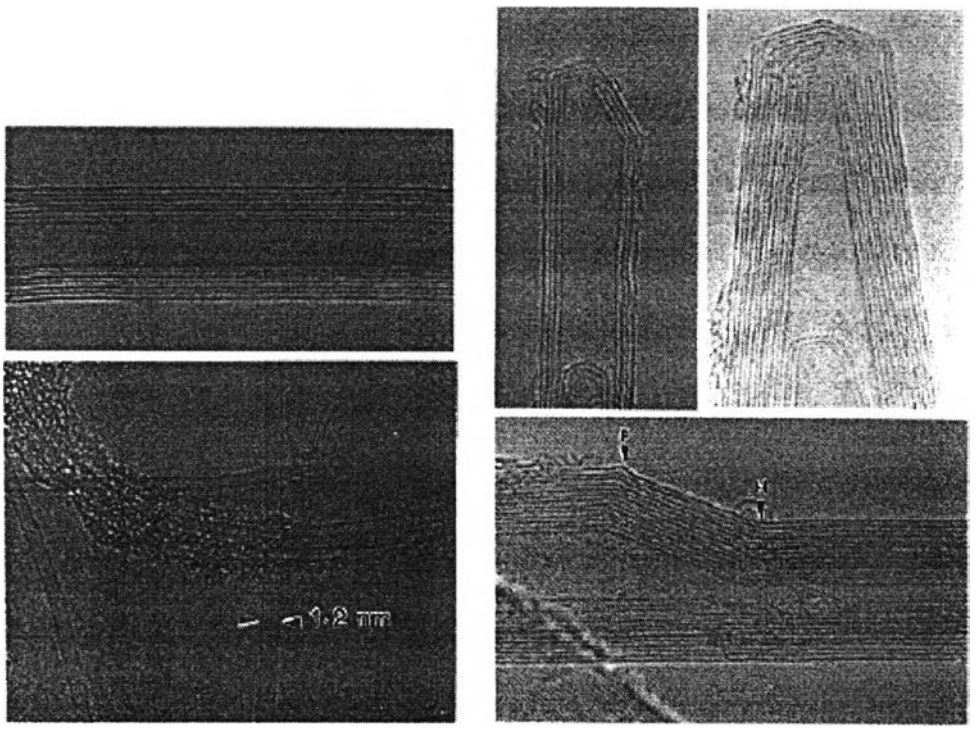


Figure A.4 Different structures of MWNTs.

Top-left: cross-section of a MWNT the different walls are obvious, they are separated by 0.34 nm. Rotation around the symmetry axis gives us the MWNT.

Top-right: Symmetrical or non-symmetrical cone shaped end caps of MWNTs.

Bottom-left: A SWNT with a diameter of 1,2 nm and a bundle of SWNTs covered with amorphous carbon.

Bottom-right: A MWNT with defects. In point P a pentagon defect and in point H a heptagon defect.6

In Figure 3.2.4 carbon cones are also shown. It can be considered as a gradual transition from a large diameter to a smaller one without defects in the wall of the cone but with fewer pentagons in the end cap.

A.1.1 Special properties of carbon nanotubes

Electronic, molecular and structural properties of carbon nanotubes are determined to a large extent by their nearly one dimensional structure. The most important properties of CNTs and their molecular background are stated below.

A.1.2 Chemical reactivity The chemical reactivity of a CNT is, compared with a graphene sheet, enhanced as a direct result of the curvature of the CNT surface. Carbon nanotube reactivity is directly related to the pi-orbital mismatch caused by an increased curvature. Therefore, a distinction must be made between the sidewall and the end caps of a nanotube. For the same reason, a smaller nanotube diameter results in increased reactivity. Covalent chemical modification of either sidewalls or end caps has shown to be possible. For example, the solubility of CNTs in different solvents can be controlled this way. Though, direct investigation of chemical modifications on nanotube behaviour is difficult as the crude nanotube samples are still not pure enough.

A1.1.3 Electrical conductivity Depending on their chiral vector, carbon nanotubes with a small diameter are either semi-conducting or metallic. The differences in conducting properties are caused by the molecular structure that results in a different band structure and thus a different band gap. The differences in conductivity can easily be derived from the graphene sheet properties. It was shown that a (n,m) nanotube is metallic as accounts that: $n=m$ or $(n-m) = 3i$, where i is an integer and n and m are defining the nanotube. The resistance to conduction is

determined by quantum mechanical aspects and was proved to be independent of the nanotube length.

A.1.4 Optical activity Theoretical studies have revealed that the optical activity of chiral nanotubes disappears if the nanotubes become larger. Therefore, it is expected that other physical properties are influenced by these parameters too. Use of the optical activity might result in optical devices in which CNTs play an important role.

A1.5 Mechanical strength Carbon nanotubes have a very large Young modulus in their axial direction. The nanotube as a whole is very flexible because of the great length. Therefore, these compounds are potentially suitable for applications in composite materials that need anisotropic properties.

A.2 Applications of carbon nanotubes

A.2.1 Energy storage

Graphite, carbonaceous materials and carbon fiber electrodes are commonly used in fuel cells, batteries and other electrochemical applications. Advantages of considering nanotubes for energy storage are their small dimensions, smooth surface topology and perfect surface specificity. The efficiency of fuel cells is determined by the electron transfer rate at the carbon electrodes, which is the fastest on nanotubes following ideal Nernstian behaviour.

A.2.2 Hydrogen storage

The advantage of hydrogen as energy source is that its combustion product is water. In addition, hydrogen can be easily regenerated. For this reason, a suitable hydrogen storage system is necessary, satisfying a combination of both volume and

weight limitations. The two commonly used means to store hydrogen are gas phase and electrochemical adsorption.

Because of their cylindrical and hollow geometry, and nanometer-scale diameters, it has been predicted that carbon nanotubes can store a liquid or a gas in the inner cores through a capillary effect. As a threshold for economical storage, the Department of Energy has set storage requirements of 6.5 % by weight as the minimum level for hydrogen fuel cells. It is reported that SWNTs were able to meet and sometimes exceed this level by using gas phase adsorption. Yet, most experimental reports of high storage capacities are rather controversial so that it is difficult to assess the applications potential.

A.2.3 Lithium intercalation

The basic principle of rechargeable lithium batteries is electrochemical intercalation and de-intercalation of lithium in both electrodes. An ideal battery has a high-energy capacity, fast charging time and a long cycle time. The capacity is determined by the lithium saturation concentration of the electrode materials. For Li, this is the highest in nanotubes if all the interstitial sites (inter-shell van der Waals spaces, inter-tube channels and inner cores) are accessible for Li intercalation. SWNTs have shown to possess both highly reversible and irreversible capacities. Because of the large observed voltage hysteresis, Li-intercalation in nanotubes is still unsuitable for battery application.

A.2.4 Electrochemical supercapacitors

Supercapacitors have a high capacitance and potentially applicable in electronic devices. Typically, they are comprised two electrodes separated by an insulating material that is ionically conducting in electrochemical devices. The capacity of an electrochemical supercap inversely depends on the separation between

the charge on the electrode and the counter charge in the electrolyte. Because this separation is about a nanometre for nanotubes in electrodes, very large capacities result from the high nanotube surface area accessible to the electrolyte. In this way, a large amount of charge injection occurs if only a small voltage is applied. This charge injection is used for energy storage in nanotube supercapacitors

A.2.5 Transistors

The field-effect transistor can be constructed of only one semi-conducting SWNT. By applying a voltage to a gate electrode, the nanotube can be switched from a conducting to an insulating state. Such carbon nanotube transistors can be coupled together, working as a logical switch, which is the basic component of computers.

A.2.6 Nanoprobes and sensors

Because of their flexibility, nanotubes can also be used in scanning probe instruments. Since MWNT-tips are conducting, they can be used in STM and AFM instruments. Advantages are the improved resolution in comparison with conventional Si or metal tips and the tips do not suffer from crashes with the surfaces because of their high elasticity.

A.2.7 Composite materials

Because of the stiffness of carbon nanotubes, they are ideal candidates for structural applications. For example, they may be used as reinforcements in high strength, low weight, and high performance composites.

A.2.8 Templates

Because of the small channels, strong capillary forces exist in nanotubes. These forces are strong enough to hold gases and fluids in nanotubes. In this way, it

may be possible to fill the cavities of the nanotubes to create nanowires. The critical issue here is the wetting characteristic of nanotubes. Because of their smaller pore sizes, filling of SWNTs is more difficult than filling of MWNTs.lalongkorn University, 2004.

APPENDIX B

Experiment results

Table B.1 TEM images of arc discharge, liquid nitrogen, and c-c electrodes

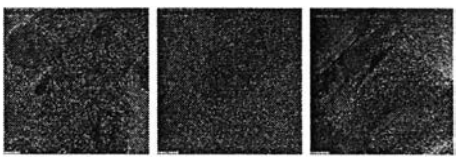
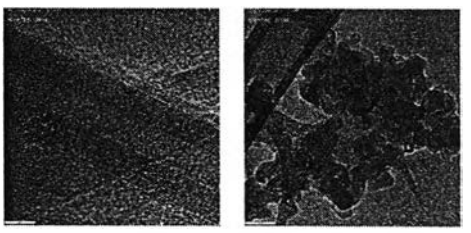
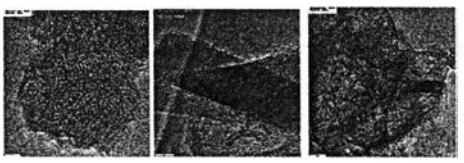
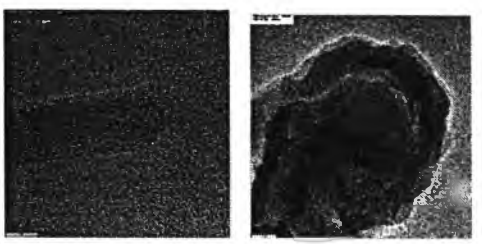
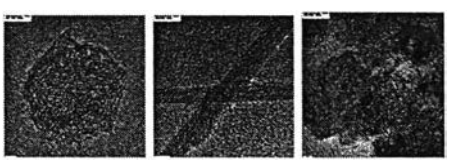
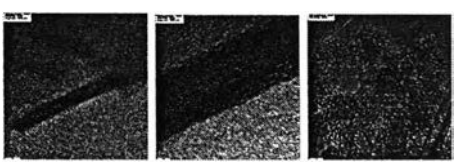
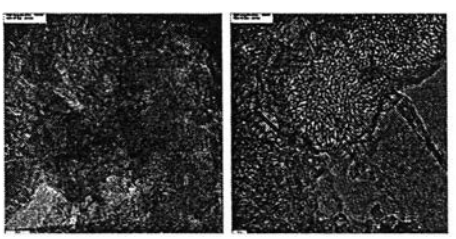
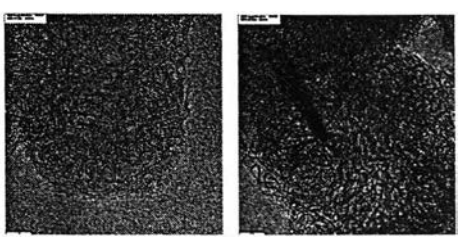
Current (A)	Carbon-carbon electrodes	
	Sedimenation	Cathode deposite
50	- CNHs high - CNTs lower - CNPs groups medium 	- CNTs high - CNPs groups medium 
75	- CNHs higher - CNTs lower - CNPs groups medium 	- CNTs low - CNPs groups high 
100	- CNHs higher - CNTs lower - CNPs groups high 	- CNTs lower - CNPs groups higher 
125	- CNHs higher - CNPs groups higher 	- CNPs groups only like carbon black 

Table B.2 TEM images of arc discharge, liquid nitrogen, and Fe-c electrodes

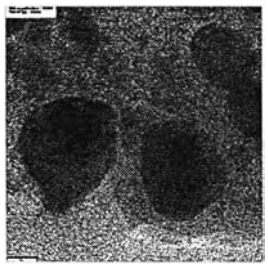
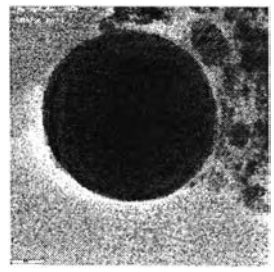
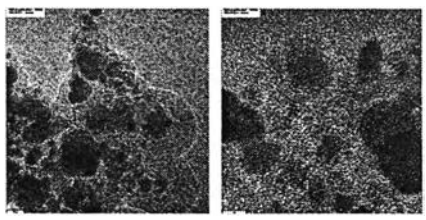
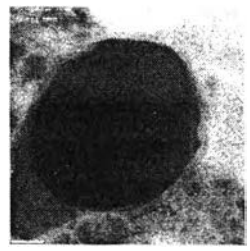
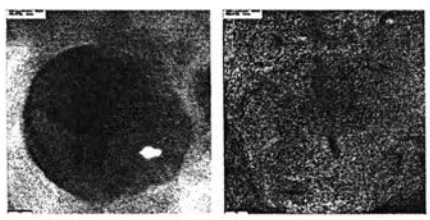
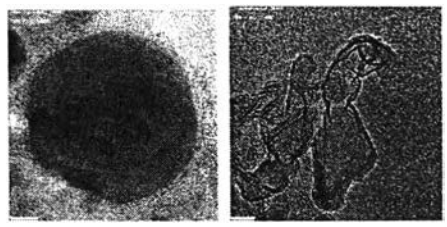
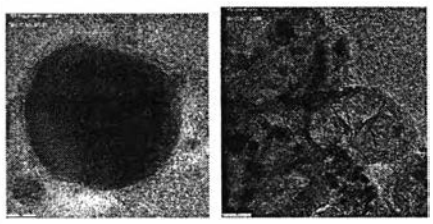
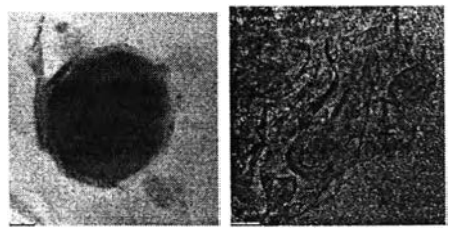
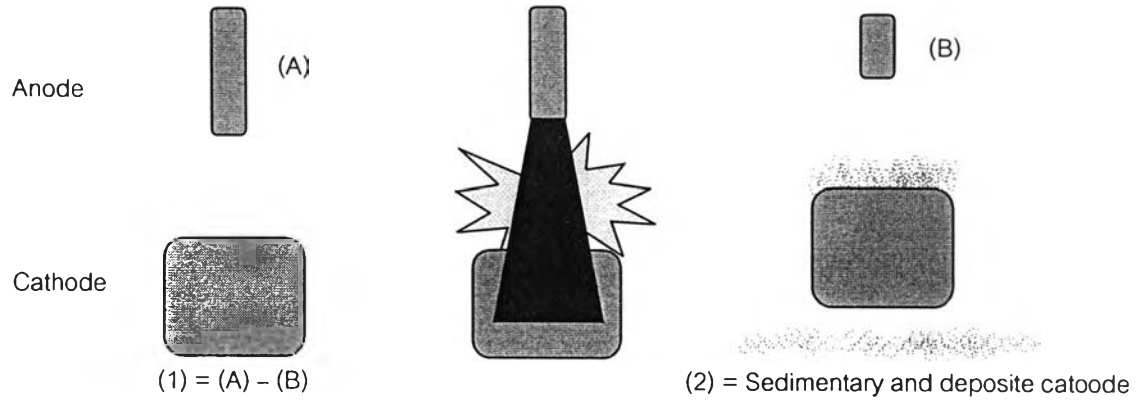
Iron-carbon electrodes			
I (A)	Anode 3mm	I (A)	Anode 6mm
100	- Agglomeration Fe coated by carbon 	175	- CNCs only 
125	- Agglomeration Fe coated by carbon - CNCs growth 	200	- CNCs only 
150	- CNCs high - CNPs groups low 	225	- CNCs high - CNPs groups high 
175 - 200	- CNCs high - CNPs groups higher 	250	- CNCs high - CNPs groups higher 

Table B.3 The yield of arc discharge, liquid nitrogen

The calculations of yield (%)



C-C electrodes

Anode ID (mm)	Current(A)	Carbon anode 6mm Raw material (g/batch), (1)	Carbon anode 6mm Product (g/batch), (2)	Carbon anode 6mm Yield (%), (2) * 100 / (1)
3	50	0.4120	0.5116	91.61
	75	0.7717	1.3125	58.80
	100	0.2304	0.4488	51.34
	125	0.4845	1.1092	43.68

A current 50A can use lowest in this work.

Fe-C electrodes

Anode ID (mm)	Current(A)	Carbon anode 3mm Raw material (g/batch), (1)	Carbon anode 3mm Product (g/batch), (2)	Carbon anode 3mm Yield (%), (2) *100 / (1)
3	100	0.2116	0.1647	77.84
	125	0.4278	0.3012	70.41
	150	0.3578	0.2372	66.29
	175	0.7337	0.4475	60.99
	200	0.5218	0.3137	60.12

A current 100A can use lowest in this work.

Anode ID (mm)	Current(A)	Carbon anode 6mm Raw material (g/batch), (1)	Carbon anode 6mm Product (g/batch), (2)	Carbon anode 6mm Yield (%), (2) *100 / (1)
6	175	0.4075	0.3616	88.74
	200	0.5331	0.3616	58.84
	225	0.4965	0.1780	35.85
	250	0.5807	0.1913	32.94

A current 175A can use lowest in this work.

Table B.4 DLS peak diameter of arc discharge, liquid nitrogen

C-C electrodes

Anode ID (mm)	Current(A)	Sedimentary product (nm)	Cathode deposit (nm)
3	50	305.80	270.00
	75	318.90	285.80
	100	339.60	295.70
	125	340.50	295.70

Fe-C electrodes

Anode ID (mm)	Current(A)	Carbon anode ID(3nm)	Carbon anode ID(6nm)
3-6	100	195.00	N/A
	125	231.30	N/A
	150	273.30	N/A
	175	306.80	143.00
	200	341.00	172.20
	225	N/A	210.20
	250	N/A	240.50

Remark: No-Available become operation could not be conducted

Table B.5 G/D ratio of arc discharge, liquid nitrogen

C-C electrodes

Anode ID (mm)	Current(A)	Sedimentary product (nm)	Cathode deposit (nm)
3	50	1.58	2.40
	75	1.54	2.16
	100	0.87	2.05
	125	0.31	1.10

Fe-C electrodes

Anode ID (mm)	Current(A)	Carbon anode ID(3nm)	Carbon anode ID(6nm)
3-6	100	0.36	N/A
	125	0.55	N/A
	150	0.82	N/A
	175	1.13	1.10
	200	1.84	1.26
	225	N/A	1.44
	250	N/A	0.72

Remark: No-Available become operation could not be conducted

Table B.6 DSC data for i-PP/CNPs nanocomposites,

as a function of concentrations CNPs.

Samples of CNPs	Concentrations (%)	Melting Temperature (T_m, ^oC)	Heat flow rate (J/g)
C-C 50A (CNPs)	0	168.66	90.78
	5	163.05	85.16
	10	162.96	80.60
	15	162.15	76.57
C-C 125A (CNPs)	0	168.66	90.78
	5	163.57	91.93
	10	163.12	55.06
	15	162.16	47.96
Fe-C 175A (CNCs)	0	168.66	90.78
	5	163.10	91.56
	10	162.79	76.72
	15	162.19	74.32

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