

Carbon Nanotube Alignment Methods

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Abstract

The outstanding properties of carbon nanotubes (CNTs) exist in their nanoscale form. The CNTs must be maintained aligned in the device to preserve these properties in the macroscale and bulk form. Recently, many studies addressed the alignment of CNTs at different scales for different applications. For example, CNTs are aligned vertically simultaneously as they grow on a substrate. Pre-synthesized CNTs can be aligned horizontally on a large scale under the influence of external forces such as electric and magnetic forces. This chapter reviews the latest techniques and methods regarding the horizontal alignment of CNTs. The alignment methods are classified based on the force used to achieve the alignment. The chapter concludes by discussing each method's advantages, disadvantages, and potential applications.

Keywords: carbon nanotubes, alignment, assembly, forces, applications

1. Introduction

Discovering carbon nanotubes (CNTs) by Iijima was an innovation in the field of science and engineering [1]. The remarkable electronic, mechanical, optical, and chemical properties of CNTs made them a promising material in future industries and applications [2–8].

CNT applications have not seen widespread adoption because of the CNT anisotropic nature and the difficulty in placing them into the desired location. Another reason is that CNTs are synthesized in a high-temperature environment, which is incompatible with the current fabrication methods [9]. In addition, there are difficulties in controlling the CNT structure during production [10].

Enforcing the CNT usage with the mentioned limitation may produce materials with considerable variations in their properties and devices that possess anisotropic behavior. Researchers have made efforts to overcome these limitations by improving the synthesis methods to produce high-quality pure CNT that can be grown directly into the device or by further processing the CNT using alignment techniques to purify, transfer, and assemble the CNT to the desired location [11].

In devices, CNTs can be aligned perpendicular or parallel to the substrate. In the first case, the CNTs are aligned vertically simultaneously as they grow on the substrate, as shown in **Figure 1a**. The vertical alignment challenges are the catalyst removal process, compatibility with the device fabrication methods, and the difficulties in transferring the aligned CNTs from the substrate to the target application [12–14].

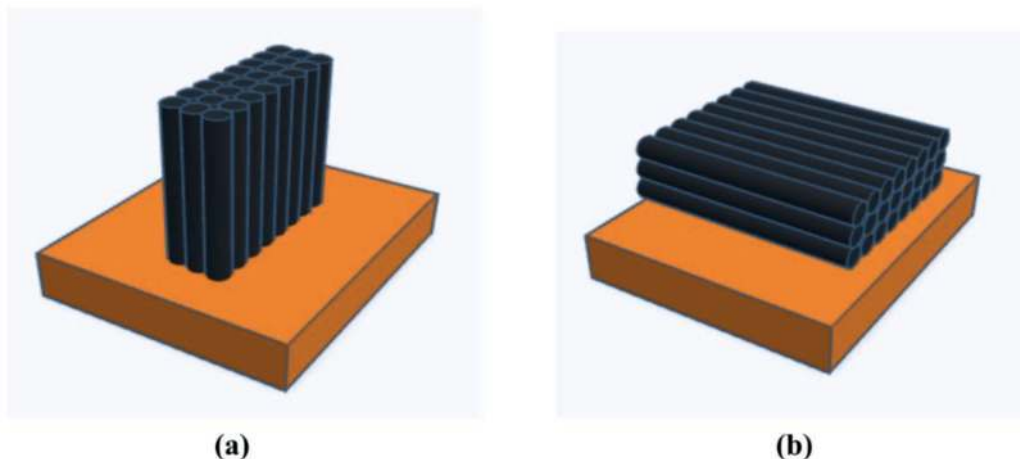


Figure 1.
(a) Vertically aligned CNTs. (b) Horizontally aligned CNTs.

However, pre-synthesized CNTs can be aligned vertically on a substrate under the influence of external forces such as electric fields and magnetic fields [15, 16]. In the case of horizontal alignment, the CNTs are assembled in parallel with the substrate surface, as shown in **Figure 1b**. Several methods are used to assemble and align CNTs vertically [17].

This chapter briefly reviews the methods that are used to align CNTs. The methods are classified based on the force used to achieve the alignment.

2. Alignment by magnetic forces

The principle of CNTs alignment by magnetic force is when strong magnetic fields are applied on CNTs at a macroscopic scale (Magnetophoresis). The CNT segments are aligned in parallel with the magnetic field lines to form long wires up to centimeters in length. The source of the magnetic field can be a permanent magnet, a superconducting magnet, or a resistive-coil magnet [18–20]. The response of CNTs to magnetic fields can also be employed in CNT separation [21].

The magnetic properties of CNTs are known to be very weak; hence, it is hard to assemble CNTs using low magnetic fields. The strength of the required magnetic field depends on the type of CNT. In the case of unmodified CNTs, the required field to align CNTs is in the range of 5 T–25 T, whereas MWCNTs require a less intense magnetic field than SWCNTs under the same conditions [22]. For example, Kimura et al. aligned MWCNTs in a polyester matrix using a superconducting magnet of 10 T. MWCNTs were dispersed in an unsaturated polyester-styrene monomer and then placed in a mold inside a magnet (see **Figure 2a**). MWCNTs are aligned as the polyester matrix polymerization is carried out [19]. The magnetic field intensity to align SWCNT in the same condition was 25 T, as demonstrated by Camponeschi et al. [23].

CNTs are coated with magnetite nanoparticles to effectively enhance their response to low magnetic fields. The required magnetic field to align CNTs is reduced by 1 to 3 orders of magnitude when CNTs are coated or decorated with magnetic nanoparticles such as Ni and Fe_2O_3 [24, 25]. For example, Wu et al. used a weak magnetic field (~ 50 mT) to align magnetite-carbon nanofiber hybrids ($\text{Fe}_3\text{O}_4/\text{CNT}$) and form a

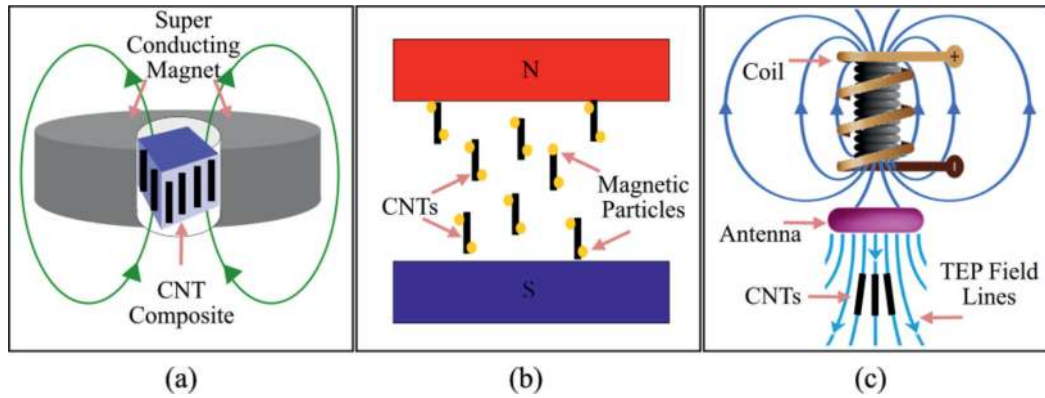


Figure 2. Alignment by magnetic field using: (a) superconducting magnet [19]. (b) permanent magnet [26]. (c) resistive-coil magnet [20].

chain-like structure in an epoxy resin. **Figure 2b** shows the principle of aligning CNTs decorated with magnetite nanoparticles using a permanent magnet [26].

Teslaphoresis (TEP) is a non-conventional magnetophoretic force proposed by Bornhoeft et al. to overcome the small-scale structure limitation of the alignment methods. They used a Tesla coil's antenna supplied with a high-voltage force to transmit a near-field radio frequency of 2 MHz. The antenna projected a gradient field into the free space. **Figure 2c** shows that CNT wires are self-assembled along the TEP field lines, which are generated by a Tesla coil's antenna [20].

3. Alignment by acoustic forces

Acoustophoresis is a method used to pattern, release, and assemble CNTs using acoustic waves [27]. The acoustic radiation force is usually generated from a piezoelectric surface and then propagates through a CNT suspension, facilitating the tube movement [28]. The acoustic waves are classified into surface acoustic waves (SAWs) and bulk acoustic waves (BAWs).

Seemann KM et al. demonstrated SWCNTs and MWCNTs alignment between pre-structured interdigitated electrodes (IDE) using SAWs. The IDE is printed on a Lithium Niobite (LiNbO_3) surface to generate an acoustic field within a gap filled with an aqueous CNT suspension, as shown in **Figure 3a**. The SAWs' propagation within the CNT medium will cause a CNT motion, and the CNTs will be deposited in a specific pattern depending on the wavelength [29].

As demonstrated by Haslam et al., standing BAWs are used to align MWCNTs inside polymer composite resin. The composite is placed between two piezoelectric plates attached to electrical electrodes, as shown in **Figure 3b**. The electrodes supply the piezoelectric plates with an electrical signal to convert them to BAWs. The BAWs propagated through the composite matrix and aligned the MWCNTs in the wave's direction [30].

Zhichao Ma et al. used numerical simulation and experimentation to investigate the mechanism of the SAW-based patterning technique. They found that two different fields affect the patterning process, the acoustic pressure field, and the electric field. The acoustic radiation causes a micro-fluidic flow, while the electric field along the piezoelectric substrate causes a dipole moment. Both the micro-fluidic flow and

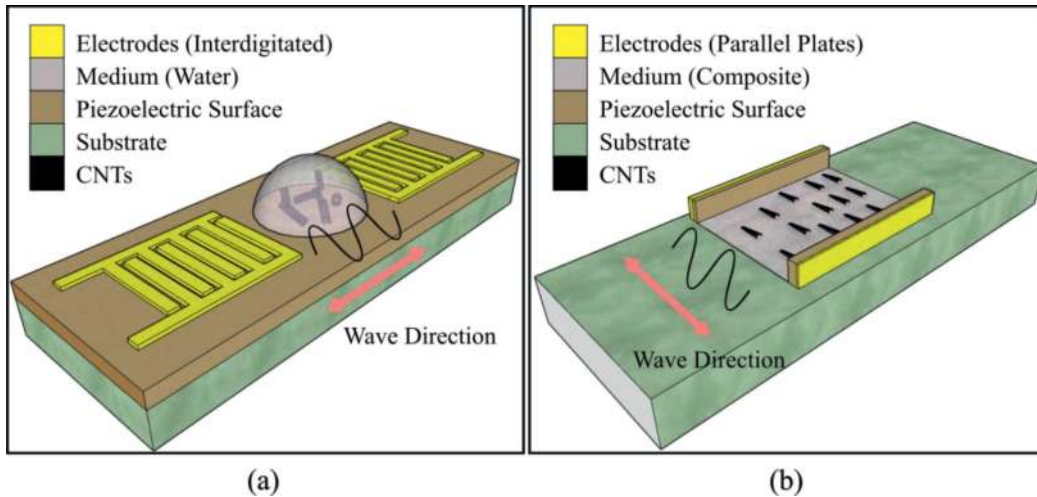


Figure 3. Alignment by acoustic waves. (a) SAW structure [29]. (b) BAW structure [30].

dipole moment contribute to the patterning and alignment process [31]. Besides the assembly, acoustic waves were used in the synthesis of M-SWCNTs to produce monochromatic ultralong tubes [32].

4. Alignment by chemical forces

Alignment by chemical forces does not require any external field or force because CNTs are self-assembled based on charged molecules or chemical interaction between CNTs and a stimulatory material. For example, a solution-based chemical method such as evaporation-driven dip-casting is used to assemble CNTs on a large-scale area. A hydrophilic and hydrophobic interphase structure is formed on a silicon substrate to deposit SWCNTs, as shown in **Figure 4a**. The CNTs are directly deposited on the SiO₂ hydrophilic substrate without being contaminated by the hydrophobic strip regions [33].

Alignment relay technique (ART) is a new chemical alignment method first reported by Selmani and Schipper. The method is based on preparing a molecularly functionalized surface to sort and align CNTs simultaneously. The alignment process is

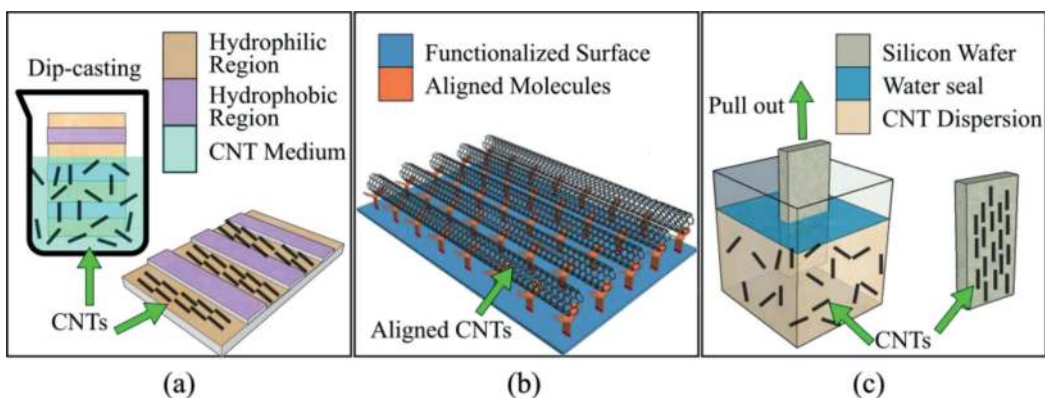


Figure 4. Alignment by chemical methods. (a) Evaporation-driven Dip-casting [33]. (b) Alignment relay technique [34]. (c) Electrostatic repulsion Dip-coating [36].

passed from liquid crystals (LCs) to small molecules that can interact with CNTs selectively. The CNTs are deposited on the aligned molecules and self-aligned following the molecules' pattern, as shown in **Figure 4b** [34]. Mark J. MacLachlan reported CNTs alignment by ART on an ITO surface functionalized with Iptycene molecules [35].

A combination of electrostatic repulsion and dip-coating method is used to assemble S-SWCNT on an untreated silicon wafer. The wafer dipped into a bi-phasic dispersion of deionized water and chloroform. The dispersion contains S-SWCNTs with positively charged molecules. After the substrate was fully immersed, it was pulled out at a precise speed of 200-1000 $\mu\text{m/s}$ to form a thin film of S-SWCNTs deposited on the silicon wafer, as shown in **Figure 4c** [36].

5. Alignment by mechanical forces

Alignment by mechanical forces is most prevalent in polymer matrixes and CNT fibers and can be classified into three different categories, pure shear-induced techniques, extrusion-induced techniques, and flow-induced techniques [37].

The shear-induced techniques use direct shear force, which can be applied in various forms, such as cutting, rubbing, or pushing. In the cutting methods, a knife or blade is used to slice through a CNT composite material. CNTs can be seen on the cut surface aligned with the cutting direction, as shown in **Figure 5a** [38]. The alignment by pushing (or rubbing) is usually used with raw CNT powder synthesized by chemical vapor deposition (CVD). The vertically synthesized CNTs are rubbed against a smooth plastic surface to form a high-density thin film where the alignment is in the rubbing direction, as shown in **Figure 5b** [39]. Along with cutting and rubbing, dispersed CNTs in a polymer matrix can be aligned by pulling [40], friction [41], stretching [42], fracture [43], peeling [44], and uniaxial pressure [45]. The selection of the method depends on the polymer hardness, elasticity, size, and sample stability temperature.

Extrusion techniques such as melt fiber spinning, direct spinning, and electrospinning are used to fabricate long and continuous CNT yarns, wires, and fibers [46–48]. In these techniques, the CNTs are dispersed in a medium and then self-assembled in the drawing direction. However, the differences between the methods are the number of stages and the CNT source where the fiber spun from. For example, the melt fiber

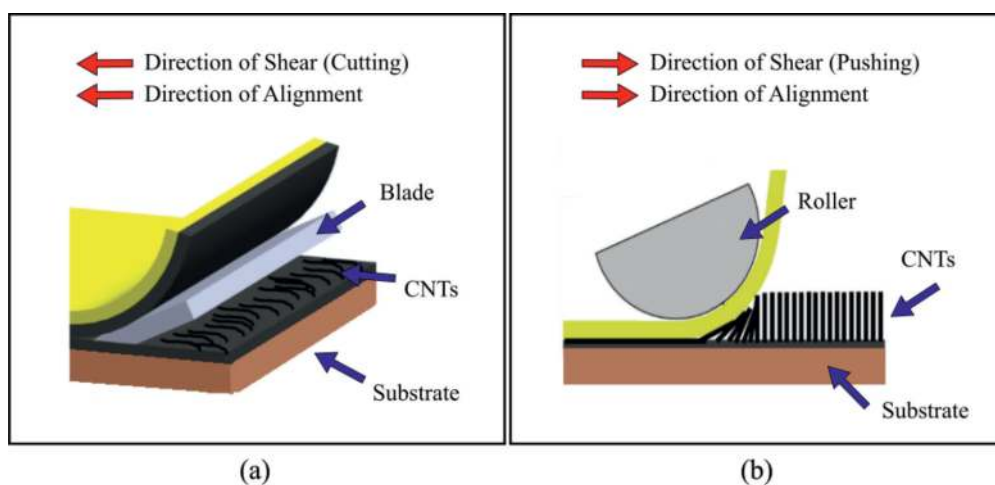


Figure 5. Alignment by mechanical forces [37]. (a) Cutting [38]. (b) Pushing [39].

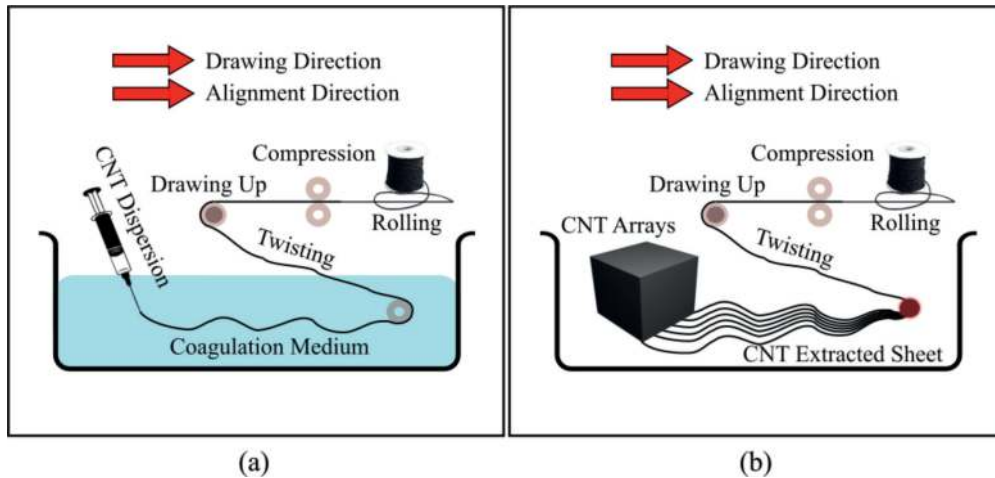


Figure 6. Alignment by direct spinning in fibers. (a) Wet spinning [50]. (b) Dry spinning [51].

spinning method is used to align CNTs in thermoplastic polymers. Both SWCNTs and MWCNTs are dispersed into molten thermoplastic polycarbonate (PC) using a twin-screw extruder. The dispersion process is followed by melt spinning alignment to produce a fiber with aligned CNTs [49].

Alignment by direct spinning is classified into wet-spinning and dry-spinning methods. In the wet-spinning method, the fibers are prepared via coagulation spinning, where homogeneously CNT dispersion is injected into a co-flowing stream of a polymer solution, as shown in **Figure 6a** [50]. In the second method, the fibers are assembled from vertically aligned CNT arrays or CNT aerogels, as shown in **Figure 6b** [51]. A twisting phase is introduced in both methods during the spinning to align the CNTs in the drawing direction.

Flow-induced techniques, such as gas flow, are used to align CNTs vertically and horizontally on various substrate surfaces. The gas flow method is simple, easy to scale up, and can be used during the growth of CNTs to align them vertically. The method was also used as a post-growth alignment method to align CNTs horizontally on substrates.

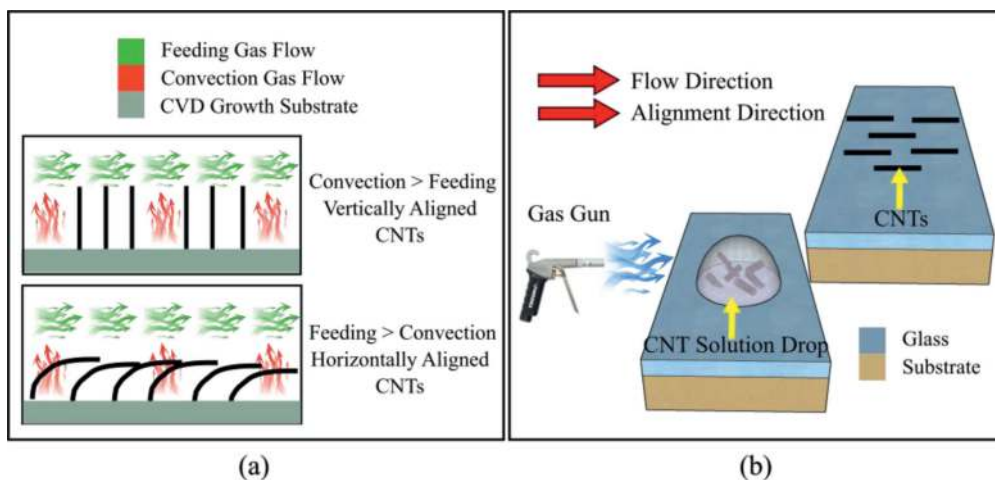


Figure 7. Alignment by flow-induced techniques. (a) During growth alignment [54]. (b) Post-growth alignment [55].

The alignment during the growth means that CNTs are aligned as they grow by the CVD process. The CVD process uses two stable laminar gas flows to direct the growth of CNTs during their synthesis. These laminar flows are the feeding gas flow and the convection gas flow [52]. The CNTs are vertically aligned due to the temperature difference between the substrate and the surrounding environment. The temperature difference causes a convection gas flow to keep the CNTs growing outward from the substrate surface [53]. At a high feeding gas flow rate, the feeding gas flow dominates the convection gas flow, and therefore, the CNTs are horizontally aligned in the direction of the feeding gas flow, as shown in **Figure 7a** [54].

Flow-induced techniques are also used to align post-grown CNTs horizontally onto prefabricated electrode structures. A drop of a CNT solution is pipetted on a smooth surface and subjected to a gas flow force. The shear force resulting from the gas flow produced a torque on the tubes and led to their alignment, as shown in **Figure 7b**. In addition to the alignment, gas flow simultaneously spreads the solution drops and helps in the drying process [55].

6. Alignment by electric forces

Electrophoretic (EP) and DEP are electric forces that are used to align CNTs between two or more electrodes. The alignment by EP occurs when a surface-charged CNT is subjected to a uniform electric field. The alignment using DEP is based on the CNT dielectric polarization factor, where a non-uniform electric field is used to induce a dipole moment within the CNT itself. EP is mostly used to align CNTs in nanocomposite using parallel plate electrodes, while DEP is used to align CNTs across planar electrodes to form two-terminal devices such as sensors and CNT transistors [56, 57].

Direct current electrophoresis (DC-EP) and alternating current electrophoresis (AC-EP) were first reported by Yamamoto K et al. in 1996 and 1998, respectively, to orientate CNTs using an electric field. In the DC-EP alignment, a CNT sample was obtained from the carbon arc-discharge method, then ultrasonically dispersed in isopropyl alcohol (IPA). The solution was centrifugated to remove large particles and then dropped onto coplanar electrodes made of aluminum with a gap width of 0.4mm. The electric field that was applied to the electrodes was 25×10^3 V in the case of DC-EP and 2.2×10^3 V_{rms} with frequencies ranging from 10 Hz to 10 MHz in the case of AC-EP. Isopropyl ionization helps the CNTs move and align in response to the electric field amplitude and frequency variation [58, 59].

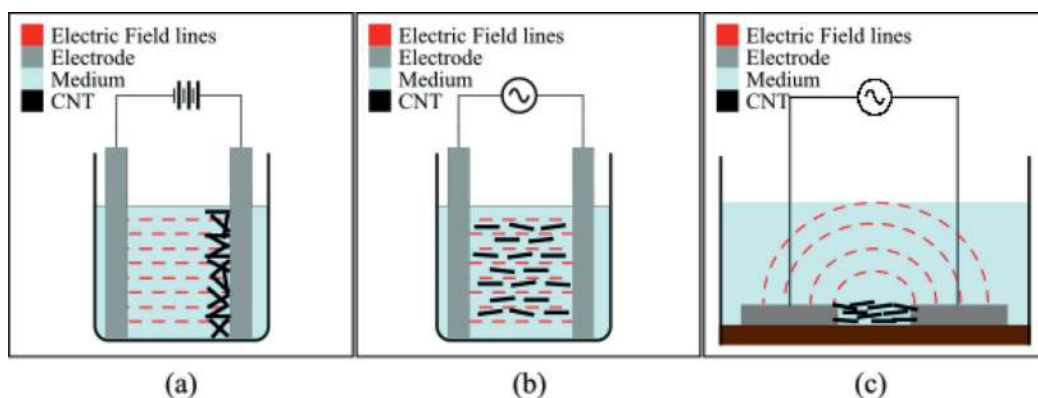
The first time DEP was used in CNT integration was in 2003 by Krupke et al. to separate metallic bundles of SWCNT from a solution and assemble them into a device. The SWCNTs were grown by laser ablation, purified with nitric acid, and suspended in dimethylformamide (DMF). Electrodes were prepared with standard e-beam lithography and connected to an AC power supplier. After dropping the CNT suspension on the chip, the generator was switched on to align the CNTs. After alignment, the drop is blown off gently with nitrogen gas [60]. Later in 2007, A. Vijayaraghavan et al. successfully produced high-density SWCNT devices fabricated on a single chip using alternating current DEP. Several million devices were packaged in a square centimeter [61]. Recent studies concluded that the quality of aligned CNTs using DEP depends on many factors, such as the electrode design [62, 63], suspension quality [64], and CNTs enrichment [65, 66].

The differences between EP and DEP and the resulting CNT motion from each force (translational and rotational) are summarized in **Table 1**. The translational

Force	Electric field	CNT behavior	Electrode configuration
DC-EP	Uniform	CNTs Move Toward One Electrode Based on their Surface Charge.	Parallel Plate Electrodes
AC-EP	Uniform	CNTs Rotate Following the Electric Field Lines	Parallel Plate Electrodes
DEP	Non-Uniform	CNT Rotates Following the Electric Field Lines and Moves Toward the Region with the High Electric Field	Planner Electrodes, IDE

Table 1.

The behavior of a CNT subjected to an electric field.

**Figure 8.**

Alignment by electric forces. (a) DC-EP. (b) AC-EP. (c) DEP.

motion of CNTs is governed by the electrophoretic mobility of charged CNT's surface and tips, while the rotational motion is governed by the relaxation mechanism and the dipole moment created by the locally non-uniform electric field [67]. **Figure 8a** shows a translation motion of CNTs toward the electrode with opposite charges because the electrodes were connected to a DC source, and DC-EP occurred. If the DC source is replaced with an AC source, the CNTs will experience a rotation motion and align with the electric field line due to the AC-EP (see **Figure 8b**). The CNTs would experience both translational and rotational motion if the electrode configuration changed from parallel plates to planar electrodes due to the DEP force, as shown in **Figure 8c**. In the DEP method, electric fields are usually generated by sinusoidal voltage sources [68]. Alternating pulsed currents are also used to assist the alignment [69].

7. Alignment by other methods

The CNT alignment methods vary based on the CNT sample size, from an individual tube to bulk CNT powder. The focused ion beam (FIB) method is an example of controlling an individual tube's orientation. The FIB method uses a focused beam of ions to control an individual tube's alignment angle after coating the tube with metal nanoparticles. The orientation is achieved by attaching the CNT to a pyramidal tip end of a nanoprobe then the CNT is exposed to an external ion beam source. The beam intensity and beam scanning cycles determine the degree of alignment of the metal-coated CNT [70]. On the other side, some methods provide a large-scale CNT

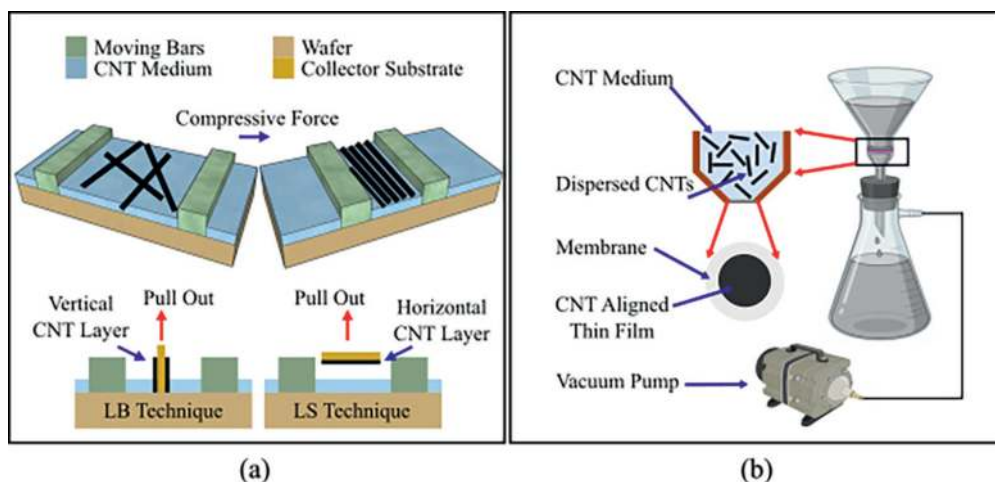


Figure 9. Other alignment methods. (a) Langmuir-Blodgett and Langmuir-Schaefer methods. (b) Filtration method.

alignment, such as Langmuir-Blodgett method (LB) [71], Langmuir-Schaefer (LS) method [72], and vacuum filtration method [73].

The LB method is used to fabricate monolayers of aligned SWCNTs on oxide substrates. The degree of alignment and packing is improved by pressure cycling and thermal annealing during layers fabrication [71]. The LS method is versatile for the fabrication of high-density arrays of more than 500 tubes/ μm . The method is used to assemble high-purity S-SWCNT from 1,2-dichloroethane (DCE) for high-performance transistors [72]. **Figure 9a** illustrates the alignment using LB and LS methods where a uniaxial compressive force is applied using moving bars to assemble CNTs into well-ordered arrays. The main difference between LB and LS is the collection mechanism where the assembled monolayer arrays are vertically transferred in the case of LB and horizontally transferred in the case of LS using a collector substrate.

Filtration methods are used to prepare high-packing density well-aligned CNT thin films. The degree of alignment is controlled by the filtration rate and the CNT concentration in the solution [73]. The filtration method consists of a conventional filtration setup and vacuum pump to control the medium flow rate through the membrane, as shown in **Figure 9b**.

Ceramic filters [74], blown bubbles [75], viscous shear stress [76], confined shear alignment [77], and nematic liquid alignment [78] are additional film-assembly methods that provide a large-scale alignment regarding the type of CNTs. The confined shear alignment method is used to deposit a uniform film of aligned carbon nanotubes across an $8 \times 8 \text{ cm}^2$ region [77]. The nematic liquid alignment occurs because CNTs exhibit a liquid crystal phase when they are dispersed into a nematic liquid crystal solvent [78].

8. Chapter summary

The superior properties of CNTs are observed when the CNTs are aligned or deposited in a specific order. Random distributed CNTs showed unpredicted behavior and poor electrical performance compared with the aligned CNTs. For those reasons, CNTs alignment is an essential step in the fabrication of CNT-based devices and materials. **Table 2** summarizes the alignment methods and their working scale,

Method	Scale (m)	Application	Advantages	Challenges	Ref.
DEP	10^{-7} – 10^{-5}	CNTFET, Sensors	Low Power, Room Temperature, Easy Setup	CNTs dispersity, Limited by the Electrode Geometry, Electrothermal Heating	[79, 80]
EP, Electric Field	10^{-4} – 10^{-2}	Composites	Simple and Flexible Setup	Requires High Voltage For Large Sample, Electrodes in Contact with the CNT Sample	[81, 82]
Vacuum Filtration	10^{-2}	Thin Films	Simple Setup, Scalable	Membrane Removal, Flow Rate Control of the CNT Medium	[83]
Teslaphoresis Magnetophoresis	10^{-1}	Connectors	Electrodeless, Real-time Method	Requires a High Magnetic Field, Controllability of the Magnetic Field, Magnetic Particles Removal	[20, 84]
Twisting	10^0 – 10^1	Fibers, Wires	Scalable, Relatively Cheap	Complicated Setup, Difficulties in Controlling the Tube-Tube Contacts	[85]
Acoustic	10^{-6} – 10^{-4}	CNT Patterns	The sample has no contact with the Electrodes	Limited to Piezo Material Surfaces	[86]

Table 2.
CNTs post alignment methods, applications, and challenges.

applications, advantages, and challenges. It is clear that each method works fine on a specific scale. Outside that scale, challenges start to appear and limit the methods from being universal methods. Thus, the alignment method's selection depends on the application geometry and the compatibility with its fabrication process.

Conflict of interest


The authors declare no conflict of interest.

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