

Chapter 2

Synthesis, Characterization, and Applications of Carbon Nanotubes Functionalized with Magnetic Nanoparticles

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2.1 Introduction

Nanomaterials have a characteristic dimension of the order of 100 nm. They occur as compact materials or in dispersions. The deviation of their properties from those of bulk materials with the same chemical constituents has led to research across a wide range of applications. Various 1D nanomaterials include nanospheres, nanorods, nanobelts, nanorings, nanotubes, nanohelics, nanowires, and nanosheets, each with unique properties. Here, we discuss carbon nanotubes and magnetic nanoparticles and explain how their properties can be advantageously merged to overcome certain barriers and meet specific objectives.

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2.1.1 Carbon Nanotubes

Carbon nanotubes are anisotropic 1-D structures. After multi-walled carbon nanotubes were discovered using high-resolution transmission electron microscopy [1, 2], research revealed the structure and properties of other types of CNTs, such as single-walled nanotubes (SWNTs) and double-walled nanotubes (DWNTs) along with carbonaceous nanomaterials like graphene. SWNTs consist of a single 1D graphene sheet rolled into a tube with a diameter of 1–2 nm and a relatively high aspect ratio [2]. MWNTs consist of multiple graphene sheets rolled into cylinders of outer diameter 10–80 nm [3]. The structure of CNTs is revealed through the chiral vector $\vec{c}$ (expressed by two indices n and m) and the chiral angle θ . Figure 2.1 illustrates the three broad CNT types that depend on the rolling orientation of the chiral vector, namely (a) armchair ($n = m$), (b) zigzag ($m = 0$), and (c) chiral ($n \geq 0; m \geq 0$) nanotubes, where $0^\circ < \theta < 30^\circ$, i.e., θ is the chirality angle. The unique electrical properties of a CNT depend on its chirality and tube diameter. CNTs can be either metallic (armchair nanotubes) when $|n - m| = 3q$ where q is an integer, or semiconducting for all other cases. The extraordinary electrical properties of SWNTs have pointed the way for fabricating novel electronics [4]. The shape and structure of a CNT results in a high axial thermal conductivity of $1750\text{--}5800 \text{ W m}^{-1} \text{ K}^{-1}$ at room temperature [5]. The electrical resistivity of CNTs has been measured in the range $10^{-4}\text{--}10^{-3} \Omega \text{ cm}$ along with an exceedingly high current carrying capacity up to 10^9 A cm^{-2} [5–7]. CNTs have high thermal stability (up to 2800°C in vacuum and about 750°C in air), high surface area ($200\text{--}900 \text{ m}^2 \text{ g}^{-1}$), low density ($1\text{--}2 \text{ g cm}^{-3}$), and high Young's modulus ($1\text{--}1.8 \text{ TPa}$) [3, 5, 6]. These properties make CNTs useful for sensors [8, 9], high strength materials [10], nanoelectronics [11, 12], fuel storage [13], energy storage [14], and biomedicine [15–18].

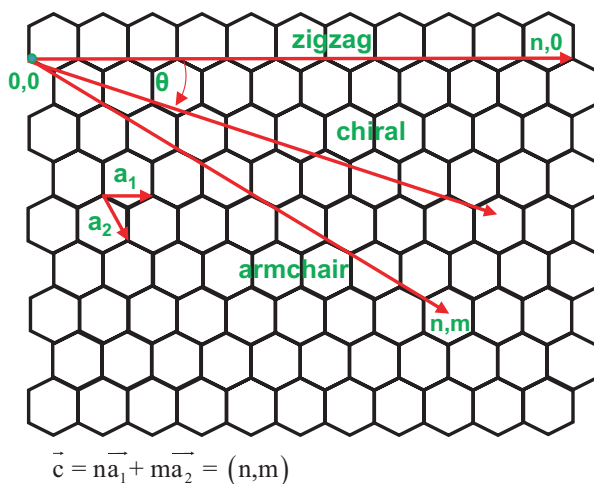


Fig. 2.1 Schematic of the graphene sheet and the different structures of a nanotube that can be rolled based on the chiral vector $\vec{c}$ characterized by the indices n and m

Commercially available CNTs are typically produced by chemical vapor deposition [19–21], and are entangled and randomly oriented due to short-range van der Waals forces between adjacent CNTs. This entanglement diminishes the effective properties of CNTs [22–25]. Being nonpolar, the carbon atoms of CNTs have a high affinity toward nonpolar materials, such as organic solvents, oils, and hydrocarbons. CNT's hydrophobicity limits their dispersion stability with polar solvents and polymers [26, 27]. Pure CNTs are diamagnetic but those synthesized with metal nanoparticles as catalysts can exhibit ferromagnetic behavior due to the presence of the magnetic catalyst [28], which is easily lost during acid treatment.

The diamagnetic susceptibility of CNTs limits remote control, or action from a distance, using a magnetic field. The chemical inertness of CNTs poses a serious difficulty for synthesizing composites with materials that are important for device applications. Hence, CNT functionalization with MNPs is a strategy to chaperone the nanotubes, manipulate, and organize them on demand [29–33].

2.1.2 Magnetic Nanoparticles

The magnetic properties of MNPs are used in many applications, e.g., microwave absorption [34], electrochemical sensing [35], ferrofluids [36], energy storage [37], magnetic resonance imaging, and data storage [38]. Since these magnetic properties are dependent on MNP size and shape, as shown in Fig. 2.2, different synthesis methods have been explored, including microemulsion [39], thermal decomposition [40], co-precipitation [41], sol-gel [42], wet chemical [43], self-assembly [44], spray pyrolysis [45], solvothermal method [46], template directed [47], and

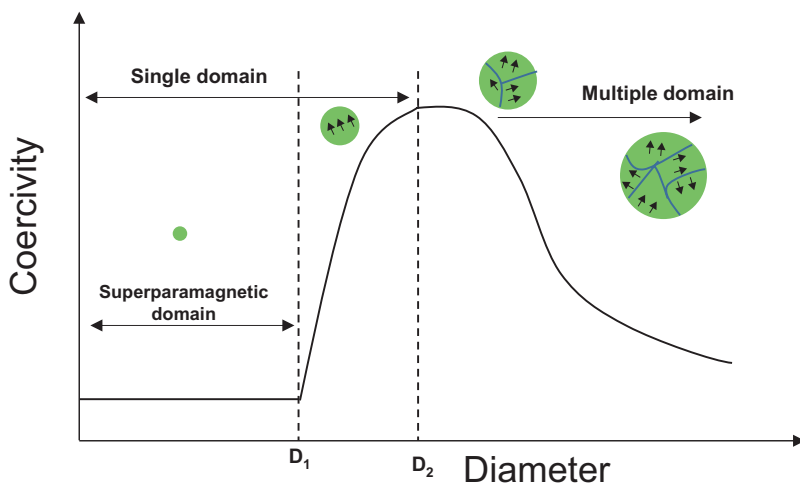


Fig. 2.2 Schematic representation of the change in coercivity with the size of a magnetic nanoparticle

deposition [48]. Although the physical properties of MNPs are improved with a large surface area to volume ratio, their agglomeration is an important concern. The crystallinity and the degree of defects or impurities of MNPs depend on the method of synthesis, which influences their magnetic behavior [49].

The five basic types of magnetism are ferromagnetism, antiferromagnetism, ferrimagnetism, paramagnetism, and diamagnetism. Ferrimagnetism occurs for compound materials, such as ferrites whereas the other types arise in pure elements. The spinning of electrons creates magnetic moments. Ferromagnetic materials have aligned magnetic moments that offer spontaneous magnetization in materials such as Fe, Ni, and Co. In antiferromagnetic materials, the magnetic moments are arranged in an antiparallel fashion so that the net magnetic moment is zero, a behavior that is observed at low temperatures. Ferrimagnets retain their magnetization even in the absence of a field but have antiparallel magnetic moments similar to antiferromagnets that are of unequal magnitude. Materials with uncoupled magnetic moments display paramagnetism with a small positive magnetic susceptibility. Materials that are repelled by a magnetic field and have a slightly negative susceptibility display diamagnetism.

The Weiss, or magnetic, domain is a volume of magnetic material in which all of the magnetic moments are aligned in the same direction. As the size of a ferromagnetic nanoparticle decreases, its magnetization becomes more uniform until a dimension D_2 when the domain walls within the particle disappear, resulting in a single domain. Further size reduction below D_1 results in thermal fluctuations overcoming the magnetic moment in the single domain so that the particle becomes superparamagnetic. Conversely, with an increase in particle size, the number of domain boundaries within a particle increases and thus its coercivity also decreases.

The repeatable synthesis of MNPs with a particular morphology and size is of consequence for monodisperse colloids. The magnetic response of MNPs to an external magnetic field can be utilized to tailor CNTs into aligned structures that harness their unique properties. The following section discusses recent advances of CNT functionalization with MNPs using different routes and the applications of the magnetized CNTs.

2.2 Functionalization of Carbon Nanotubes

The physical and chemical properties of CNTs can be enhanced in comparison to those of pristine CNTs by introducing external molecular groups or radicals on their surfaces [50]. These added functional groups improve the compatibility of CNTs with a dispersing medium and with solvents, polymer and organic molecules [51], improving the processability and solubility of the constituent CNTs. The introduction of a molecular group can either be through chemical (e.g., covalent interaction) or physical (e.g., van der Waals interaction, adsorption) means. Different CNT functionalization methods are classified based on the chemistry involved and illustrated in Fig. 2.3.

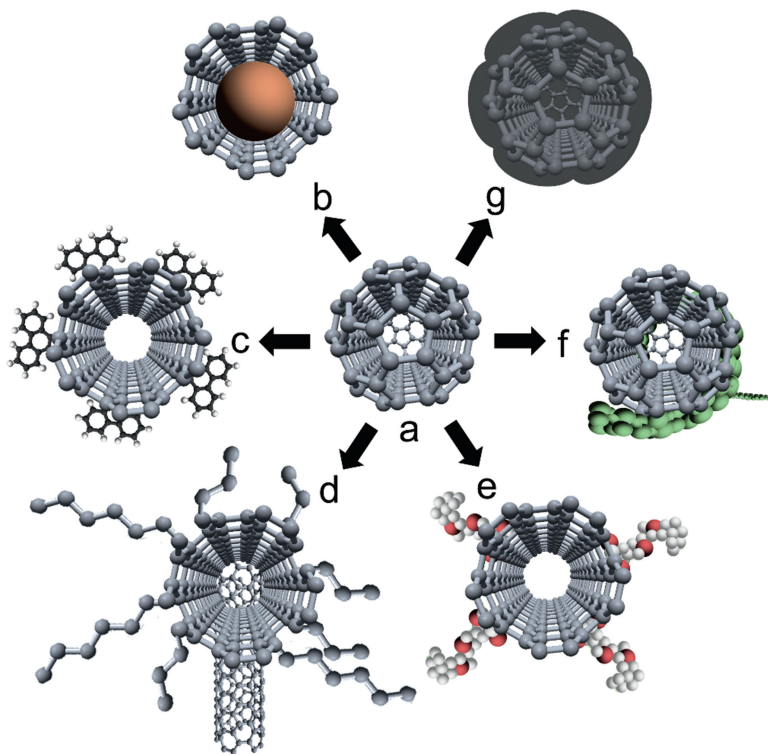


Fig. 2.3 Schematic of different methods of functionalizing SWNTs: (a) Single-walled carbon nanotube, (b) endohedral functionalization with, for example, C₆₀, (c) covalent sidewall functionalization, (d) defect-group functionalization, (e) noncovalent exohedral functionalization with surfactants, (f) noncovalent exohedral functionalization with polymers, and (g) metal plating of carbon nanotubes

2.2.1 Covalent Functionalization

Several routes used to covalently functionalize CNTs include hydrogenation [52], electrochemical functionalization [53], thiolation [54], oxidative purification [55], halogenation [56], esterification [57], amidation [58], and cycloaddition [59]. Covalent functionalization allows functional groups to form covalent linkages on the carbon scaffold of nanotubes. CNTs inevitably contain defect sites during their production, which make the nanotubes susceptible to attack by reactive molecular groups. Acid treatment transforms defect sites into active molecular groups, such as COOH, C-OH, and C=O [51, 60] that are covalently attached to the CNT wall. Metal or metal oxide nanoparticles, charged polymer chains, and charged molecular groups can be covalently attached to these active sites, producing a strong bond in comparison to noncovalent functionalization, but at the expense of significant surface destruction.

2.2.2 *Noncovalent Functionalization*

The drawback of covalent functionalization is the damage done to the CNT structure. Noncovalent functionalization involves weak dipole–dipole interactions, such as van der Waals forces between the CNT surface and an external molecule. Different noncovalently functionalized molecules include those of polymers, metals, and biological materials. Polymer chains can be wrapped around a CNT wall [61] and biomaterials, such as DNA and proteins, can be conjugated to it [62–66]. Both covalent and noncovalent functionalization are exohedral, where functionalization occurs on the outside of the CNT wall [67].

The organization of aligned long CNTs improves the bulk properties of a composite material, e.g., by growing vertical and horizontal CNTs on catalyst coated substrates [68], production of CNT rolls through CVD [69], and spinning CNTs into yarns and sheets [70], but these methods are complicated and expensive. In the following section, different methods of decorating CNTs with MNPs, a more straightforward and inexpensive approach, are discussed along with the benefits arising from magnetized CNTs.

2.3 Covalent Functionalization of CNTs with MNPs

The remarkable thermal, mechanical, and electrical properties of CNTs have made them a promising material for a wide range of applications, including metal matrix composites, nanosensors, and reinforced polymer composites. These properties can be leveraged to form composite materials by embedding them into a polymer matrix [22, 71], particularly by aligning them in a particular direction [24]. CNTs are diamagnetic with a diamagnetic susceptibility χ of 10^{-5} emu g⁻¹ [72, 73], making them resistant to manipulation with a magnetic field. However, magnetizing CNTs with superparamagnetic nanoparticles enables such a response, e.g., by intercalating them within CNTs [74–77], and synthesizing MNPs on the walls of CNTs [78–80]. The synthesis of ferritic nanoparticles is a convenient method of magnetizing CNTs since it requires inexpensive reagents and uses common laboratory apparatus. Ferritic nanoparticles can be synthesized on CNTs using several routes, such as surface imprinting of magnetite (Fe₃O₄) crystals [81], hydrothermal decomposition of iron compounds [82, 83], and in situ co-precipitation of ferrite crystals [84–86]. Surface imprinting requires a high-temperature autoclave whereas hydrothermal decomposition requires both a high temperature (~250 °C) and an inert environment. Co-precipitation is thus the most convenient method among these for decorating CNTs with ferritic nanoparticles.

2.3.1 Co-precipitation of MNPs: Methodology

The co-precipitation of MNPs on CNTs is carried through two steps. CNTs are initially treated with strong oxidizing acids such as HNO_3 and H_2SO_4 . The NO_3^- and HSO_4^- radicals attack the C–C bonds at defect sites on the CNT surfaces that are formed during their production, forming functional groups, e.g., C=O, C–OH, and COOH. These active groups act as nucleation sites for the magnetite nanocrystals that are co-precipitated from a solution containing Fe^{2+} and Fe^{3+} ions [78, 87, 88].

A detailed and comparative study of decorating CNTs with magnetite nanoparticles using HNO_3 , H_2SO_4 , and a 1:1 mixture of HNO_3 and H_2SO_4 has been performed to determine the best practice to functionalize CNTs with minimum weight loss and higher magnetization density [89]. The effects of the intermediate stage of filtration and washing of the acid-treated CNTs are also investigated to understand its influence on the yield of the magnetized CNTs. Acid treatment of CNTs increases the oxygen content of the nanotubes. XPS analysis of different CNT samples with identical masses that have been treated with different acids is shown in Fig. 2.4. The increase in the oxygen content when CNTs are treated with a mixture of both acids is significantly higher than when individual acids are used for treatment, indicating severe structural damage to the CNTs, which consequently reduces their lengths. At the intermediate stage of washing, these shortened CNTs are washed away and lost to the filtrate. Attempts to skip the stage of washing during magnetizing CNTs resulted in acid residues in the sample which reacts with the iron ions in the solution.

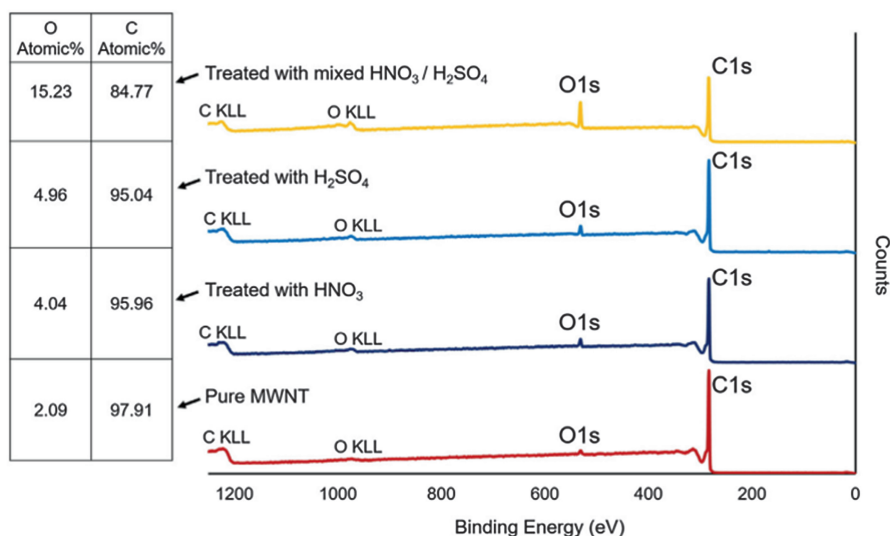


Fig. 2.4 Comparison of acid-treatment routes. XPS analysis shows that treatment with HNO_3 or H_2SO_4 leads to a moderate increase in oxygen content (4–5%) relative to pure CNTs (~2%). In contrast, treatment with a 1:1 mixture of the two acids produces a much higher (15.23%) oxygen content. Reprinted from [89] with permission from Elsevier

Table. 2.1 Yields of the various material phases, as a fraction of their stoichiometrically designed values

	HNO ₃	H ₂ SO ₄	(HNO ₃ :H ₂ SO ₄) 1:1 (v/v)	HNO ₃	H ₂ SO ₄	(HNO ₃ :H ₂ SO ₄) 1:1 (v/v)
Material phase	With the washing and drying stage			Without the washing and drying stage		
CNTs	90.2%	91.6%	54%	100%	78%	82%
Fe ₃ O ₄	100%	94%	85.2%	76.8%	31%	60%
mCNTs (total yield)	95.3%	92.8%	69.6%	88.4%	54.5%	71%
Fe ₃ O ₄ :CNTs (w/w)	1.11	1.03	1.58	0.77	0.39	0.73

The magnetization ratio γ is the ratio of Fe₃O₄:CNTs (w/w). The intended value of this ratio is unity for all samples. Reprinted from [89] with permission from Elsevier

As a consequence, undesired materials are found to be co-precipitated such as Akaganeite (Fe³⁺ O(OH, Cl), PDF No. 00-013-0157), magnetic iron oxide hematite (α -Fe₂O₃, PDF No. 00-032-0469), ferrous sulfate (FeSO₄, PDF No. 00-042-0229) which compromised the magnetization ratio (MNP:CNT) of CNT. Table 2.1 lists the yield of CNTs, ferrite MNPs, magnetized CNTs, and the final magnetization weight ratio (γ) at different stages of decorating CNTs with MNPs that includes the acid treatment, washing, and drying. The filtration and drying step was found to be necessary after the acid treatment to avoid the co-precipitation of unnecessary non-magnetic products.

The best decoration of CNTs with MNPs is observed when the CNTs are functionalized using either HNO₃ or H₂SO₄ and including an intermediate stage of filtration and drying. The functionalized mCNTs with the ferrite nanoparticles formed in the size range of 8.5–11.3 nm are superparamagnetic and their magnetic saturation is measured to be in the range of 34–38 emu g⁻¹ [89–91]. The covalently functionalized CNTs can be then directed and aligned using external magnetic field.

2.3.2 Co-precipitation of MNPs: Conductive and Magnetoresponse Colloidal Ink

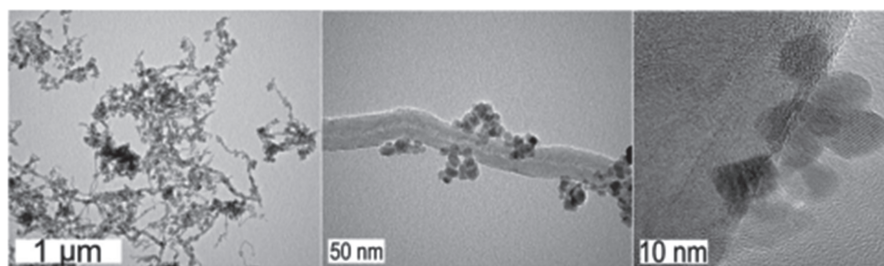
Nanofluids are colloidal dispersions of nanoparticles in a liquid [92]. Substantial effort has been made to increase the conductivity of liquids using conductive fillers, e.g., nanoparticles, ionic liquids, and nanotubes. Typically, conductive nanofluids are suspensions of gold, copper, and silver nanoparticles but these are expensive and have low oxidation resistance and only fair dispersion [93]. CNTs have superior electrical conductivity and it has been shown that upon addition of just 0.5% (w/w) of CNTs to an aqueous medium, the electrical conductivity increases by an order of magnitude [94]. Use of CNTs and graphene to prepare conductive colloidal suspensions has been reported but their agglomeration due to inter-CNT attraction poses a

serious problem [95–98]. Temporary dispersions of CNTs are achieved by ultrasonication [99] but CNTs need to be functionalized with surfactant molecules in order to achieve long-term stability [100]. Ferrofluids are a type of nanofluids consisting of MNPs that respond to a magnetic field [29, 30, 32]. MNPs coated with surfactant produce interparticle repulsion thus increasing their colloidal stability [97, 101]. Ferrofluids that can be manipulated using a magnetic field typically have poor electrical conductivity whereas CNT dispersions provide much higher electrical conductivity. A method to combine the properties of both CNTs and MNPs allows the magnetic manipulation of conductive colloids that is useful for printing electronic circuits and sensors.

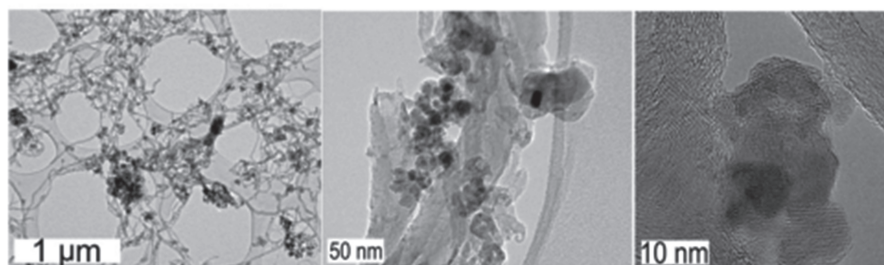
Magneto-responsive conductive colloids are synthesized by co-precipitating MNPs on CNTs and dispersing them in aqueous phase [102]. The material content of the MNPs and the magnetization weight ratio are varied to study their influence on both of the electrical and magnetic properties of the dispersion. The MNPs are placed on CNTs by co-precipitation [89]. Three different MNPs that have co-precipitated on the CNTs for the magnetization weight ratio of unity are magnetite (S1), Mn-Cu-Zn ferrite (S2), and Cu-Zn Ferrite (S3). These MNPs are attached to the outside surface of CNTs via covalent bonds. The nanoparticles co-precipitated on CNTs, of 10–15 nm size, successfully decorate CNTs as is evident from Fig. 2.5. The XRD analysis shows that all the samples are crystalline with spinel structure. As the magnetization ratio increases, the decoration density of MNPs on CNTs increases with the nanoparticles occupying the available activation sites until a certain value when all the sites are occupied. The excess metal ions are then co-precipitated over the deposited layer of MNPs perpendicular to the axis of CNTs.

Magnetite has a high Curie temperature whereas Mn-Cu-Zn ferrite and Cu-Zn ferrite have lower Curie temperatures [103–105]. The magnetic property of the colloidal dispersion containing magnetic CNTs (mCNTs) can thus be tailored by changing the material of the co-precipitated MNPs. The hysteresis curve for the three different mCNTs shows a complete absence of coercive field (Fig. 2.6a), indicating that the nanoparticles are superparamagnetic at the room temperature. The magnetic saturations M_s of the three specimens are measured to be 30.7, 10.5, and 16.6 emu g⁻¹ for S1, S2, and S3 respectively. The sensitivities of each specimen to varying temperature are different for each sample, as shown in Fig. 2.6b. This demonstrates how the magnetic properties of functionalized CNTs can be tuned using different nanoparticles.

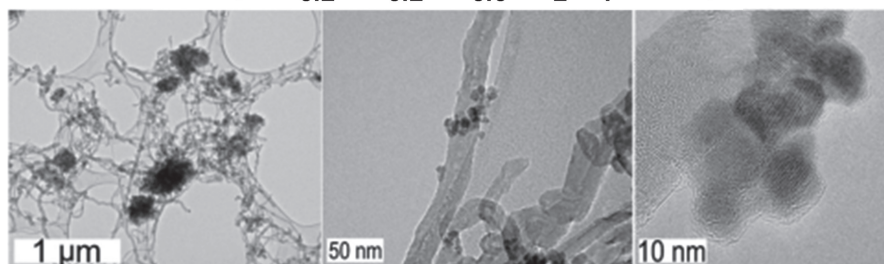
Tetramethylammonium hydroxide (TMAH: 25%) is used to suspend the functionalized CNTs in the aqueous phase. TMAH, on one hand, increases the electrical conductivity of the solution and, on the other hand, it acts as an ionic surfactant to stabilize the functionalized CNTs in a colloidal suspension. A schematic of the proposed rearrangement of the ions under the application of an electric field is shown in Fig. 2.6c. This polarization shortens the ion transport path and thus reduces the electrical resistance of the suspension. The addition of 4 wt% of the mCNTs results in increased electrical conductivity of the colloidal suspension (cf. Fig. 2.6d), and the increase is different for different nanoparticles. This methodology of preparing magneto-responsive and electrically conducting colloidal dispersions is useful for applications where a magnetic response must be coupled with the material electrical conductivity.



S1 (Fe₃O₄-CNT)



S2 (Mn_{0.2}Cu_{0.2}Zn_{0.6}Fe₂O₄-CNT)



S3 (Cu_{0.4}Zn_{0.6}Fe₂O₄-CNT)

Fig. 2.5 TEM images of samples S1–S3 (from top to bottom) confirm that all samples corresponding to $\gamma = 1$ are successfully decorated with highly crystalline (but different) MNPs, synthesized within the narrow size distribution of 10–15 nm. Reprinted from [102] with permission from Elsevier

2.4 Noncovalent Functionalization of CNTs with MNPs

2.4.1 Metal Plating of CNT

Functionalization of CNTs with MNPs allows their manipulation and alignment using a magnetic field. The electrical conductivity of the aligned CNTs still depends on the interfacial contacts between adjacent nanotubes. Researchers have

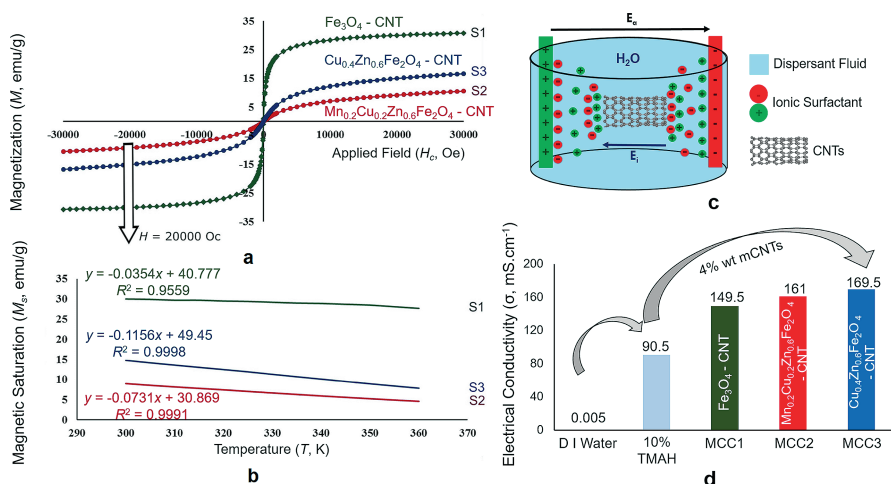


Fig. 2.6 (a) Magnetic hysteresis curves of the dry powders show no evidence of remanence, i.e., the mCNTs are superparamagnetic. (b) For all samples, M_s decreases with increasing temperature. Magnetite (S1) has the strongest magnetization and weakest sensitivity to temperature, Mn–Cu–Zn ferrite (S2) has the weakest magnetization while Cu–Zn ferrite (S3) has the strongest temperature sensitivity. (c) CNTs placed in an ionic medium between two electrodes charged by an electric field E_x polarizes and become oriented along the direction of the field. (d) Dissolving 10% (w/w) of tetramethyl ammonium hydroxide (TMAH) in DI water increases the electrical conductivity to 90.5 mS cm⁻¹. Dispersing 4% (w/w) of the different mCNTs in the TMAH solution enhances the electrical conductivity by 65–90%. Reprinted from [102] with permission from Elsevier

demonstrated deposition of metals or metal-based compounds over the nanotubes to decrease the contact resistance and also improve their resistance to oxidation [106]. Electroless plating of CNTs with metals like Ni, Cu, or Co can be performed under ambient conditions to produce uniform metal coatings. Electroless plating is a non-destructive and rapid method to deposit metal, which is initiated by activation-sensitization process that introduces catalytic nuclei on the surfaces of CNTs. A reduction reaction for the metal ions on this new catalytic surface forms a coating layer composed of metal. The metal-coated CNTs thus possess superior hardness, wear resistance, high electrical conductivity, and magnetic properties. A continuous and uniform layer of nickel can be electroplated on the surface of CNTs in a water bath without sensitizing or activating the surface with catalysts [107].

We have provided a controlled process for plating nickel over CNTs using electroless plating for different weight ratios of Ni:CNT (γ_c) [108], the schematic of which is shown in Fig. 2.7. Sensitization of CNTs using tin ions followed by activation with palladium ions reduces the nickel ions to nickel, which is deposited on the CNT outer surfaces. Nickel then acts as auto-catalyst that continues to reduce nickel ions until their complete depletion from the solution. This allows us to control the thickness of the deposited nickel layer over the MWNTs and thus their material properties.

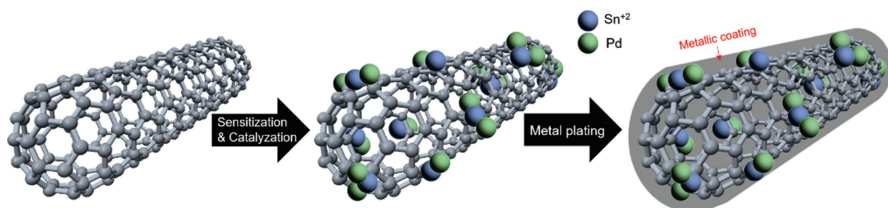


Fig. 2.7 CNTs magnetized with Ni by electroless deposition. (a) MWNTs were catalyzed through two chemical treatment steps using acid solutions of SnCl_2 for sensitization and PdCl_2 for activation. Electroless deposition of Ni on the resulting activated MWNTs used a plating solution containing nickel salt and reducing agent, where nickel ions accept electrons from the reducing agent to form metallic nickel through metal reduction

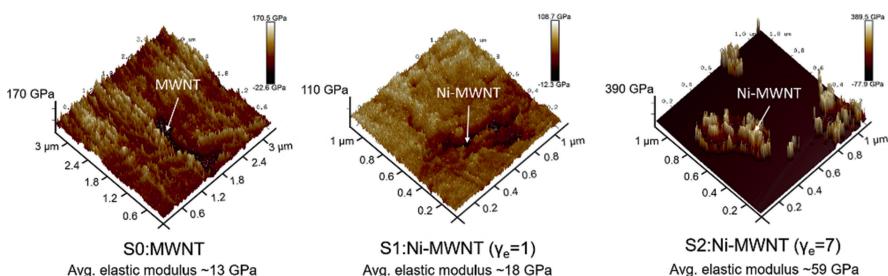


Fig. 2.8 AFM mechanical sketch up of samples S0, SN1, and SN2. Pure MWNTs (S0) had an average elastic modulus $E \sim 13$ GPa while Ni-MWNT samples for $\gamma_e = 1$ and 7 had values of $E \sim 18$ (46% increase) and 59 GPa (370% increase), respectively. Increasing Ni:MWNT weight ratio γ_e enhances the measured modulus

The morphology and thickness of the deposited layer of nickel and its mechanical and magnetic properties depends on the Ni:CNT weight ratio in the reacting mixture. The magnetic property of the hybrid nanomaterial increases with nickel weight fraction. As γ_e increases from 1 to 7, the magnetic saturation (M_s) and remnant magnetization (M_r) increase from 4.1 and 0.51 emu g^{-1} to 9.5 and 1.01 emu g^{-1} respectively. The average elastic modulus E of the hybrid CNTs ($\gamma_e = 7$) measured using AFM in the radial direction shows a threefold increase over that for pristine CNTs. Figure 2.8 shows the effect of nickel deposition on the elastic modulus of the CNTs, providing a solution to control it. The increase in elastic modulus is attributed to the thickness of the nickel deposit over the CNT surfaces, which increases with γ_e .

2.4.2 Physical Attachment of MNPs on CNT

CNTs can be decorated by ferrites nanoparticles, which improve the optical, magnetic, and electrochemical properties of pristine CNTs. The vapor deposition of Ni atoms allows decoration of nickel nanoparticles on the CNT surfaces. The process

can be continued longer to coat the CNT with a layer of Ni [109]. The CNT surfaces can be sensitized and catalyzed to further reduce the metal ions to form nanoparticles. Both metal plating and physical attachment of MNPs onto the surface of CNTs require several steps that are time-consuming and laborious. Each method of functionalization has its advantages but CNT manipulation with a magnetic field is the objective, a simple one-step method is more desirable in comparison to a longer chemical route.

2.4.3 Entanglement of NiNP in CNT Network

Covalent functionalization of MNPs on CNTs often requires expensive chemicals and an experienced skillset. Entanglement of nickel nanoparticles (NiNP) in the matrix of CNTs using probe sonication is another novel method to create a magnetic ink [110]. Sonication of a dispersion of NiNPs and CNTs in kerosene (Fig. 2.9a) allows nickel to become entangled within the CNT network as shown in the TEM image (Fig. 2.9b). The high surface energy of NiNPs and pi-interactions are responsible for the clustering of NiNPs and their conjugation with CNTs. The conjugated NiNP-CNT material responds as a bulk to a magnetic field, which is evident from Fig. 2.9a. The conjugated material shows moderate magnetization saturation value of 14.61 emu g^{-1} (Fig. 2.9c). The nanoparticles are individually superparamagnetic but with a coercive field the material becomes ferromagnetic due to the entanglement of the nanoparticles responsible for inter-NiNP magnetostatic interactions for particles in close proximity. Prolonged sonication in most cases does not result in more homogeneously distributed NiNPs and thus is not necessary. Therefore, adequate magnetization usually occurs within a few minutes. For this reason, mechanical magnetization allows for rapid entanglement of NiNPs and offers one of the quickest methods to magnetize CNTs while forgoing harsh chemical pre-treatments familiar to other magnetization routes.

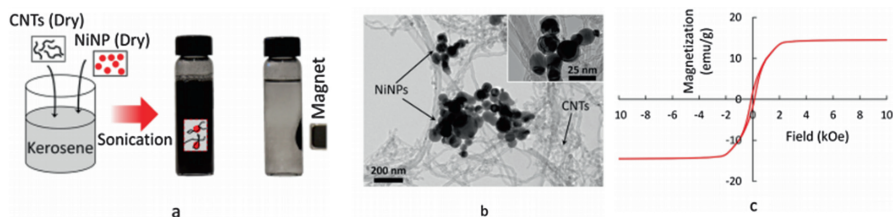


Fig. 2.9 Nickel nanoparticle entangled carbon nanotubes. (a) NiNPs and CNTs are dispersed in kerosene by probe sonication. The CNTs entangle the NiNPs, enabling the latter to act as magnetic chaperones without detachment or separation in a strong gradient magnetic field. (b) TEM images show clusters of NiNPs enmeshed in CNTs. (c) SQUID magnetometry shows that the NiNP-CNT, containing 33% (w/w) NiNPs, have a saturation magnetization $M_s = 14.61 \text{ emu g}^{-1}$, which is commensurate with the mass fraction of nickel in the sample. Reprinted (adapted) with permission from [110]. Copyright (2016) American Chemical Society

2.4.4 Printing Sensors with Magnetized CNT Ink

We printed the synthesized magnetic ink into U shapes over polydimethylsiloxane (PDMS) surface using an iron template and a permanent magnet. The iron template placed on the permanent magnet concentrates the magnetic lines of force, producing a high gradient magnetic field. This field concentrates the NiNP-CNTs and aligns them along the magnetic lines of force (cf. Fig. 2.10a). On drying of the solvent in the magnetic ink, the NiNP-CNTs are embedded in the PDMS matrix (cf. Fig. 2.10b). The embedded U-shaped NiNP-CNT network on PDMS forms a continuous electrically conductive path, which we tested as an alternative to flexible sensors. It is an alternative to rigid conducting wires and appropriate for wearable electronics [111]. An easy, inexpensive, and facile fabrication method for flexible sensors using NiNP-CNT networks is used to fabricate strain gauge [112] and detect chemical and biological species [113, 114].

A simple voltage divider circuit is used to determine the response of the U-shaped NiNP-CNT network subjected to mechanical deformation, including both bending and elongation. The resistance of the NiNP-CNT circuit increases as the space between the interconnects increases and the percolation of the CNT network

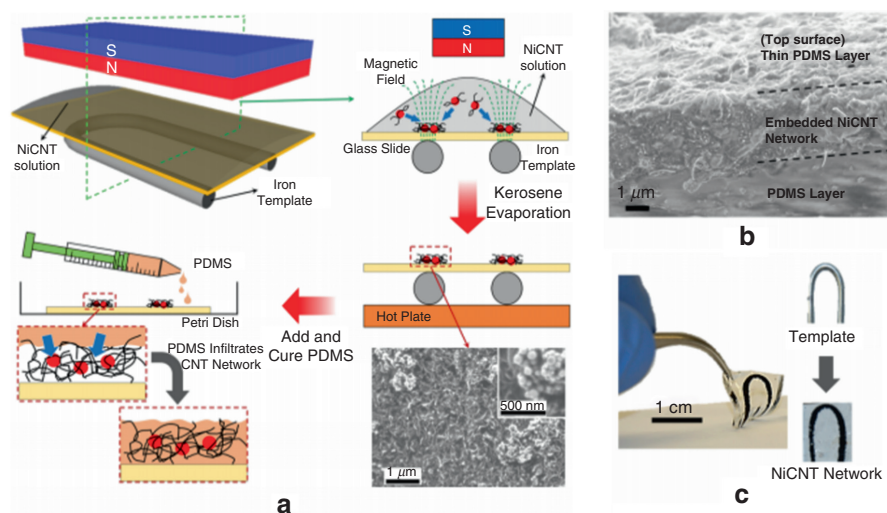


Fig. 2.10 Magnetic printing with NiNP-CNT: (a) A U-shaped soft magnetic wire is used as a template. The wire produces localized gradients in the magnetic field, which settles the dispersed NiNP-CNT on the coverslip surface immediately adjacent to it. The kerosene is then evaporated by heating, yielding a U-shaped dense network of CNTs. The NiNP-CNT U network is placed inside a Petri dish, covered with liquid PDMS, and heated to cure the PDMS. The PDMS infiltrates the printed structure, and after curing, it lends a polymer matrix to the NiNP-CNT. (b) SEM image of a cross-section of such a structure reveals a $\sim 2.5 \mu\text{m}$ thick NiNP-CNT-PDMS composite network embedded in pure PDMS. (c) When the PDMS is peeled off, it forms a flexible and stretchable membrane with an embedded NiNP-CNT structure. Reprinted (adapted) with permission from [110]. Copyright (2016) American Chemical Society

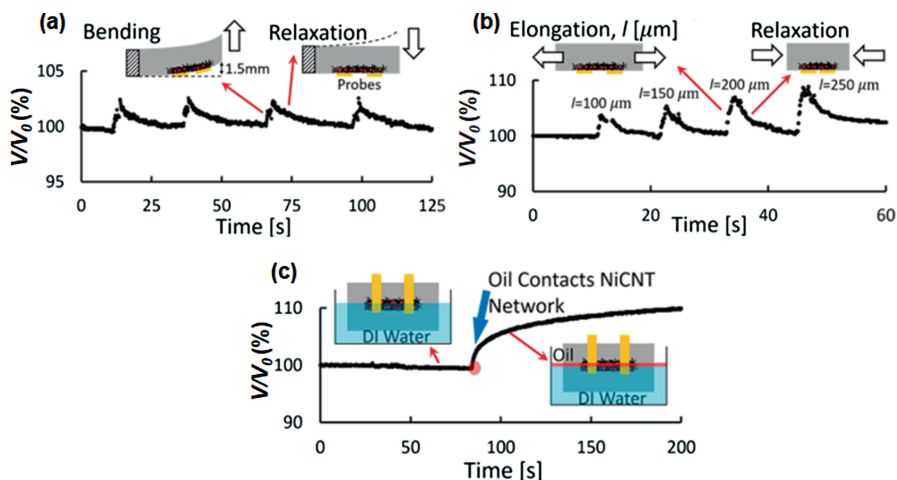


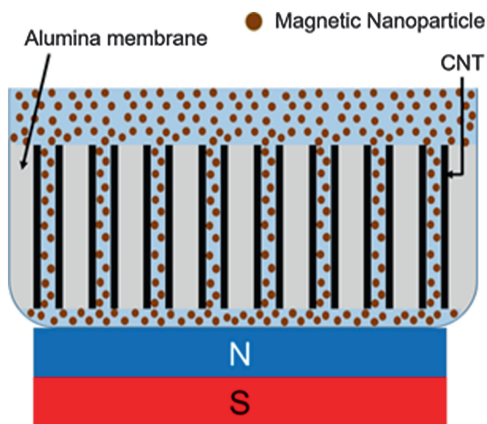
Fig. 2.11 The voltage drop V across the NiNP-CNT network rises when the block is subjected to (a) bending by displacement of the free end or (b) elongation of $l = 100\ \mu\text{m}$, $150\ \mu\text{m}$, $200\ \mu\text{m}$, and $250\ \mu\text{m}$ and (c) when the sensor is held at the air–water interface in a beaker and $\sim 1\ \text{mL}$ of oleic acid is dropped on the water, V rises when sensor contacts the oil. Reprinted (adapted) with permission from [110]. Copyright (2016) American Chemical Society

decreases. This increases the voltage, as shown in Fig. 2.11a, b. As the PDMS is relaxed, the distance between the interconnects reduces and the voltage gradually returns to its initial value. Continuous and cyclical mechanical deformation of the network leaves a permanent resistance change, as evident from Fig. 2.11b where the steady voltage on relaxation increases over the cycle. The printed NiNP-CNT network also responds to the presence of oil since the PDMS, being oleophilic, absorbs oil, affecting the percolation of the CNT networks and thus changing the resistance of the circuit (cf. Fig. 2.11c). This method of printing sensors with magnetic ink is made possible by combining the unique properties of MNPs and CNTs using non-covalent functionalization. The technique can be readily scaled up and integrated with existing nozzle-based printing of flexible circuitry and sensors that have complex geometries.

2.5 Endohedral Functionalization of CNT with MNPs

A CNT is a nano-sized container that has a protective carbon shell to encapsulate nanomaterials within. Thus a CNT is a smart nanoscale carrier that can be filled with tailored materials for target applications such as memory devices, optical transducers, wearable electronics, and medicine. MNPs can be intercalated inside CNTs during the synthesis process using the chemical vapor deposition of metal-locenes [76, 115]. External molecules are encapsulated by a capillary effect inside

Fig. 2.12 Intercalation of magnetic nanomaterial inside CNTs. A schematic describing encapsulation process driven by the capillary effect and accelerated with a magnetic field



the CNTs [61]. MNPs can also be added to a CNT dispersion and encapsulated within the nanotubes. The solvent carrying MNPs wets the inside volume of CNTs and, after drying, the MNPs remain encapsulated as shown in Fig. 2.12.

2.6 Future Outlook

The covalent and noncovalent functionalization of CNTs with MNPs has produced a new class of nanoscale that exploits the unique properties of both CNTs and MNPs. MNP-functionalized CNTs can be used to prepare polymer composites and bulk materials with enhanced mechanical, electrical, and thermal properties. The properties can be tuned, depending on the direction of alignment of the CNT network. The materials can also potentially be used in energy storage applications such as supercapacitors [116] and lithium ion batteries. Functionalized CNTs have high sensitivity toward the respective external stimuli which could be used to develop custom cost-effective sensing platforms. Environmental sustainability demands the use of less hazardous functionalizing agents which could replace the acids that are currently used to functionalize CNTs. Crack patterns of the deposited magnetized CNTs need further attention which might reduce the critical percolation threshold for the sensing activity.

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