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Materials made of carbon nanotubes. The carbon nanotube forest

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Abstract. Publications concerning research on unique material made of carbon nanotubes (CNTs) aligned perpendicular to the substrate surface, namely, CNT forest, are analyzed and summarized. The morphology, volume and surface density of CNT forest, methods of its synthesis and modification, as well as the properties and potential fields of application are considered. The bibliography includes 679 references.

I. Introduction

Carbon nanotubes (CNTs) and carbon nanofibres † recently have become industrial goods. They serve a basis for and are components of various novel materials. Production and application of CNTs and materials based on them is an industry and the design of such systems has become a particular branch of materials science. There are two groups of materials designed in the last 10 – 15 years:[1, 2]

— materials containing CNTs (nanocomposites) and

— materials made of CNTs.

The first group includes diverse nanocomposites based on industrial polymers. There is considerable literature on the topic, including more than 1000 publications. This group also includes composites based on metals and alloys, ceramics, concrete, glasses, biocomposites and hybrid materials. The content of fillers in them is at most 3 mass % – 5 mass %; often, the mechanical, transport or some other properties of the matrix can be improved by introducing much smaller amounts of additives.

The second group includes materials made of CNTs containing small amounts of binders (at most 3 mass % – 5 mass%) or no binders at all. It also includes nanopaper (nano-felt), *i.e.*, mats made of entangled CNTs, thin CNT coatings, CNT fibres and CNT aerogels.

This review is devoted to a material from the second group, *viz.*, the CNT forest in which nanotubes are aligned normal to the substrate surface, similarly to silicon and other semiconductor nanowires fabricated by the VLS (vapor – liquid – solid) process. The properties of CNT forests are versatile, methods of synthesis are easier than the VLS process; this offers great prospects for applications of this unique material.

Vertically aligned carbon nanotubes are comprehensively considered in a review [3] which includes sections devoted to the synthesis of vertically aligned multi-walled and single-walled CNTs, to micropatterning of vertically aligned multi-walled CNTs and to modification of vertically aligned CNTs. The template and template-free methods of synthesis of multi-walled CNTs and the supergrowth technique are considered. The emphasis is placed on micropatterning of CNT arrays using various lithographic techniques and multicomponent micropatterns.‡ Information on the physical properties and applications of aligned CNTs was also generalized [4] (see §). A comprehensive review [5] on mass production of CNTs also touched the synthesis of vertically aligned CNT arrays. A difficult-to-access study,[6] a number of sections in a review [7] and a report [8] are now out of date.

The author of this review tried to cover as many recent publications as possible because of increasing interest in CNT forests.

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† For the sake of simplicity, from this point on we draw no distinction between CNTs and carbon nanofibres.

‡ In that review, the bibliography includes 278 references. Among them, about twenty references date to 2009 and only one reference dates to 2010.

§ The bibliography includes at most 12 references dating to 2010.

II. The morphology and density of carbon nanotube forest

Various kinds of CNT forest differ in, *e.g.*, diameter and number of graphene layers in CNTs, CNT size distribution, as well as in length and surface density of CNTs. Carbon nanotube forests may include 'regular',[3] bamboo-like,[9–11] helical[12,13] and branched structures (see below). Often, the top of a CNT forest differs from its base part and represents a network of entangled CNTs. In some case, CNT forests may contain small CNT bundles comprising at most ten CNTs[14,15] or having the average diameter smaller than 16 nm.[16] Plasma-activated pyrolysis on a Ni–Mo catalyst results in cone-shaped CNT bundles whose mass (v) and surface (ρ) densities are interrelated[17]

$$v \propto \rho^{-3/2}$$

A CNT forest-on-forest can be grown. The number of layers in such a system can vary from one (see, *e.g.*, Refs 18 and 19) to two,[20] four,[21] eight[22] and even forty.[23] Layered structures are usually obtained by floating catalyst pyrolysis[24,25] (see below), but they can also be synthesized with immobilized catalyst using pulsed delivery of the feedstock gas or by substrate cooling between the deposition cycles.[26,27] It is noteworthy that each new layer can be formed under the preceding layer, thus lifting it.[22,27,28]

Second- and third-order CNT forest structures with branched 'stems' (Fig. 1) were reported.[29] Second-order CNTs were grown by the pyrolytic method on the catalyst deposited on pre-grown first-order CNTs. Third-order CNTs were grown analogously. Under particular conditions, the floating catalyst synthesis gives a specific texture of the CNT forest, where the CNT tops are packed more densely than the bases and form honeycomb structures.[30]

Nanotubes in CNT forests are not perfectly aligned to one another. Depending on the height of CNT forest (film thickness), growth conditions, the CNT diameter and surface density, most CNTs are to some extent tortuous.[31–37] Therefore, the length of some CNTs can exceed the height of the CNT forest. Moreover, different CNTs in the same nanotube forest often differ in length and tortuosity.

Carbon nanotube-array double helices[38] and conical structures[39] arranged normal to the substrate surface can also be treated as related to CNT forest.

The layer number distribution of CNTs can be determined by X-ray diffraction from the character of the (002) reflection in diffraction patterns.[40]

The diameters (d) and surface densities of CNTs reported in different studies are presented below.

Ref.	41	42	43	44	45	46
d/nm	90	50	44	24	10	< 5
ρ/cm^{-2}	10^{10}	7×10^{8}	3×10^{10}	10^{12}	1.53×10^{10}	10^{12}

The lowest surface density is close to 5×10^{8} cm^{-2} (see Ref. 47) and even to $10^{6}-10^{8}$ cm^{-2} in the case of electrochemical deposited catalyst (see Refs 48 and 49). Even lower surface density of CNT forest can be attained using photolithographic methods.[50] Yet another procedure for obtaining a low-surface-density CNT forest involves its transfer from one substrate to another.[51] Carbon nanotube forests with $\rho > 10^{12}$ cm^{-2} are difficult to grow.

Figure 2 presents the surface density of CNTs in nanotube forests plotted *vs.* CNT diameter.[52–57] High surface density was attained[52,55] by step-by-step deposition of catalyst, *viz.*, deposition of a Fe film was followed by thermal annealing to form nanoparticles and subsequent oxidation of the nanoparticles; then, these steps were repeated. Aluminium deposition can be used instead of iron oxidation. Iron oxide was reduced prior to growing a CNT forest. A method of multistep growth to obtain CNT arrays with high surface density was developed.[53] In this case, the volume occupancy of multi-walled CNTs can be as high as 30% – 40% of the total volume, being nearly half the theoretically possible value.

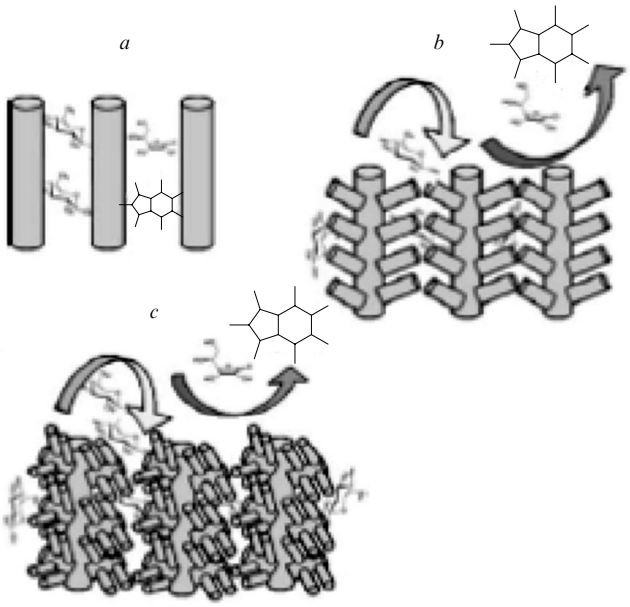


Figure 1. A scheme of successive growth of branched CNT forest.[29]
First-order (*a*), second-order (*b*) and third-order (*c*) CNTs.

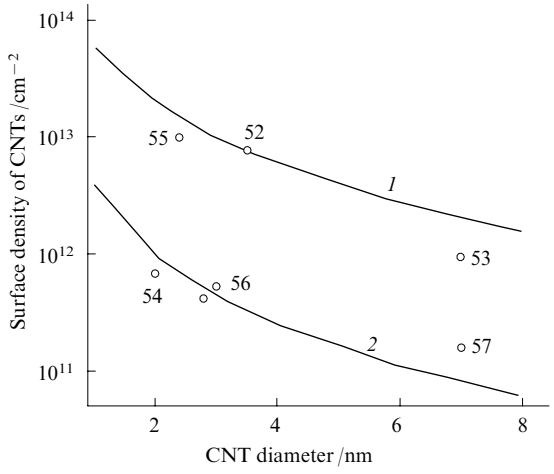


Figure 2. Surface density of CNTs in carbon nanotube forest plotted *vs.* CNT diameter.
Maximum (*1*) and typical (*2*) surface density; numbers near open circles are the numbers of references.

Carbon nanotubes grown on aluminium sublayer on silica, tantalum and titanium with Fe films as catalysts have higher surface density and smaller diameters; this was explained by mixing of iron and aluminium.[58]

The bulk occupation of CNTs in a nanotube forest is usually ~1% – 2%, being at most 10%.[57, 59, 60] The apparent density of single-walled CNTs in nanotube forest is 1.2 cm^{-3}; it increases upon heating in air or in CO_2 atmosphere and can reach a value of 2.2 g cm^{-3}.[61] The density of a single-walled CNT forest grown by the supergrowth method (see below) is 0.037 g cm^{-3}, its surface density is 5.2×10^{11} cm^{-2} and CNTs occupy 3.6% of the substrate surface area.[62] The density of double-walled CNT forests can be low, namely, 0.015 g cm^{-3} (Ref. 63) or even 0.009 g cm^{-3}.[64] High density of a nanotube forest (0.06 – 0.07 g cm^{-3}) was also reported.[54]

Oxidative annealing favours uncapping of CNTs (removal of 'caps' at nanotube ends) and an increase in their specific surface area from 650 to 1900 m^2 g^{-1}. Changes in selected parameters of CNTs upon thermal annealing are illustrated in Fig. 3.

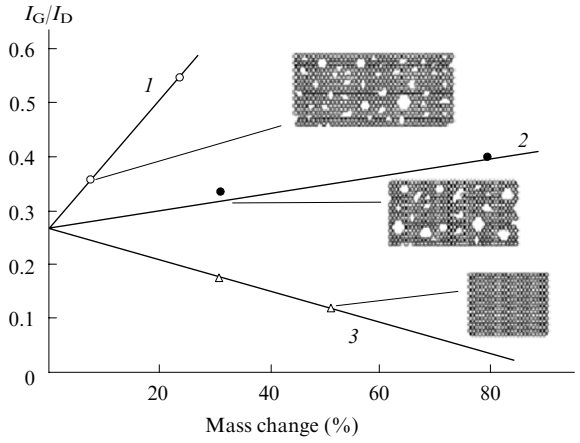


Figure 3. I_G/I_D Raman mode intensity ratio plotted *vs.* change in mass: heating in air at 723 (*1*) and 823 K (*2*); heating in CO_2 at 1223 K (*3*).[61]
Insets: unrolled CNTs with estimated pore sizes. The I_G/I_D ratio characterizes the degree of disordering of carbon nanotubes in CNT forest.

The surface of nanotube forests is often covered with a 'crust' formed by entangled CNTs.[65 – 67] It is typical of both individual CNTs and CNT micropillars grown from catalyst islands lithographically patterned on the substrate surface (Fig. 4). If the surface density of CNT micropillars is low, the 'crust' layer is formed in the initial stages of growth. Regularities of growth of CNT pillars were documented.[67] Horizontal cross-sections of CNT forest exhibit different interlayer distances in multi-walled CNTs, as well as different number of layers and degree of ordering.[68]

Lithographic patterning of catalysts makes it possible to grow various configurations of CNT forests (see, *e.g.*, Refs 69 – 73). In particular, cylindrical pillars 2, 5 or 10 μm in diameter arranged in ordered manner can be formed.[74] Polymers can also be used for catalyst patterning.[75] The morphology of structures formed from CNT

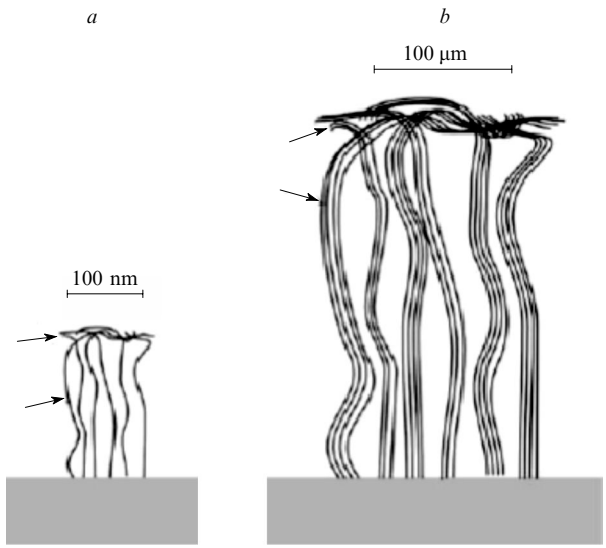


Figure 4. Formation of surface 'crust' from individual CNTs (*a*) and CNT pillars (*b*).[67]

forests is affected by the geometry-related gas mixture flow.[76]

Arrays of identically bent CNTs and zigzag-shaped CNTs were reported.[77] A method for determination of the surface density of CNTs in a carbon nanotube forest was proposed.[78]

III. Methods of synthesis of carbon nanotube forests

A CNT forest can be grown in different manner, *viz.*, by catalytic pyrolysis on supported catalysts; using various versions of the CVD method (see, *e.g.*, Refs 21, 28, 79 and 80); by the template method with Al_2O_3 membranes; by graphite sputtering (this technique is used much more rarely),[11] by electrophoresis or dielectrophoresis of CNTs from dispersions[81, 82] and by chemical grafting of CNTs onto substrates.[83 – 87] The last-mentioned method (see a review[86]) is much more labour-consuming and leads to structures whose properties differ from those of pyrolytically grown CNT forests. Attempts to synthesize a nanotube forest by the molecular beam technique led to success only when acetylene diluted with hydrogen and helium was used, although a total of 12 hydrocarbons were tested.[88]

Pyrolysis using unpatterned catalyst has the following advantages over the synthesis using powder or floating catalysts:[89]

— much higher purity of CNTs (this excludes purification steps);

— simpler control of CNT diameter;

— the possibility of fabrication of diverse structures from CNT arrays for immediate utilization in various devices; this makes the technological chain much shorter.

The pioneering study on a CNT forest grown spontaneously normal to the substrate surface was reported in 1997.[90] That work as well as Refs 91 – 96 started a long chain of similar and improved research. It was established that CNTs most often grow from the base, *i.e.*, catalyst particles remain on the substrate surface and alignment of CNTs normal to this surface is due to restrictions imposed

on CNT growth along other directions by adjacent CNTs; van der Waals interactions between carbon nanotubes impart high rigidity to CNT arrays.[7,97]

A superaligned CNT forest was synthesized in 2002;[98] CNT forests on 4- and 8-inch silicon wafers were grown in 2005[99] and 2008,[100] respectively.

Carbon nanotube forests were synthesized using catalysts of different compositions (mostly containing Fe, Co, Ni) including bimetallic[101–103] and unusual catalysts such as Fe–V–O nanoparticles[104] or magnetic alloys $Fe_{19}Mn_{80}$, CoCrPt, *etc.*[105] The authors of many studies utilized layered catalysts (see, *e.g.*, Refs 106 and 107 and a review[108]). Catalysts were prepared using atomic layer deposition,[109] by abrasion of substrate[110] and by local oxidation of substrate using an atomic force microscope probe.[111]

The longest CNTs (1 mm and longer) were obtained using Fe catalysts only. At 450 °C, iron particles on amorphous carbon surface are transformed to Fe_3C and then become coated with a number of graphite layers.[112] A TEM study revealed that graphite layers are formed on carbide edges, propagate across them and then penetrate the CNT cap.[113] The particle becomes highly deformed and 'ejects' the CNT. It is assumed that high catalytic activity of iron in the CNT growth is associated with the unfilled d-orbital, which favours cleavage of bonds in the starting hydrocarbon and formation of unstable carbide. The latter acts as carbon-transfer agent and deposits it in the form of CNTs.[114] A similar carbide cycle mechanism was earlier proposed and studied by researchers from the scientific school headed by the Corresponding Member of the Russian Academy of Sciences R A Buyanov (Novosibirsk, Russian Federation).

The substrate can be coated with the catalyst both prior to depositing CNTs and during the deposition. The latter case is known as the floating catalyst approach. Here, the catalyst is formed by thermal decomposition of, *e.g.*, ferrocene,[115–117] nickelocene, iron carbonyls or iron phthalocyanine.[23,118] Catalyst precursors are vaporized individually or used as solutions in benzene, toluene, xylene or other organic solvents. The floating catalyst techniques are utilized to fabricate structures based on CNT forests (including CNT forests of different height) on patterned substrates.[119]

When using supported catalysts, the CNT diameter and number of CNT layers and the morphology of the CNT forest are determined by the thickness of the deposited catalyst layer,[54,60,120–128] by the catalyst particle size[129] and by the character of pretreatment of the catalyst. Some trends are illustrated in Fig. 5.

The CNT diameter does not necessarily depend on the thickness of the deposited catalyst layer, namely, three- and double-walled CNTs were obtained at a Fe film thickness of 0.8 and 0.4 nm, respectively; no single-walled CNTs were synthesized at an iron layer thickness of 0.2 nm.[123] The authors cited reported the effect of orientation of MgO single-crystalline substrate on the growth of CNTs, namely, under identical conditions, CNTs grown on the (110) plane were 20% and 55% longer than those grown on the (100) and (111) planes, respectively. This can be due to different binding energies of iron to the MgO surface of different orientation.

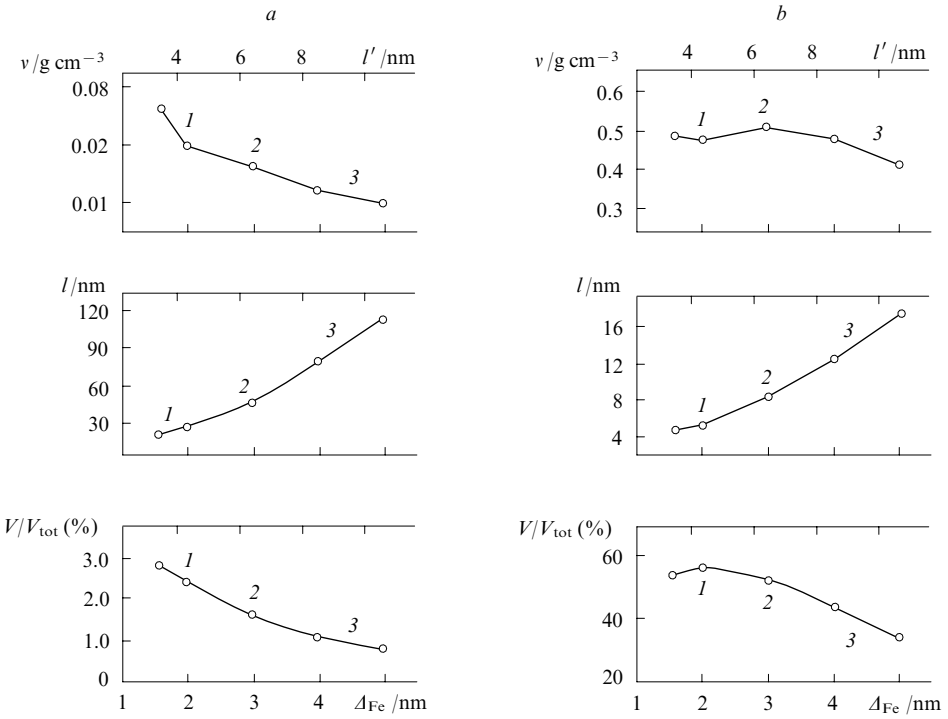


Figure 5. Structural properties of CNT forest (*a*) and capillary densified CNT forest (*b*) plotted *vs*. Fe film thickness (Δ_{Fe}).[60]
Single- (*1*), double- (*2*) and multi-walled CNTs (*3*); *v* is the mass density, *l* is the initial CNT spacing, l' is the CNT spacing after densification, *V* is the volume of CNTs in CNT forest and V_{tot} is the total volume of the CNT forest.

Taking a Mo/Fe/Al catalyst as an example, it was shown that the morphology and growth rate of CNT forest are determined by the metal ratio in the catalyst.[130, 131]

To increase the surface density of CNTs, it was proposed to utilize an $Al_2O_3/Fe/Al_2O_3$ layered catalyst with layers 0.5, 0.5 and >5 nm thick, respectively.[54, 106] A method was developed to determine the forest density depending on the growth conditions.[132] The morphology of a CNT forest grown on silicon is affected by the aluminium buffer film a few nanometres thick.[133] Under optimum conditions, the growth rate of almost perfectly aligned CNTs reaches a value of 87 μm min^{-1}.

Contrary to this, a weak dependence of CNT forest height on the Al_2O_3 film thickness was reported.[125] It was proposed to deposit a thin Al_2O_3 film to make the properties of the coating more reproducible.[134] At low surface concentrations of catalyst particles, a random network of CNTs parallel to the substrate surface ('grass') is formed instead of a CNT forest.[101]

The number of layers, their total height, diameter and the character of CNT alignment depend on the duration of preliminary reduction of catalyst in hydrogen stream. In optimal conditions, a double-walled CNT forest of height 9 mm was grown in 10 h.[135] Treatment of a Fe/Al film in NH_3 atmosphere leads to its transformation to nanoclusters; this makes it possible to grow CNT forests at 350 – 500 °C.[136] Etching of Ni-catalyst with NH_3 plasma influences the growth of CNT forest similarly.[137]

By varying the conditions of catalyst pre-treatment with hydrogen, the average CNT diameter can be doubled (from 7.4 to 13.6 nm), the surface density can be increased by an order of magnitude from 3.9×10^9 cm^{-2} to 4.9×10^{10} and the growth rate can be increased from 32 to 154 μm min^{-1}.[138] Pre-treatment of a deposited catalyst with acetylene at low partial pressure of the hydrocarbon (12 Pa) causes the catalyst particle size to decrease and the CNT surface density to increase.[139] Catalyst patterning for plasma-assisted growth of CNT forests can be performed using polystyrene-*block*-poly(methyl methacrylate) templates.[140] A photothermal method for growth of a CNT forest with an outer CNT diameter of ~10 nm and an inner diameter of 5 – 6 nm at 370 °C was proposed.[141]

In addition to catalyst deposition, immersion [142 – 149] and spin-coating followed by drying and reduction of oxides are used. The immersion technique was, in particular, utilized to coat a polished quartz substrate with a catalyst in a solution of cobalt and tungsten acetates (their concentrations were 0.01 mass %)[142] and to coat a silicon substrate by immersion into a dispersion of centrifugally classified $Fe_{80}V_{20}$ particles.[145] Spin-coating was done with a magnetic liquid, namely, stabilized dispersion of iron oxides.[150] A single-walled CNT forest was grown using a catalyst whose particles were produced by deposition from a colloid solution.[151, 152] The number of deposited iron nanoparticles was exactly controlled.[151]

Catalyst particles necessary to grow double-walled CNTs were deposited by laser ablation and using a pulsed arc deposition system.[153]

Co-deposition of titanium and cobalt onto a silicon substrate excludes the formation of the surface oxide layer because titanium precludes the formation of cobalt silicide.[130]

Various configurations of CNT forests are obtained using photolithography (see, *e.g.*, Ref. 154), by masking of substrates,[155] by coating of substrates with layers of self-assembling microspheres,[50] by local hydrophobization and subsequent silver plating of the surface,[156] by creation of hydrophobic and hydrophilic regions on the substrate surface[148] and by catalyst printing with polysiloxane stamps.[157]

The CNT diameter also depends on the gas feed rate.[158]

Carbon nanotube forests can be grown on substrates of different shape and composition, *e.g.*, single-crystalline silicon [89, 159, 160] including silicon wafers;[99, 100, 161 – 164] porous silicon,[161, 165, 166] aluminium,[167] alumina-coated silicon or quartz,[168] rolled aluminium foil,[169] on nickel,[170] foamed nickel, chromium,[161] copper,[105, 171] metallic foil and wires (no metal was specified),[172 – 175] Inconel 600 alloy,[118, 173] stainless steel foil,[176] stainless steel mesh,[177] bronze cantilevers,[178] foamed FeCrAlY alloy,[179] titanium,[180] $CoSi_2$,[181] single-crystalline MgO,[123, 182, 183] glass,[91] quartz and Al_2O_3,[184] (see also Refs 185 – 187), graphite,[188] carbon fibres and carbon fabrics,[189 – 194] carbon paper,[195, 196] glassy carbon,[197, 198] quartz fibres,[109, 199] SiC fibres,[200] ceramic fibres and fabrics,[201 – 203] Si_3N_4,[161] ceramic spheres,[199, 204 – 208] sapphire,[123] GaAs,[105] *etc.* However, no CNT forest was grown on Al_2O_3 or ZrO_2,[204] as well as on $SrTiO_3$, $LaAlO_3$ or stabilized ZrO_2.[123] Most often, CNT forests grow on silicon coated with a thin Al_2O_3 film due to very low surface diffusion coefficients of Al_2O_3 and iron.[209]

Carbon nanotube growth on various metals and alloys was studied.[210] Noteworthy are publications concerned with the synthesis and properties of CNT forest – multilayered graphene [211] and CNT forest – shape memory alloy [212] flexible hybrid films. The method for growth of a CNT forest on a pyrolyzed photoresist film can be regarded as versatile.[197] A poorly ordered CNT forest was grown on mesoporous SiO_2.[213] A study on floating catalyst synthesis of CNT forest from hexane (with ferrocene as catalyst) at 770 – 790 °C revealed selective influence of the substrate, *viz.*, growth was observed only on the oxidized silicon or silica rather than single-crystalline silicon.[78]

A large number of studies devoted to plasma-enhanced synthesis of nanotube forest have been reviewed.[214] Also, by specifically orienting the substrate in the plasma discharge zone one can fabricate a tilted CNT forest.[215]

So-called odako (big serpent in Japanese) growth of a CNT forest involves deposition of a Fe film 1 nm thick on grafoil (material fabricated from exfoliated graphite) or on carbon fibres followed by the deposition of an Al_2O_3 film 5 nm thick and subsequent pyrolysis of a mixture of C_2H_2, H_2 and water vapour at a reduced total pressure.[216] Carbon nanotube forest grows between the substrate and the Al_2O_3 film which thus becomes separated into individual folded fragments.

The CNT forest morphology depends on the substrate properties.[186, 217] In the case of strong interaction between catalyst particles (*e.g.*, Fe/Al_2O_3) and substrate, the particles are immobilized and favour the base growth of CNT forest. Here, catalyst particles remain on the substrate and the nanotube ends are free. If the interaction is weak (SiO_2 substrate), catalyst particles diffuse across the surface; this leads to formation of 'grass' and to tip growth of CNTs at which catalyst particles move with the tip of the growing CNTs.[218] Some substrates (TiN, $SrTiO_3$, $LaAlO_3$ and stabilized ZrO_2) are unsuitable for growing CNT forests of high surface density. The situation can be changed by oxidative or reductive pre-treatment of the catalyst.

Base growth of a CNT forest is possible only for thin CNTs.[24, 56, 159] This was proved by tracer analysis.[219] Relatively thick multi-walled CNTs can form a nanotube forest by the tip growth of mixed growth mechanisms.[220, 221] Under particular conditions, catalyst particles adopt cylindrical shape and draw into the nanotube cavity.

The CNT – substrate adhesion strength was estimated as 1 to 10 pJ per nanotube.[222] It depends strongly on the character of interactions between the CNT, catalyst particles and substrate.

A carbon nanotube forest was also grown on polymeric substrates, in particular, on a Cr-coated polyimide film.[170]

Floating catalyst synthesis of a CNT forest from xylene on partially oxidized silicon surface with ferrocene as catalyst at 800 °C occurs selectively, *viz.*, only SiO_2 is covered.[182] This is similar to published results.[78]

The growth of a CNT forest from a mixture of C_2H_4 and H_2 is strongly influenced by the pressure at which the deposited catalyst film is annealed.[123]

A breakthrough in the synthesis of CNT forests is associated with the elaboration of methods for continuous CNT growth and increasing the growth rate. Catalyst poisoning was prevented by adding small amounts of water vapour to the mixture of C_2H_4 and H_2.[56, 62, 63, 163, 186, 223 – 227] Minor additives of water vapour added during CNT growth from ethanol vapours had a positive effect; a mechanism involving the formation of unstable iron oxides was proposed.[228]

This process was named supergrowth. Studies in this field offered prospects for the synthesis of relatively long CNT arrays. In particular, fine tuning of conditions allowed the CNT length to gradually increase from a previously record high value of 2.5 mm (see¶).[229 – 233]

The effect of the thickness of the deposited iron layer on the growth conditions was studied by the combinatorial masked deposition method.[234]

The efficiency of catalysts in the supergrowth regime is much higher than in other processes, namely, *cf.* a value of 50 000% (up to 500 g of CNTs per gramme of catalyst) *vs.* nearly 100% (1 g of CNTs per gramme of catalyst) for the floating catalyst approach, 300% for the HiPco method, 500% for the laser ablation technique which is also of limited use, or 800% for CNT synthesis from ethanol.

A single-walled CNT forest fabricated by the supergrowth method contained more than 99.5 mass % CNTs and the carbon content was 99.98%.[235] The average CNT diameter was unusually large (2.5 nm).[62]

The degree of utilization of the gaseous source of carbon depends on the gas supply method and reaches a value of 32% for gas delivery from the top; this is noticeably higher than for other methods of CNT synthesis.[236]

Pioneering publications devoted to supergrowth of CNTs gave a powerful impetus to studies of ethylene pyrolysis. Not only water vapours capable of removing carbon coatings on catalyst particles (and thus slow down its poisoning),[237] but also ethanol can be used as additive to C_2H_4; this led to threefold increase in the catalyst lifetime and a fivefold increase in the yield with respect to carbon.[238] The addition of oxygen,[239] air,[232] carbon dioxide [102, 240, 241] and other vapourized oxygen-containing substances give identical results.[242] It was also established that an increase in the concentration of CO_2 mixed with C_2H_4 causes the number of layers and the CNT diameter to decrease.[240] Therefore, the CNT diameter can be controlled by varying not only the thickness of the catalyst layer, but also the composition of the initial gas mixture.

Seven additives to three main sources of carbon (C_2H_2, CH_4 and CO) were tested and general conditions for accelerated growth of CNTs were formulated.[242] The key requirements include the oxidation and gasification of non-tubular carbon residue on the catalyst surface.

Carbon impurities can be removed using some organic compounds. For instance, oxygen-containing aromatics (methyl benzoate, benzaldehyde; ~0.02%) mixed with C_2H_4 act in different manner compared to water vapour and not only accelerate the growth, but also significantly increase the average CNT diameter in CNT forests.[243] The addition of cyclohexane to CH_4 favours growth of a CNT forest,[244] although this is hardly related to the removal of carbon coating on the catalyst particles. Most probably, cyclohexane participates in a complex chain of gas-phase reactions.

Pure ethanol can be utilized as source of carbon.[126, 127, 142, 143, 245 – 260] These studies are not related to supergrowth, but also showed that the addition of minor amounts of acetylene to ethanol causes a tenfold acceleration of the process.[261]

Parameters of pyrolysis at 1500 and 1700 °C and the morphology of single-walled CNTs in nanotube arrays are influenced by the composition of alcohol (methanol, ethanol, propan-2-ol).[262] The higher the molecular mass of the alcohol the lower the initial growth rate. Pyrolysis of methanol is accompanied by changes in CNT diameter and the process accomplishes with etching of carbon nanotubes.

Acetylene was used to grow CNT arrays.[26, 139, 161, 241] Supergrowth of CNT forests from acetylene diluted with argon is possible.[263, 264] There exist optimal acetylene feed rate at constant argon feed rate and the argon feed rate at constant overall gas feed rate. For a horizontal reactor 5 cm in diameter, these are 200 $cm^3\ min^{-1}$ for C_2H_2 and 500 $cm^3\ min^{-1}$ for $C_2H_2 + Ar$ provided that water vapours are saturated with argon at 60 °C and a pyrolysis temperature of 810 °C is attained after heating for 1 min.

In the case of acetylene pyrolysis, the height of the CNT array is proportional to $t^{0.5}$ (t is the duration of the process).[265] The addition of water vapour enhances the operating life of Fe/Al_2O_3 catalyst.[265] Detailed analysis of the kinetics of the CNT forest growth from acetylene on a Fe catalyst was reported.[266] The reaction order with respect to C_2H_2 is 0.6 at partial pressures ranging from $\sim 10^{-5}$ to 10^2 mbar and the activation energy lies between 0.92 and 0.98 eV (less than 100 kJ mol^{-1}). The reaction is limited by carbon diffusion into the catalyst which is preceded by 'pre-equilibrium' dissociation of acetylene on the catalyst surface.

High growth rates of single-walled CNT forest from acetylene were attained by finely tuning the synthesis conditions; no oxygen-containing additives were used.[267] A key to success was utilizing a low partial pressure of acetylene. The authors of that study believe that water vapours increase the catalyst lifetime in the presence of excess acetylene, but deactivate the smallest catalyst particles and reduce the quality of CNTs. As the process developed, the

¶ In 2007, it was reported on the Internet that researchers from the University of Cincinnati (USA) V Shanov and M J Schulz obtained 18 mm long CNTs; however, no publication in scientific journals is available so far.

CNTs diameter increased even with no additives. At a deposited iron layer thickness of 0.70 nm, acetylene concentration in argon of 0.30 vol.% and a temperature of 800 °C, the CNT forest height reached 1.3 – 1.4 mm after 14 min. Specific features of the growth of a CNT forest under pulsed acetylene delivery was studied.[268]

A relatively high growth rate of a CNT forest comprising nanotubes 5 – 7 nm in diameter, namely, 0.6 mm after 3 min, was attained when acetylene was diluted with hydrogen and nitrogen at 650 and 700 °C, respectively.[269] It is believed that the duration of CNT forest growth is governed by the degree of CNT ordering in the initial steps of the process. Studies [49, 91, 270 – 276] are mainly devoted to plasma-enhanced growth of CNT forest from acetylene – NH_3 mixture. Activated nitrogen compounds influence the mechanism of the process. External magnetic field causes the degree of ordering of CNT forest to increase and the defectness of carbon nanofibres to decrease;[277] however, practical value of this method for the synthesis of CNT forest is doubtful.

Pioneering studies on plasma-enhanced synthesis of CNT forests from CH_4 at a pressure of 15 mTorr were carried out in 1999 – 2001.[95, 278 – 280]

Carbon nanotube forests can be synthesized from ethylenediamine,[13] poly(ethylene terephthalate),[281] acetonitrile,[282] acetylene with acetonitrile and pyridine additives,[283] as well as from a solution of ferrocene in turpentine oil.[284] A CNT forest was obtained by thermal disproportionation of carbon monoxide on Co – Mo-catalyst,[101] from C_3H_8 using EPR technique,[285] as well from polyethylene, polypropylene and poly(vinyl chloride) by the pyrolytic method.[199]

The aim of many studies was to decrease the temperature at which CNTs grow. To this end, Fe catalyst layer of subnanometre thickness [121] and various plasma-activated techniques were used (in addition to the studies mentioned above, see Refs 50, 102, 120, 159, 170, 286 – 300).

At a substrate temperature of 600°C under plasma activation, the growth rate was 270 μm h^{-1}.[290] The lowest substrate temperature (200 °C) was attained by Lee *et al.*[290] 287. It is assumed that plasma activation favours alignment of CNTs perpendicular to the substrate surface;[46] this make it possible to synthesize less tortuous CNTs compared to those grown by pyrolytic methods.[159] However, plasma induces defect formation and subsequent reduction of CNT conductivity. Using floating catalyst CVD in alternating electric field, the substrate temperature was decreased to 280 °C.[301]

A more promising method for increasing CNT growth rate consists in overheating of initial gas mixture to temperatures above the substrate temperature (so-called parameter decoupling). A $C_2H_4 + H_2 + CO$ gas mixture for catalytic pyrolysis was heated to 1000 °C prior to contacting the catalyst.[225] The average growth rate at a substrate temperature of 810 °C was 7 μm min^{-1}, which is 15 times higher than without thermal activation of gases. One can assume that the decisive role is played by two parameters, the temperature and the duration of contact of the starting hydrocarbons in the hot zone. The technique was improved more recently.[174, 302, 303] Arrhenius plots of the CNT growth rate under overheating and with no overheating of gases are shown in Fig. 6. As can be seen, in the former case the CNT growth rate increased appreciably at a relatively low substrate temperature (475 °C). However, variation of the

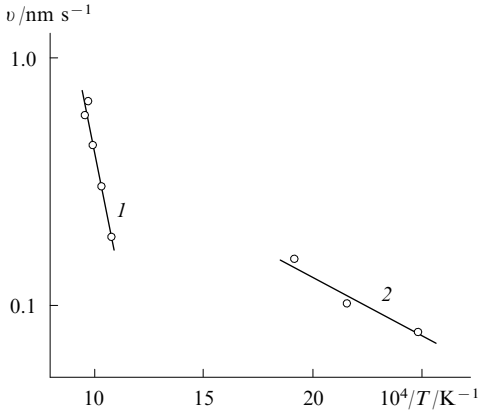


Figure 6. Arrhenius plots of CNT forest growth rate under gas overheating at a substrate temperature of 475 °C (*1*) and with no gas overheating at different substrate temperatures (*2*).[174]
Activation energy /eV: 0.9 (*1*) and 0.1 (*2*).

temperature of gases at constant catalyst temperature leads to changes in CNT morphology.[303] It should be noted that other authors [302, 303] observed the same regularities of the growth of CNT forest (from changes in CNT length) as those mentioned above † in the formation of CNTs on powder catalysts (based on results of gravimetric measurements): the higher the temperature the higher the rate of formation of CNTs and the shorter the operating life of the catalyst and the shorter the growth time (Fig. 7). This suggests the same mechanism in both cases.

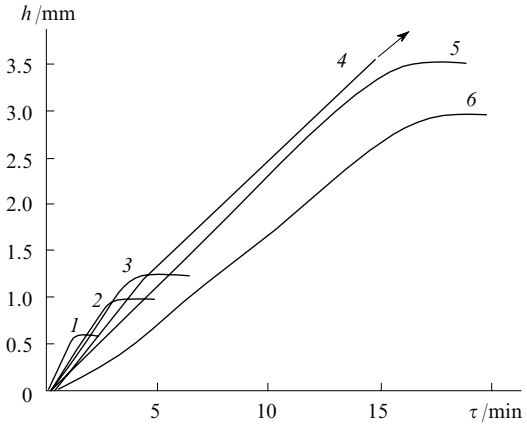


Figure 7. CNT height-*vs.*-time plots at different reactant gas mixture temperatures: 1120 (*1*), 1070 (*2*), 1040 (*3*), 1020 (*4*), 1000 (*5*) and 980 °C (*6*).[302]
The substrate temperature is 825 °C.

On overheating, alkynes appear in the gas phase; they accelerate the CNT growth.[305] In the case of synthesis from a $C_2H_4 + H_2$ mixture on Fe/Al_2O_3 catalyst, the most pronounced acceleration effect came from acetylene, methylacetylene and vinylacetylene; in all cases, a concerted

† See Fig. 109 in Ref. 304.

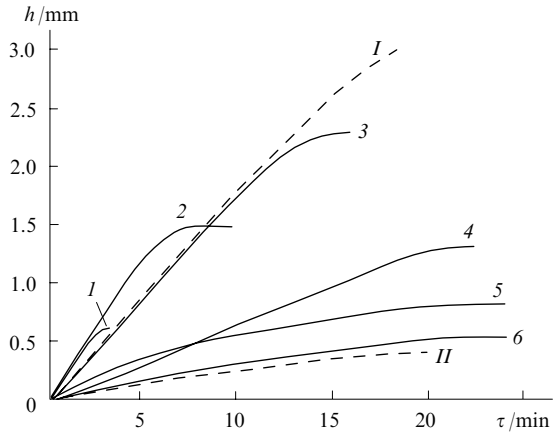


Figure 8. Effect of the composition of the additives to the $C_2H_4-H_2$ mixture and process conditions on CNT growth kinetics.[305]
With preheating without additives (*I*); without preheating without additives (*II*); in the presence of acetylene (*1*), methylacetylene (*2*), vinylacetylene (*3*), buta-1,3-diene (*4*), methane (*5*), ethane or propene (*6*) without preheating.

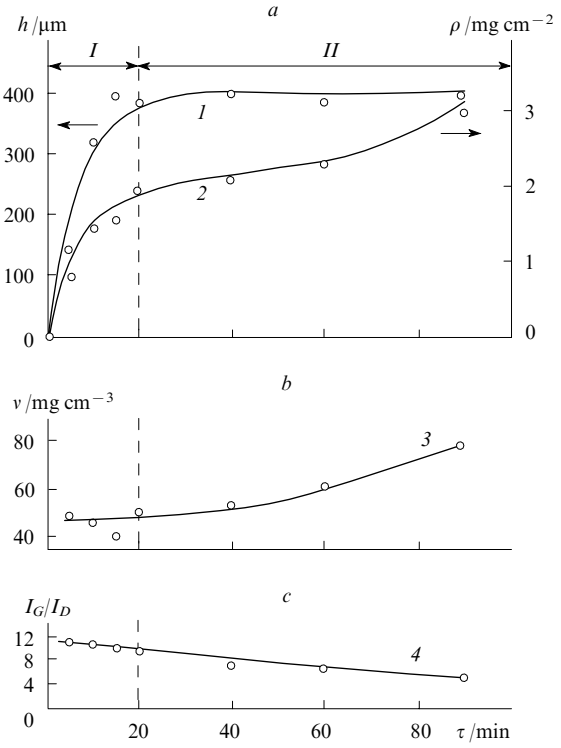


Figure 9. Growth dynamics of CNT forest.[235]
(*a*) changes in CNT height (*1*) and surface density (*2*); (*b*) changes in CNT density, (*c*) changes in the intensity ratio of the G and D modes in Raman spectra; intense CNT growth (*I*) and deceleration of CNT growth (*II*).

acceleration mechanism was revealed. Changes in the CNT forest growth rate upon pre-heating of gases and upon introduction of small (less than 1 vol.%) alkyne additives with no heating are illustrated in Fig. 8. Under particular conditions, the yield of CNTs increased 15-fold. The addition of C_6H_6 and CH_4 did not accelerate the process. Among more than 40 products formed in the gas phase upon heating of $C_2H_4+H_2$ mixture, some can slow down the growth of CNTs.[306]

Overheating of gases was used in another study.[307] The growth rate of multi-walled CNTs from a $C_2H_4+H_2+Ar$ mixture was 71 μm min^{-1} at a substrate temperature of 750 °C.

The growth rate of CNT forests was measured in different studies (Refs 9, 32, 121, 123, 223, 225, 229, 235, 246, 251, 256, 265, 266 and 308 – 317) (only the measuring technique was reported in Ref. 312). The growth rate of a CNT forest synthesized from C_2H_4 diluted with hydrogen and argon at 750 °C was as high as 140 μm min^{-1}.[314] It gradually decreased with time in accordance with the equation for the height of the CNT forest

$$h(t) = \beta\tau_0(1-e^{-t/\tau_0}),$$

where t is the duration of growth, β is the initial growth rate and τ_0 is the catalyst lifetime. The maximal height is given by[223]

$$h_{max} = \beta\tau_0$$

According to this equation, the growth is not accompanied by an increase in the surface density of CNTs. An analogous equation was reported in another publication.[256] At the same time, more recent results[235] point to an increase in the surface density and density of CNTs during the process (Fig. 9).

Pyrolysis of a mixture of C_2H_4 with hydrogen and small amount of water vapour at a particular Fe film thickness makes it possible to conduct selective growth of double-walled CNTs.[124] Selective growth of vertically aligned single-, double- and triple-walled CNT arrays under radiation heating is possible only at a particular surface density of catalyst particles.[19] Also, the larger the number of layers in CNT walls the longer the lifetime of the catalyst. Under identical conditions, the growth rate decreases in the order: single-walled > double-walled > multi-walled CNTs.[60]

Conclusions on the regime of the growth are contradictory. It was shown that the CNT forest growth rate is determined by the Knudsen diffusion of gases and the diffusion coefficient is of the order of 10^{-4} cm^2 s^{-1}.[217] Diffusion of reactants during the growth of CNT forest was also studied.[149] In addition,[229] the growth rate is limited by the process on the catalyst surface rather than by diffusion to the catalyst in the gas phase (as was stated in Ref. 217), a decrease in catalyst activity[223, 255, 318, 319] or catalyst poisoning.[313] A concerted mechanism of growth and deceleration of growth was proposed[33] and the influence of catalyst morphology on the forest growth mechanism was proved.[196] Microscopy studies revealed that water vapours do favour removal of graphene from catalyst particles.[237] However, some authors reported an abrupt cessation of the growth of CNT forest, but none of them proposed an unambiguous explanation for this fact.[32]

Synchrotron radiation measurements of the growth rate of a CNT forest disclosed that the growth of CNTs is preceded by the formation of carbide (cementite) Fe_3C;[320] this is consistent with the results obtained in other studies.[112 – 114]

A dimensionless criterion for the determination of the diffusion mechanism of CNT growth deceleration was proposed.[255] The mechanism of plasma-activated process differs from the mechanism of conventional thermal process owing to the presence of highly reactive atoms and ions.[321]

The rate and duration of CNT growth at optimal value of the $[C_2H_4]$/[water vapour] ratio depends on two parameters, *viz*., the residence time of gases in the hot zone (t) and the carbon flux (F_C).[322] The former is given by

$$t = \frac{273\,Al}{FT},$$

where A is the nozzle cross-section for top gas delivery, l is the length of the gas heating zone, F is the total inlet gas flow and T is the furnace temperature. The equation for the carbon flux has the form

$$F_C = \frac{f}{11.2\,A},$$

where f is the C_2H_4 flow.

At $t = 7$ s, the growth rate of single-walled CNTs was 620 μm min^{-1}; the yield with respect to carbon increased to ~15% as t increased to 15 s; the activation energy (E_0) of CNT growth was independent of t, being equal to 2.83 – 2.84 eV. The activation energy of CNT synthesis from $C_2H_4 + H_2 + He$ mixture is 2.6, 2.8 and 2.4 eV for Fe,[323] Ni and Co catalyst, respectively.

When using ethanol, the initial growth rate is maximum and then exponentially decreases with time; therefore, the duration of the process should not exceed 15 – 20 min.[251] The growth rate and morphology of CNT forests depend strongly on the rate and geometry of gas mixture flow.[260] A model for diffusion-limited CNT growth was reported.[324]

To increase the growth rate, a method was proposed, consisting in very fast heating and subsequent rapid cooling of the substrate coated with the catalyst.[325] At a pressure of 10 mm Hg in the reactor, the growth rate was 50 μm min^{-1} at 900 °C.

Conditions for transformation from the growth of entangled CNTs to the growth of a CNT forest were studied.[326] For pyrolysis of C_2H_4 on Fe/Al_2O_3 catalyst at atmospheric pressure and a temperature of 750 °C, such a transformation was attained by pre-heating of the catalyst in an H_2 – Ar mixture and by changing the gas flow distribution. The dependence of some properties of the process on the surface area of the sample was pointed out.

By adding NH_3 one can introduce nitrogen into CNTs in directed manner.[327]

To attain high adhesion of CNTs to substrates, rapid cooling of CNT forest in the reaction gas atmosphere was recommended.[34] This does not ensure good Ohmic contact of CNTs with substrates because electrical resistance of CNT arrays is usually 4 – 6 orders of magnitude higher than that of individual CNTs.[328] The formation of an Ohmic contact is a complicated task. To solve it, utilization of titanium electrodes (substrate) was recommended.[329] Cooling in a helium stream makes it possible to remove CNT forests from substrates. To enhance forest – substrate adhesion, it was recommended to use a 10 nm thick Fe-catalyst film and high concentration of C_2H_4 during the synthesis.[330]

Catalyst patterning was also done using nanostructured polysiloxane stamps; here, 'ink' was prepared in reverse micelles.[157] Under particular conditions, catalyst can be regenerated upon removal of CNT forests.[331]

By deposition on nickel strips 20 – 30 μm wide on silicon substrate and subsequent pyrolysis (no conditions were reported) was grown a CNT forest underneath the Ni layers.[182] It is believed that this can ensure using CNTs electric contact between the substrate and lifted-off nickel strips.

A mechanochemical model for growth termination of CNT forests was proposed.[332]

Studies on high-performance growth of CNT forests are carried out. A reactor was designed for simultaneous growth of CNT forests on fifty 150-mm wafers.[333] A breakthrough became continuous growth of CNT forest on a moving substrate.[334] At a velocity of a substrate with catalyst in the horizontal plane 0.2 – 1.0 mm s^{-1}, the average CNT length was ~0.9 mm and decreased as this velocity increased. A protopype of a continuous-operation setup for growth of CNT forest was reported.[335] Continuous growth of CNT forests on recirculating substrates was developed.[230] Before the publication of these studies and Ref. 336, it was assumed that continuous growth of CNT forests is only possible using the floating catalyst approach.[115]

A continuous process of the growth of CNT forest uses[107, 337, 338] natural vermiculite as catalyst. Vermiculite particles ground to 100 – 500 μm in size were preliminarily stratified, exposed to ion exchange with iron and tungsten salts, dried and reduced with hydrogen. A mixture of C_2H_4 with hydrogen was the source of CNTs. Pyrolysis led to an increase in the volume of catalyst particles due to the growth of CNT forest between vermiculite layers. Using a fluidized bed reactor, a CNT forest was grown with CNTs 6 – 10 nm in diameter and 5 – 7 μm long. The yield of CNTs was 3.8 g per gramme of catalyst.

Overall production of aligned and agglomerated CNTs is estimated at tonnes annually.[4] In most cases, they are synthesized in fluidized bed reactors. Among the reactors used to grow CNT forests, vessels with moving beds (moving substrates) are most often used. A similar reactor was also developed in Russia.[339]

In 2011, the first pilot plant for production of CNT forests with an overall production capacity to 10 t annually (from 100 to 500 g h^{-1}) utilizing supergrowth on substrates 0.5 × 0.5 m in size was put into operation in Japan.[340] A scheme of the process is shown in Fig. 10.

It is assumed that by 2014, a plant with a production capacity from 10 to 50 t annually with a reactor with 20 m long and 1 m wide a conveyer belt will be put in operation.

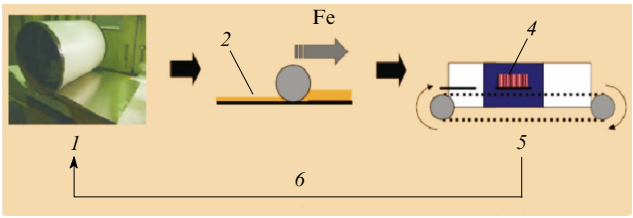


Figure 10. Supergrowth: a schematic.[340]
Metallic foil (*1*), iron salt and Al_2O_3 (*2*), catalyst levelling (*3*), CNT forest (*4*), conveyer for continuous CVD growth (*5*) and foil recycling (*6*).

Technological advantages of this process are such that the expected prime cost of pure CNTs will abruptly decrease to nearly \$12 per kg, thus ousting not only archaic electric arc and laser-thermal methods, but also thermal decomposition of carbon oxide as well as inefficient pyrolytic processes from the market.

A method for local growth of CNT forest by directed heating (no photolithography) was reported.[341, 342] A schematic of this process is shown in Fig. 11.

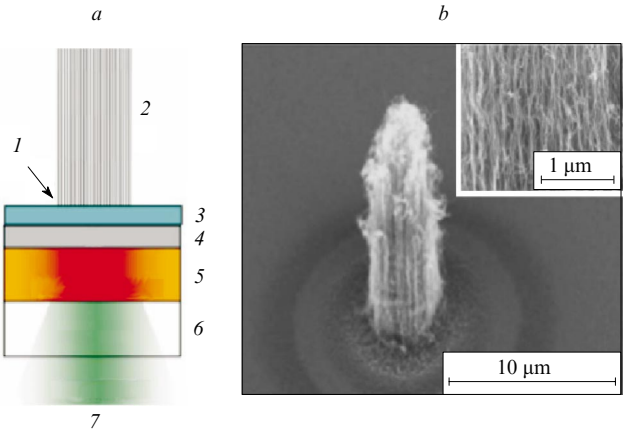


Figure 11. Growth of CNT pillars by local laser heating (*a*) and micrographs of pillar (*b*).[341]
$C_2H_2 + H_2$ mixture inlet (*1*), CNT pillar (*2*), Fe film (*3*), aluminium layer (*4*), chromium layer for heat distribution (*5*), quartz substrate (*7*) and laser beam (*7*).

The growth of CNT forests can be stimulated by laser radiation and in other ways.[343]

Carbon nanotube forests are often synthesized using porous alumina membranes. Specific features of these membranes are very large pore aspect ratio and relatively regular arrangement of pores. Pioneering studies on this method of synthesis[344 – 350] were reported in 1998 – 1999; a number of reviews are also available (see, *e.g.*, Ref. 351).

Usually, CNTs synthesized by the template method have both ends open[352] and their diameter is somewhat smaller than the pore diameter.[353] The larger the template pore diameter the more probable the deviation of the CNT diameter from the pore diameter (*cf.* CNT diameters 50 – 220 nm *vs.* pore diameters in the range 180 – 220 nm[354]). Nanotubes growing from the pore bottom have the pore diameter, whereas those growing on the outer surface of the membrane or from the walls have diameters equal to the diameter of catalyst particles.

Membranes make it possible to synthesize specific kinds of CNTs, namely, highly ordered arrays,[350] CNTs joined along their axes,[355] Y-shaped CNTs (nanotubes resemble the pore shape),[348] as well as nanotubes-in-nanotubes.[356, 357] Porous membranes can be utilized for the synthesis of CNTs from different materials and by the layer-by-layer method.[358]

The template method using alumina membranes is suitable for silicon technology.[351] A thin aluminium film is deposited in vacuum directly on a silicon substrate and then anodized.[359, 360] Then, metal catalyst is deposited on the bottom of the pores and CNTs grow inside nanopores.[361]

To equalize the CNT length (to cut CNT tips grown out of the membrane surface), sonication in alcohol or in water was proposed.[362]

A process was developed of CNT growth at specified intertubular distances using a membrane fabricated by nano-imprinting on aluminium film prior to electrochemical treatment.[363] In this case, etching of metal occurs at sites where preliminary local mechanical action was done.

A template synthesis of ordered CNT array containing nitrogen was described.[364] A CNTs/Pd – Sn/CNTs hierarchical structure was obtained by a relatively simple procedure.[365]

By preliminarily coating membrane pore walls with Fe nanoparticles one can synthesize a ferromagnetic material representing ferromagnetic particle-doped CNTs.[366]

A number of studies are devoted to the synthesis of CNTs using Al_2O_3 membranes and properties of CNT arrays.[38, 41, 367 – 381] Not only Al_2O_3 membranes, but also nuclear membranes,[382] porous silicon[351, 383] and mesoporous silicates[213] are used to grow CNT arrays. A porous polystyrene-*block*-poly(methyl methacrylate) nanotemplate was tested successfully.[140]

An exotic method of synthesis of CNT forest from alcohol flame in electric field was reported.[384] Aligned forest of bamboo-like nanofibres was obtained by magnetron sputtering of graphite;[11] however, this technique also remains of limited use. High-voltage electrophoretic deposition[385] and filtration of CNT dispersion in a magnetic field[386] were also reported.

Pyrolysis of a mixture of C_{60} fullerene with ferrocene at 1050 °C in NH_3 atmosphere gave a nitrogen-containing CNT forest.[387] Such systems are formed by plasma activation of a mixture of hydrocarbons with NH_3 or N_2.[278, 280]

IV. Properties of CNT forest

Since base growth is the main growth mechanism of CNT forests, CNTs can exert pressure on the substrate surface. For instance, the pressure produced by five-walled CNTs 9 nm in diameter is 0.16 nN.[31] Loading of CNT forests causes the growth rate to decrease with increasing load.

The specific surface area of CNT forest produced using iron phthalocyanine as catalyst is not large and lies between 28 and 57 $m^2\ g^{-1}$ and the specific pore volume is from 0.05 to 0.11 $cm^3\ g^{-1}$.[388]

Mechanical properties of CNT forests depend on the CNT length, diameter and surface density. The compressive strength of ~200-nm CNT mats with very high surface density is close to 800 MPa;[389] as the CNT length increases to a few millimetres, the strength decreases to 1.8 MPa.[390] The bending modulus of CNTs in CNT forest[391] and the compression modulus of the CNT forest at CNT height of 15 – 500 μm (~0.25 MPa) were determined. The Young modulus of an aligned CNT array (CNTs were less than 12 nm in diameter) exceeds a value of 400 GPa.[392] Mechanical properties of densely packed single-walled CNTs were reported.[393] The mechanical and adhesive properties of entangled CNT forest (CNT 'turf') were studied.[394]

The ignition temperatures of CNT forest in air (~525 °C) differ insignificantly from those of free-standing CNTs and somewhat increase as CNTs become longer.[395]

There is a number of studies[396 – 406] and a thesis[407] devoted to thermal conductivity of CNT forest. The thermal conductivity coefficient of a CNT forest can reach a value of

70 W m^{-1} K^{-1},[395] being higher than those of conventionally used solders. At the same time, the total thermal resistance of the CNT forest surface is 15 W mm^2 K^{-1}, which is also higher than those of solders. Since samples can differ significantly in length, diameter, defectness, tortuosity and surface density of CNTs and in the character of their bonding to substrates, these parameters may be either larger or smaller. To improve the transport properties of CNTs, they are coated with metals,[397] their cavities are filled with metals[398] or the CNT forest is densified.[399]

The conductivity of a single-walled CNT forest is 0.56 S mm^{-1} along CNT axes, being independent of CNT length, and a tenfold lower when measured along the normal to CNT axes.[259] The contact resistance of a 100-μm CNT forest grown on a Mo substrate is 5×10^4 Ω μm^2.[408] Interestingly, the electrical resistance of amorphous CNTs grown by the template method is of the same order of magnitude as that of well-graphitized multi-walled CNTs, although the structures of these materials are different.[409] The forest exhibits the piezoresistive effect (reversible decrease in resistance) upon compression.[410]

Diffusion of gases across the surface of CNT forest causes its compression due to the downstream deviation of CNT tips.[411] A model describing the behaviour of a CNT forest under shear deformation was developed.

Axial compression of CNTs leads to their bending, buckling[412] and fracture,[13] the behaviour being governed by loading conditions and by the number of loading cycles.[413] At the same time, an array of vertically aligned CNTs ~40 nm in diameter exhibits very high elasticity on compression and tension. The compressive strength is 15 GPa. Compression is almost reversible (Fig. 12). A CNT forest study[414] revealed an average buckling period of 12 μm. Compression caused the film thickness (CNT forest height) to decrease to 15% of the initial value. After 1000 compression – recovery cycles, the film thickness was ~85% (*cf.* ~80% after 10 000 cycles). The film behaves analogously to an elastic sponge with open pores which is characterized by very high compressibility. Also, it exhibits high shape recovery rate (> 2000 μm s^{-1}) and is mechanically stable.

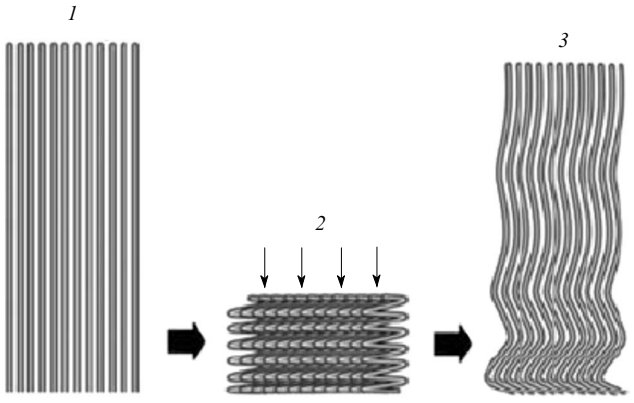


Figure 12. Cyclic compression and recovery of CNT forest.[414] Initial height of CNT forest (*1*), compressed CNT forest (*2*) and unloaded CNT forest (*3*).

Table 1. Comparison of compression data.[415]

Material	Load /MPa	Deformation (%)	Deformation rate /s^{-1}	Number of cycles
Human skeletal muscles	0.35	> 40	5	> 10^9
Polymer PVF [a] (piezoelectric)	3	0.1	> 1	> 10^6
Polyanil (conductive polymer)	180	> 2	> 1	> 10^5
PVA-PAA gel [b]	0.3	> 40	0.1	> 10^5
Multi-walled CNT forest	> 2	> 35	> 0.75	> 10^6

[a] Poly(vinylidene fluoride). [b] Polyvinyl alcohol – polyacrylamide.

Yet another study on cyclic compression is available.[415] The authors compared the properties of a CNT forest with those of other materials (Table 1). As can be seen, the CNT forest exhibits rather high but not record-high properties. Mention was also made that compression causes the CNT conductivity to increase to some extent, the new values being reproducible at a number of compression – recovery cycles of 150 000.

The deformation mechanism of CNT pillars 50 μm in diameter involves periodic buckle nucleation, sequential bottom-to-top buckling process, gradual compression of the entire structure and formation of a periodic structure (Fig. 13).[416]

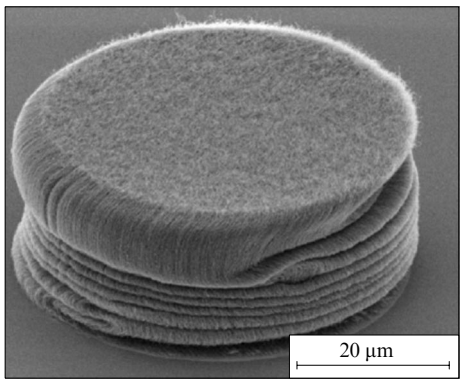


Figure 13. Micrograph of compressed CNT forest.[416]

It is believed that by coating a CNT forest with an amorphous diamond film one can obtain a hybrid material with very high elastic properties, with the damping properties being dependent on the surface density of CNTs.[417]

Experiments showed that the maximal energy reversibly stored on tension is 500 kJ m^{-3} or 7 kJ kg^{-1} for 6 mm long multi-walled CNTs.[418] The former number is tenfold larger than the corresponding value for a steel spring, the latter is half the energy density of a steel spring.

Analysis of the morphology of a helical CNT forest showed that impact causes no plastic deformation and is characterized by full shape recovery.[13] The behaviour of this forest is noticeably different from that of structures

comprising free-standing nanotubes (see also Refs 419 and 420).

A carbon nanotube forest is not stable to shear deformation and can be readily laid down on the substrate.[417]

Carbon nanotube forests can be grafted onto flexible substrates by partially embedding CNTs.[421] A quasi-static indentation and impact-induced embedding of CNT forest into a polydimethylsiloxane thin film was studied.[422] The load-displacement curves show three regions related to densification, bending and buckling of CNTs. The degree of elastic shape recovery depends on the indenter immersion depth and varies between 10% and 25%. When using a punch $\sim$30 μm in diameter with a flat working surface, the Young modulus is close to 0.4 MPa (*cf.* a value of about 50 MPa determined for uniform compression of free-standing CNT forest).[414] At an impact load equivalent to that experienced by a dropped cell phone, the CNT forest embedded into the polymer at the base behaved as impact-energy absorbing and dissipating material with much better properties compared to the polymer and CNT forest on silicon substrate.

Many properties of CNT forest depend on the morphology and surface density of CNTs.[60] For instance, the highest conductivity and current density was exhibited by a multi-walled CNT forest, while the highest thermal conductivity was determined for a single-walled CNT forest.

The advantages of CNT forests consist not only in its anisotropic properties (in particular, increased thermal and electrical conductivity along CNT axes), but also in CNT purity and in simplification of manipulations aimed at obtaining individual CNTs, fibres and films.

The interaction of a CNT forest with IR radiation was studied.[370] Also, a CNT forest is characterized by record-high ability to absorb incident light, being close to a black body.[189] The integrated reflectance of a CNT forest with the density 0.01 – 0.02 g cm^{-3} is 0.045%, being three times lower than those of known materials. The record [189] was cut in a short time, and a CNT forest with a reflectance of 0.01% in a wider spectral range was synthesized.[423]

The properties of CNT forest as a black body manifest themselves at particular film thickness which should be greater than 10 μm. The refractive index of the material equal to 1.07 indicates that the optical properties are determined by the presence of atmospheric air.[424] The extinction coefficient of the CNT forest in the visible region (0.038) is lower than that of metals and silicon. Structuring of the CNT forest makes it iridescent, which may be used in practice.[424] The optical properties of a low-density CNT forest were studied.[425]

High-intensity visible light causes electron emission from CNT arrays.[426] This was observed upon laser irradiation at 532 and 488 nm; the emission current increased pseudo-exponentially with increasing irradiation power (see also Ref. 427).

Carbon nanotube forests exhibit superhydrophobic and superlyophobic properties.[428] For water droplets, the contact angle increases from 100° for single-walled CNTs to 120° for double-walled CNTs and to 150° for multi-walled CNTs.[53] A CNT forest comprised of carbon nanotubes coated with polytetrafluoroethylene thin film is also a superhydrophobic material.[429]

The friction coefficient of a CNT forest with 6-μm CNTs is independent of moisture and ranges from 0.6 to 1.3.[430]

V. Modification of CNT forest

Modification implies changes in the shape or composition of primary CNT forest and can be achieved using mechanical, physicochemical, chemical, electrochemical or combined techniques.

Mechanical methods include microstamps, alignment of CNT forests on substrates by shear deformation, a decrease in the surface density of CNTs by mechanical 'thinning', mechanical formations of particular configurations of CNT forests, namely, pillars, ribbons, cones, *etc.* A two-step process of mechanical dispersion of CNT forests to give transparent coatings was developed.[431]

Physicochemical methods are based on capillary effect. Wetting of CNTs followed by evaporation of solvent leads to densification of the CNTs, their laying down on the substrate or formation of 3D systems of complex shape (screw-like, star-shaped, *etc.*).

Chemical methods include grafting of chemical groups, monomers that can then be polymerized, and polymers to the surface of CNTs, chemical etching, decoration of CNTs with nanoparticles, uncapping of CNTs and filling their cavities with various substances, formation of composites by filling the intertubular space or coating of CNT tips with some compounds. Electrochemical methods include, in particular, electropolymerization [432] and decoration.[433]

Combined methods include electro- and photochemical modification of CNT forests, as well as plasma and laser treatment.

A particular type of operation is represented by transfer of CNT forests from primary substrates to some other substrates.

The simplest way to reinforce electrodes based on forests is to fill the intertubular space with silica gel by chemical vapour deposition of tetraethylorthosilicate.[434]

To fabricate membranes, to increase the surface area of (i) electrodes of supercapacitors and (ii) catalyst supports as well as to reach many other goals, open CNTs are required. Nanotubes in CNT forests can be uncapped by slow oxidation by anhydrous air or by oxygen at a low heating rate.[435, 436] If CNTs have additional 'windows' on lateral surfaces, the specific surface area can be increased to 2240 m^2 g^{-1} (heating to 500 °C at a rate of 1 K min^{-1}).[436] Another method to obtain vertically aligned arrays of open CNTs is as follows: a CNT forest grown on a substrate is flipped, CNT tips are immersed into a solder and then the substrate is dissolved.[437]

Hydrophobic properties of CNT forests can be controlled by functionalization in plasma [438] and by UV irradiation in ozone atmosphere.[439] The surface of CNTs can be modified by photochemical addition of perfluoroaryl azides.[330]

Organization of CNT forests as hierarchical 2D and 3D structures is possible (see, *e.g.*, Refs 45 and 440). Carbon nanotube forest can be structured by embossing into micropatterns (resolution to 10 μm) or by chemical etching (resolution to 5 μm).[45] Methods for micro-electromechanical processing of CNT forest by a rotating electrode were developed.[441]

Wetting of CNT arrays with acetone, toluene, ethanol, isopropyl alcohol, water and subsequent evaporation of the liquid leads to densification of the CNT arrays.[16, 59, 442 – 448] Capillary forces arising during the evaporation of liquids from CNT forests cause changes in the morphology and

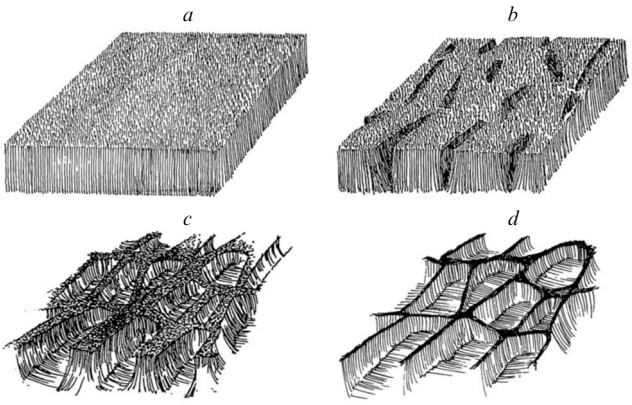


Figure 14. Formation of cellular structure in CNT forest film.[442] Initial CNT forest film (*a*), crack formation during the evaporation of liquid (*b*), bending of CNTs and association of their tips (*c*) and the final cellular structure (*d*).

assembling of adjacent 'cells' to form a 2D 'foam' (Fig. 14).[442, 443, 449] Isotropic compression[59, 445] and adhesion to substrate[450] was reported for assemblies of CNT forest microstructures. A detailed study of the influence of capillary effect on various configurations of CNT forests allowed one to reveal and classify specific changes in their microarchitectures.[451] These changes were most pronounced for CNTs arrays occupying less than 400 μm^2 on the surface at CNT aspect ratio of > 3. Examples of changes are given in Fig. 15.

Depending on the CNT length, cross-section shape and the nature of the wetting liquid, its evaporation can lead to connection of CNT tips and formation of conical (pyramidal) structures[444, 449] or CNT bundles joint along the CNT axes except base regions.[448] Using a dielectric liquid oil, the volume of the CNT forest was reduced by almost 80%.[441]

Densification of long single-walled CNTs was carried out with partial detachment of the CNTs from the substrate.[59] The CNT forest properties before and after densification are listed in Table 2 where the corresponding properties calculated for ideal dense bundles of CNTs 2.8 nm in diameter are given for comparison.

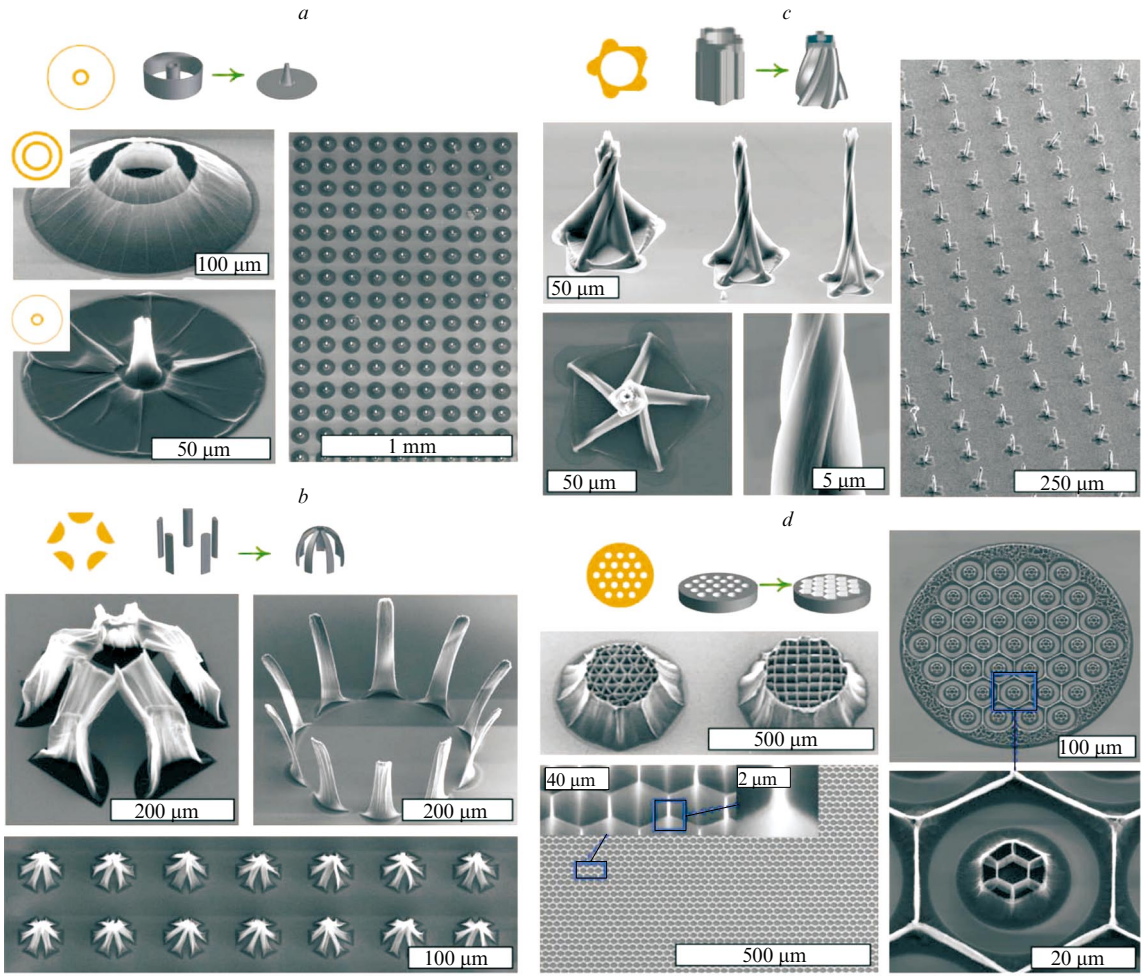


Figure 15. Micrographs of different forms of CNT forests created by capillary compression.[451] Concentric cylinders and cones, radially oriented CNT films surrounding a vertical needle (*a*), 'blooming flowers' of radially-oriented bending CNT structures (*b*), CNT micro-helices with variable diameter and pitch (*c*) and complex lattice structures formed from CNT forests with patterned internal voids (*d*).

Table 2. Structure and properties of CNT forest.[59]

Property	Initial CNT forest	Dense CNT forest	Perfectly dense CNT forest
Density /g cm^{-3}	0.029	0.57	0.78
Surface density of CNTs /cm^{-2}	4.3×10^{11}	8.3×10^{12}	1.1×10^{13}
Surface area per CNT /nm^2	234	11.9	8.7
Lattice constant /nm	16.4	3.7	3.2
Relative volume of CNTs (%)	~2.6	52	72
Vickers hardness	~0.1	7 – 10	–

By varying catalysts one can induce the formation of cellular or island structure of CNT forests, namely, CNTs move from their initial positions and leave some area on the substrate surface unoccupied.[452] The distance travelled by the CNT bases increases with increasing the height of CNT forest.

The influence of different solvents on the mechanical properties of densified CNT fibres and pillars and CNT forest (3-mm height) was studied.[413] It was shown that the maximal compression energy density of a CNT array is 7.8×10^6 kJ m^{-3}. Densification of a template-grown CNT forest was reported.[453] The electrocapillary effect was utilized to densify a CNT forest[454] and to lay down the CNT ribbons on the substrate.[455] A CNT forest can be densified mechanically; here, the volume occupied by all CNTs is decreased by 1% – 20%.[456]

Carbon nanotube forests were mechanically aligned to fabricate dense flexible ribbons.[34, 230, 328, 329] Namely, parallel ribbons of CNT forest (1 – 2 mm high and 20 or 100 μm wide) with spacings equal to the CNT height were grown and aligned to the substrate surface. The schematic of the process is shown in Fig. 16. The stronger the alignment

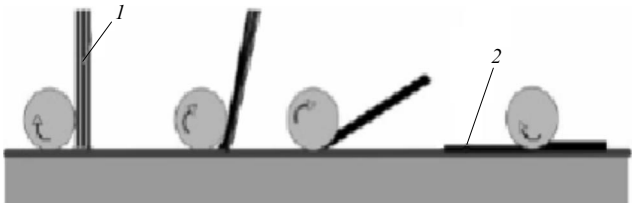


Figure 16. Rolling of vertically aligned CNT arrays into horizontally aligned, densified CNT strips.[328]
CNT forest packing fraction: 0.02 (*1*) and 0.25 (*2*).

force the smaller the film thickness and the higher the density of CNT alignment. The relative density of CNT alignment was determined as the ratio of the surface density of CNTs in the CNT forest to the theoretical surface density calculated for densified CNTs of a specified diameter (in this case, ~10 nm). It increased from an initial value of 0.02 corresponding to the CNT forest to the maximal value of 0.25 for the film. Further capillary densification caused the relative density of alignment to increase to a value of nearly 0.6. The average resistivity of films thus obtained with a volume occupation of 0.42 was 1 μΩ cm; they could be transferred onto various substrates and use as, *e.g.*, contacts in micro-electromechanical systems.‡

Ribbons from densified vertically-aligned CNT arrays were fabricated from a CNT forest grown in the form of square pillars by transfer onto another substrate.[458] A simpler way of horizontal alignment of CNT forest strips is to wet them with isopropyl alcohol and then to dry.[450] Aligned CNT forest can be transferred from one substrate to another by dry contact printing technique.[459]

Nanotubes in CNT forests can be modified, namely, decorated with, *e.g.*, Pt nanoparticles,[460] bear continuous coatings or contain metals (in particular, nickel[461]) in the inner cavity. Decoration of CNT forest with metal or oxide nanoparticles differs from the decoration of free-standing CNTs because top-to-base diffusion is hampered and a continuous coating can form on the surface of the CNT forest. The interaction of deposited metals (Au, Al, Ti, Pd) with the array comprising CNTs with an average diameter of 1.9 nm at different temperatures (25 and 300°C) and deposition rates (0.05, 0.20 and 0.40 nm s^{-1}) is accompanied by the formation of either continuous film or individual metal nanoparticles.[462] Deposition of platinum, ruthenium and silver proceeds with the formation of clusters at the top of CNTs; however, thermal annealing leads to migration of metals towards the base area of the CNT forest.[463] Metal nanoparticles can be deposited by electrostatic[464] and electrolytic[465] techniques.

Chemical modification of CNT forests can be achieved in different manner using, *e.g.*, perfluoroarylazides[330] or nucleic acids.[466] Chemical method allows individual CNTs to be cross-linked, thus improving the performance of actuators.[467]

To avoid collapse of CNT forests upon opening and chemical functionalization of CNTs, it was proposed to fill the intertubular space with spin-on glass.[466] Then, acid functionalization of CNT open ends and further transformations of grafted carboxyl groups can be done with ease.

Both pre-functionalized and untreated CNT forests are suitable for electrochemical modification.[468, 469] Not only metal deposition, but also electropolymerization of pyrrole[470, 471] and plasma polymerization of a fluoropolymer[472] were reported. Various configurations of CNT forests are formed using electric microcharges.[473]

Carbon nanotube arrays are detached from quartz substrates by immersion into water.[249] Carbon nanotube forests can be transferred to flexible polymeric substrates by contact microprinting;[155, 474] one can also fabricate complex 3D structures.[475] Transfer to polymeric substrates can be done with a hot plate,[476] by spin-coating[416] and microwave heating of thermoplastics.[477] When using polydimethylsiloxane, transfer of CNT forests onto Pt-coated glossy paper, fabrics, polymers, glass plates and metallic foil is possible.[478]

A method was proposed for transfer of CNT forest to the secondary conductive substrate coated with a solder (Sn – Pb eutectic).[437] CNT tips are immersed into the solder while the primary substrate is removed (Fig. 17). Carbon nanotubes on the secondary substrate have open ends. The

‡ Mechanical alignment was also used in another study;[457] is will be discussed in the next Section.

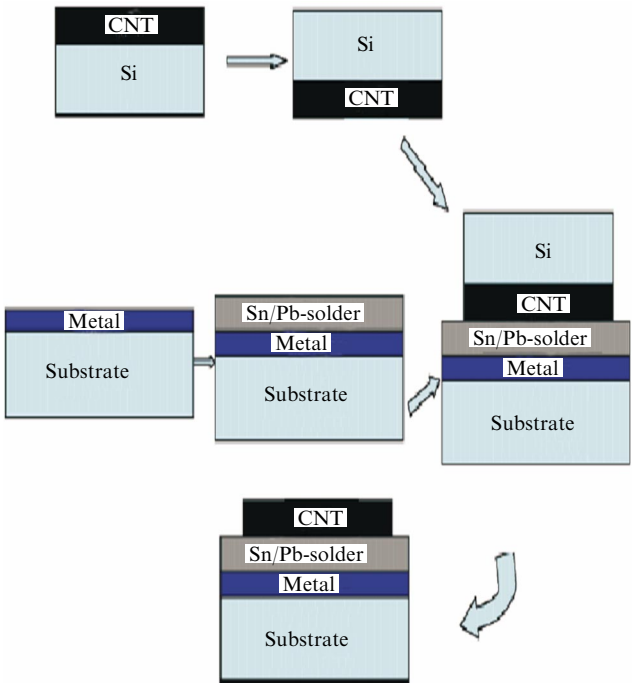


Figure 17. Schematic diagram of CNT transfer technology.[437]

secondary substrate and base regions of low-surface-density CNTs are coated a thin layer of epoxy resin, the substrate is removed and the CNT forest is transferred to another substrate which can be flexible.[479]

VI. Applications of CNT forests

Potential application fields of CNT forest are diverse and continuously extend. Some of them have been surveyed.[215, 480]

Undoubtedly, supergrowth of CNT forest will be used as high-performance method of synthesis of pure uniform CNTs.

Numerous attempts to utilize CNT forests as material for field emitters were made (Refs 46, 54, 56, 93, 159, 166, 170, 191, 246, 291, 293, 294, 376 – 378, 453, 481 – 493). Double-walled CNTs are thought to be better than single-walled ones (the latter undergo a relatively fast degradation).[172, 487] When using a CNT forest for fabrication of field emitters, optimization of the catalyst layer thickness is of particular importance.[488] Single-walled CNT field emitters fabricated on multi-walled CNTs were reported.[453] An important problem consists in the difficulty in obtaining a low surface density of CNTs because at high surface density, electromagnetic fields overlap and field emission becomes less efficient. To solve the problem, it was proposed to use honeycomb configurations of CNT forests.[453]

Manufacturing of electrodes for supercapacitors is yet another promising application field of CNT forest.[494] The first experiments were carried out using a CNT forest with random alignment of CNTs, having a smaller specific surface area compared to activated carbon. The CNT forest showed a higher capacity, namely, 102 F g^{-1} for multi-walled CNTs[495] and 180 F g^{-1} for single-walled CNts.[496] Ready accessibility of CNT surface to electrolytes, which ensures high capacity, is due to unique porosity of CNTs owing to high content of mesopores.[495 – 497] Also, CNTs are better conductors than activated carbon.[498] Another advantages of CNTs include the lack of edges typical of faceted crystals as well as the ability to be aligned in directed manner.[499]

The possibility of utilizing CNT forests in highly efficient supercapacitors was substantiated in another study.[500] Such devices show high stability on cycling.[351] Patterned configurations of CNT forests offer advantages over those with random alignment of CNTs due to specific features of adsorption of electrolyte particles on the CNTs.[387] The position of the supercapacitors based on CNT forests among other small power cells is shown in Fig. 18.

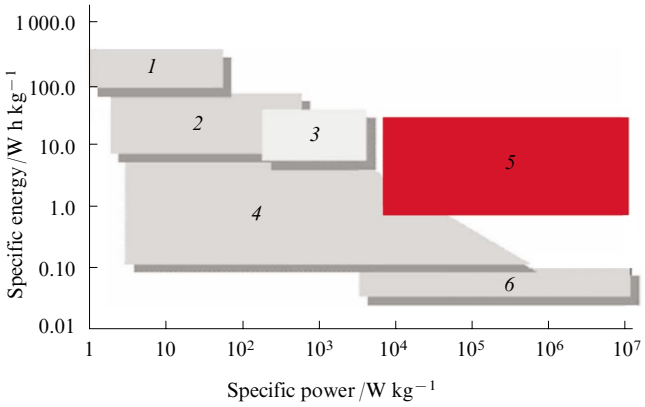


Figure 18. Properties of energy storage systems.
Fuel cells (*1*), conventional power cells (*2*), inertia devices (*3*), supercapacitors (*4*), supercapacitors with single-walled CNTs (*5*) and conventional capacitors (*6*).

Supercapacitors based on single-walled[501, 502] and multi-walled CNTs,[503] as well as on CNT/graphene sandwiches[504] were tested. Some of then remain stable after 200 000 charge – discharge cycles in the temperature range from 25 to 100 °C.[505]

Treatment of CNT forests to open CNT ends leads to an increase in the surface area of electrolyte/electrode contact.[501] For open single-walled CNTs, the specific surface area increases to 2200 m^2 g^{-1}, the specific energy of electrodes increases to 24.7 W h kg^{-1}, and the specific power increases to 98.9 kW kg^{-1} (electrolyte was a solution of tetraethylammonium tetrafluoroborate in propylene carbonate);[436] the CNT forest was mechanically densified to obtain a high specific surface area. Carbon nanotubes can also be uncapped with plasma.

A densified CNT forest was obtained by combining mechanical alignment to the substrate surface and wetting with electrolyte.[457] A suparcapacitor with densified CNT electrodes operating at a voltage of 4 V was characterized by a specific energy of electrodes of 94 W h kg^{-1} and a specific power of 210 kW kg^{-1}.

A number of studies are devoted to supercapacitors based on vertically aligned arrays of CNTs decorated with reactive transition metal oxides, *viz.*, RuO_2,[506, 507] NiO(OH),[508] Mn_3O_4.[509] A supercapacitor based on a forest

of template-grown CNTs coaxially coated with a MnO_2 layer was reported.[510] The results of particular studies on supercapacitors were documented.[59, 61, 414, 436, 511 – 514]

Micro-supercapacitors were fabricated,[385, 515] they can be assembled by the 'origami' principle.[516, 517] According to model calculations of a supercapacitor based on CNT forest, its capacity depends strongly on pore size.[518]

A Li-ion power cell based on a CNT forest coated with amorphous silicon was proposed. Silicon can provide a very high specific energy; however, bulk silicon undergoes irreversible dispersion upon intercalation with lithium. This drawback can be eliminated using patterned silicon forest.

A CNT forest is a good support for catalysts including the Fischer – Tropsch catalyst.[179] Prospects for utilizing CNT forests with platinum[460] and PtRu[519] nanoparticles dispersed on CNTs as electrocatalysts for fuel cells are considered.

The applicability of vertically aligned arrays of CNTs coated with a thin (5 nm) layer of $In_2O_3 - SnO_2$ solid solution in new-generation solar cells was studied.[520]

A CNT forest with redox-active molecules grafted onto CNTs is promising for information storage.[521] Carbon nanotube forests can serve components of micro-electromechanical systems[385, 450, 522] and have different functions up to being a kind of wicking for fluid delivery.[523] Prospects for application of CNT forests as electrical conductors grown on side surfaces of electrodes are also vital.[524] At CNTs length 0.05 μm (and longer) their resistance is lower than that of copper (Fig. 19).

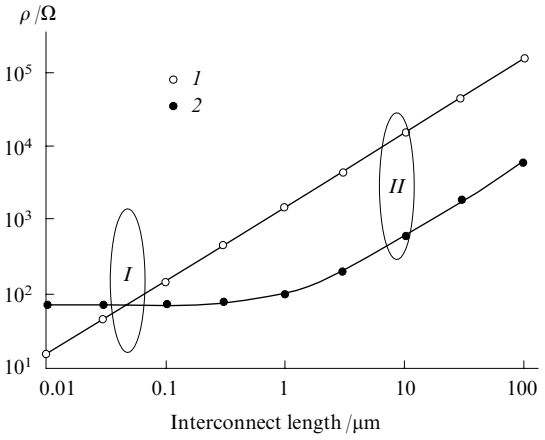


Figure 19. Electrical resistance-*vs*.-interconnect length plots for a copper (*1*) and a CNT-based interconnect (*2*).[524]
Regions *I* and *II* correspond to nearly equal and considerably different electrical resistance, respectively.

Carbon nanotube forest are suitable for drawing long thin ribbons[128, 176, 525 – 528] and twisted yarns or fibres.[7, 99, 134, 176, 447, 529 – 539] Not all types of CNT forests are appropriate for drawing purposes;[537 – 540] nevertheless, yarns were fabricated even from a powder-containing material.[536] The thickness of CNT yarns can be varied by varying the drawing angle.[541] Conditions for manufacturing of strong wires from CNT forests and method for improvement of their tensile strength were described in Ref. 532. The average tensile strength of polymer-free fibres reached a value of 800 MPa while that of fibres containing 10 mass % polystyrene was 1040 MPa.

Carbon nanotube fibres and yarns with high conductivity due to the deposition of metals[534] can serve as microelectrodes for implants, light-weight conductors for aviation and in electronics. By growing a CNT forest on both sides of metallic foil one can simultaneously draw two yarns.[176] Carbon fibres can serve sensors in composites.[542]

CNT-Ribbons are used to manufacture actuators.[507, 543] It was proposed to utilize superaligned CNT forest and fibres and ribbons fabricated from it as sources of polarized light, optically transparent electrodes and filaments, flexible and transparent membranes for loud-speakers, components of superelastic actuators and grids for high-resolution electron microscopes.[100]

To add functionality of CNT forest, one should design composite materials belonging to the following four types:

— CNT forest in a polymeric or inorganic matrix (see below);

— CNT forest with CNTs decorated with metal or oxide nanoparticles;

— CNT forest with filled CNT cavities;

— CNT forest/inorganics (vermiculite,[544] graphene,[188, 504, 545, 546] polydimethylsiloxane[547]) sandwiches.

Under particular conditions, the catalyst used to grow CNTs may to one or another degree fills their inner cavities (see, *e.g.*, Refs 180 and 548). Also, nanotube cavities can be filled with fullerene molecules.[549]

Carbon nanotube forests are utilized as components of polymeric nanocomposites fabricated using infiltration of polymers,[526, 550 – 560] infiltration into densified CNT forest,[561 – 563] into aligned CNT forest,[564] electrochemical deposition,[565] vapour phase deposition,[429, 434, 503, 550, 566 – 569] capillary wetting with polymers[570] and alignment in magnetic field.[571] Studies on vacuum infiltration of CNTs including CNT forests were reviewed.[572] Composites for self-cleaning coatings were reported.[552] Problems in the design of nanocomposites with high thermal conductivity and the thermal conductivity mechanisms were discussed.[563, 573] Application fields of polymeric composites for solar cells were considered.[574]

It was proposed to use composites based on densified CNT forests as templates (master moulds) for fabrication of polymer microstructures.[575] To this end, a CNT forest grown on a patterned catalyst is capillary densified using organic liquids and then impregnated with the polymer.

Intertubular space is filled with not only polymers, but also Si, SiO_2 and Si_3N_4.[64] Botryoidal ZnO particles were grown by CVD on carbon nanotube forest.[576] The list of nanocomposites based on inorganic substances filled with CNT forests is rather long.

Hybrid composites comprised of carbon, ceramic or glass fibres in a polymeric template with reinforcing CNT forest laminae are also promising.[189, 542, 553, 577 – 579] When fabricating layered composites, CNT forests can be introduced by direct growth on woven fabric layers,[189] by transfer using microprinting techniques,[578] by incorporation between layers of carbon fibre fabric[542, 553] and infiltration.[580] Coating of a CNT forest with polyurethane film and subsequent removal of substrate afforded CNT/polyurethane films.[559]

Carbon nanotube forests are used to fabricate superhydrophobic coatings.[330, 428, 429, 444, 581 – 584] Such coatings

on steel meshes make it possible to separate water droplets from organic liquids.[177]

Superhydrophobicity was also attained by alignment of CNT forest as ribbons forming honeycomb structures.[30] In contrast to the CNT forest with densely packed CNTs, films with the honeycomb structure were characterized by a sliding angle of less than 5°. The authors reported that the honeycomb structure was obtained immediately in the floating catalyst synthesis of CNT forest; however, usually CNT tips are joined upon evaporation of solvent from prewetted CNT forest.

A study on the influence of the pressure of a liquid on the wetting stability revealed that the CNT forest stands against a pressure of more than 10 kPa without changing the wetting angle.[444] To improve the hydrophobicity of CNT forest, its surface was coarsened by laser radiation.[585] The wetting angle on dense CNT forest varies depending on the applied potential.[586] It should be noted that superhydrophobic CNT forest coatings with relatively low surface density of CNTs are of limited use.

Carbon nanotube forests are promising as components of various sensors. Here, an important role can be played by large specific surface area of CNT forest and by the ratio of the surface area of CNTs to the substrate surface area occupied by the CNT bases. In particular, gas sensors,[463, 568, 569] bio- and immunosensors,[29, 87, 231, 466, 587 – 598] and pressure/touch sensors[484, 599] were tested. Vertically aligned CNT arrays coated with thin polypyrrole films can serve as carriers of various molecules in selective chemical sensors.[432] Films of aligned CNTs on flexible substrates were used to fabricate sensors for detection of different types of human motion.[600]

Mechanical properties, in particular, high elasticity of CNT forests allows them to be utilized as dampers and springs.[413, 414, 418, 601]

Conductive properties of CNTs underlie applications of CNT forest as electrodes,[51, 87, 227, 602] flexible conductors,[603] electrical brush contacts[178, 200, 445] and electromechanical switches.[200] The minimal diameter of micro-electrodes fabricated from CNT forests is 2 μm.[602] Engineers at 'Toshiba' (Japan) are developing integrated circuit interconnectors based on multi-walled CNTs. They reported a CNT packing density of 2×10^{12}; the deposition into substrate pits 70 nm in diameter was carried out by e-beam evaporation method at 450 °C.§ It was proposed to use capillary densified CNT micropillars as electric contacts for information systems.[446] The possibility of using a CNT forest as interface with living systems was suggested.[604]

Thermal properties of CNTs allow CNT forests to be used as heat sinks, in particular, for high-brightness light-emitting diodes,[605] silicon chips[162, 532, 606] and electronic devices.[134] The use of indium solder thin film increases the thermal conduction of CNTs to $\sim 10^6$ W m^{-2} K^{-1}.[399] The first heat sink for semiconductor chips was announced by 'Fujitsu' (Japan) in December 2005. By 2012, this company planned to put on the market a technology for fabrication of CNT IC interconnectors. The properties of densified CNT forest as thermal interface were described.[607]

The superconducting properties of NbC are improved upon coating with a CNT forest.[608]

§ http://www.toshiba.co.jp/tech/review/2011/high2011/high2011pdf/1101.pdf

Many studies are devoted to fabrication of 3D CNT forest configurations of different shape[67 – 76] (*e.g.*, microstructures from square micropillars,[609] cylindrical micropillars[67] or thin plates[328]) and complex structures on substrates.[31, 326, 450, 451] This makes it possible to utilize CNT forests as, *e.g.*, stamps for microprinting (to 50 imprints can be obtained[610]) or for horizontal alignment to form ribbons.[328]

Carbon nanotube forests are used to manufacture various intellectual materials,[231] photoelectric conversion devices[87] and magnetic nanostructures.[105] A CNT forest modified with ferrocene is promising for information storage systems.[521] Patterned CNT arrays may exhibit the properties of photonic crystals.[611, 612] Coatings based on CNT forests with low surface density of CNTs make things invisible.[613, 614] Carbon nanotube arrays on silicon and $LiTaO_3$ substrates act as detectors of IR radiation.[615, 616]

A specific potential application field of CNT forest is fabrication of dry adhesives similar to gecko foothairs.[617 – 620] Gecko lizards can run across the ceiling due to the action of van der Waals forces ($\sim$37.5 N cm^{-2}, see Ref. 621). Large-scale production of such adhesives (probably, for gloves that will allow humans climb any smooth vertical surface) is expected to start in 3 – 5 years, although it is still unclear whether technological difficulties (maximum values attained are 29 (Ref. 622) and $\sim$100 N cm^{-2} (Ref. 623) will allow them to compete with much more readily available polymeric adhesives (maximum value is nearly 3 N cm^{-2}).[622]

Carbon nanotube forests can be utilized as substrates with zero adhesion to nanoscale materials fabricated on them; this enables manipulation of such materials.[624, 625] In this respect, CNT forests are superior to fluoroplastics.

Modified CNT forest films serve substrates for growth of biological cells.[449, 470, 626, 627]

Carbon nanotube forest can be used in converters of low-potential heat to electricity.[628] Studies by Strano and co-workers[629, 630] opened new prospects for application of CNT forest in power engineering. Ignition of thin (7 nm) cyclotrimethylenetrinitramine coating on CNTs caused the combustion front to propagate at a velocity of more than 2 m s^{-1}, which is more than four orders of magnitude greater than the combustion front velocity in bulk material. The effective thermal conductivity was 1.28 kW m^{-1} K^{-1} and the electric pulse generated due to the temperature difference between the combustion front and the cold end of such a 'thermocouple' had a specific power of 7 kW kg^{-1}. This was explained by high thermal conductivity and electrical conductivity of CNTs and named 'thermopower waves'. Also, Strano and co-workers[629, 630] outlined prospects for applications of thermopower waves.

Carbon nanotube membranes represent a macromaterial related to CNT forest.[631, 632] Thin isoporous membranes with open-end CNTs as channels can be formed from CNT forests without difficulties. Here, the pore size can be rigorously controlled by choosing the CNT diameter determined by the growth conditions. The permeability of CNT membranes can also be tuned by uniaxial compression of the membranes.[631] The sequence of operations is shown in Fig. 20. Carbon nanotube membranes are compatible with micro-electromechanical systems.

In addition to inner cavities of open-end CNTs (provided that intertubular space is filled), the space between closed CNTs can serve a channel for liquids and gases. In

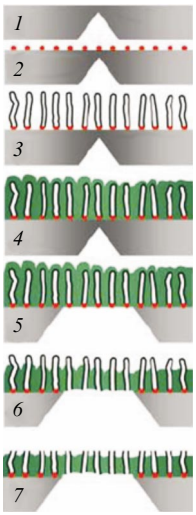


Figure 20. Schematic of the fabrication process of a CNT membrane.[633]
Pit formation (*1*), catalyst deposition (*2*), CNT forest growth (*3*), intertubular space filling (*4*), partial etching of substrate (*5*), partial dissolution of the surface layer of filler (*6*) and CNT uncapping (*7*).

the latter case, the nanotube – nanotube separation can be varied by deposition of oxides on CNTs or by electropolymerization of pyrrole.[470, 479, 565, 634, 635] The permeability and selectivity of polypyrrole-coated membranes can be controlled by applying one or another electric potential.[636]

In spite of a very small diameter of channels (< 2 nm), the high density of channels and increased velocity of transport through them [637 – 641] predetermine a much higher permeability of water and gases through CNT membranes compared to the permeability of polycarbonate membranes. According to calculations, CNT membranes can be utilized for desalination of water.[642] High rate of transport through CNTs can be due to zero friction owing to mirror-symmetrical repulsion of molecules from inner CNT walls and to concerted diffusion mechanism if nanotubes are less than 2 nm in diameter.[637, 640, 642 – 646]

Membranes from dense aligned CNTs with unsealed interstitial pores CNTs were fabricated.[647] Here, filtration occurs through both inner cavities of CNTs and interstitial pores.

One of the first experimental studies that revealed a high flow rate of liquids through CNT membranes was reported in 2000.[648] More recent studies were reviewed.[632, 646, 649]

The permeability of gaseous nitrogen and complex ions $[Ru(NH_3)_6]^{3+}$ through multi-walled CNT membranes was measured, the possibility of electrochemical modification of CNT membranes was shown and the possibility of separation of biological substances on CNT membranes was assessed.[650]

Using conductive polymers, one can fabricate catalytically active membranes based on Pt-decorated CNT forest.[651]

A study [652] showed that no detectable quantity of CNTs, CNT bundles or carbonaceous particles are released to the occupational environment during the growth of a CNT forest and subsequent handling.

* * *

Below we will outline some studies that appeared after this review was submitted for publication.

The Fe catalyst film thickness corresponding to the highest degree of ordering CNTs in the CNT forest was determined.[653, 654] It was shown that a FePt catalyst film of particular thickness can be superior over the Fe catalyst in selectivity of the growth of single-walled or double-walled CNTs.[655] New data were obtained on the kinetics of the CNT forest growth activation and deactivation.[656] A method of synthesis of single-walled CNT forest with relatively large CNT diameter was reported.[657]

Russian researchers from Nizhny Novgorod developed a technology for the synthesis of CNTs with walls from radially oriented multi-walled CNTs and from CNT – chromium and CNT – tungsten composites.[658] Methods of growth of CNT forest with fixed and floating catalysts were compared.[659] The properties of CNT forest for drawing fibres [660] and manufacturing durable conductive fabrics from CNT forests were described.[661] Methods of laser etching of CNT forests and its transfer to other substrates were developed.[662] A method of synthesis of CNT forest using waste plastic from commercially available water bottles was tested.[663]

A videoguide on microstructures fabricated from CNT forests is available.[664] It was shown that the degree of densification of CNT forest using capillary effect can be controlled by varying the duration of contact between solvent vapours and the primary CNT forest.[665]

The influence of the CNT diameter and the density of the CNT forest on its mechanical properties was studied.[659] The viscoelastic behaviour of a CNT forest was studied.[666] A method for attainment of abrupt changes in the optical properties of CNT forest was reported.[667]

A 'dry' method for coating CNTs in CNT forests with polymers for subsequent fabrication of yarn and strong fabrics was developed.[668] Carbon nanotube forests have high sorption capacity ($35\ g\ g^{-1}$) compared to petroleum.[669] A carbon nanotube forest of doped CNTs was as efficient electrocatalyst.[670] New data are available on application of CNT forests in solar cells [671, 672] and fuel cells.[673] A piezoresistive CNT forest sensor was reported.[674]

VII. Conclusion

Arrays of vertically aligned needle-shaped systems (single crystals or polycrystals) a few micrometres or from a few tens to a few hundred nanometres in diameter are known since 1964 when the VLS method was reported for the first time [675] taking silicon as an example (see Introduction). Since then, the technique has been used to obtain nanowires from other semiconductors including elements (Ge), oxides (ZnO, TiO_2, WO_3, *etc.*), selenides (CdSe, PbSe, In_2Se, *etc.*), nitrides (GaN, InGaN, *etc.*), phosphides (GaP, InP, *etc.*), antimonides (AlSb, GaSb, InSb, *etc.*) and solid solutions, as well as to fabricate heterostructures.[676, 677] Arrays of these substances grown on substrates can also be called forests. To date, such systems have been studied in quite detail.

There are some analogies between the mechanisms of forest formation from these substances and the growth mechanism of CNT forest. In particular, both

methods use metal catalysts (most often, gold in the VLS method) and the diameter of the catalyst particle specifies the diameter of semiconductor nanowires or the CNT diameter.

Also, there are significant differences. First, in the VLS method the catalyst particle is always on top of the growing nanowire, whereas in the synthesis of thin CNT forests the catalyst most often remains on the substrate. In the VLS method, the possibility of growth is usually related to the fusion diagram of the catalyst – semiconductor system. The case of CNT forest is more complicated. For instance, it is still unclear why the CNT length is limited to 15 or 20 mm even under supergrowth conditions.

At the same time, none of the semiconductor forests can compete with the relatively young material, the CNT forest, in properties, functionality and potential fields of application. Taking into account continuously increasing interest in CNT forests and rapid appearance of new publications, one can suggest that the potential of this material is far-reaching and new application fields are anticipated. Carbon nanotube forest synthesized by the supergrowth method seems to offer the greatest prospects.

Forest-like microstructures were produced photolithographically and by laser etching (see, *e.g.*, Ref. 678); however, in these cases the particle size is much larger than in the case of CNTs. In addition, forest-like microstructures should be much more expensive than CNT forest; as a consequence, they seem to be of limited use eve in the future.

Usually, a CNT forest comprises CNTs of different diameter and chirality; a fraction of CNTs exhibit metallic conductivity, while the remaining CNTs behave as semiconductors. Would the selective synthesis of a single-walled CNT forest with specified properties be possible, it could act as semiconductor material with tunable bandgap (the bandgap is determined by the CNT diameter and chirality). Solving this problem will open new application fields of CNT forests in micro- and nanoelectronics. The possibilities of selective synthesis of semiconductor CNT forest were suggested [679] and reviewed.[3] Although the material was successfully used in field-effect transistors, a road to versatile carbon semiconductor is still very long.

Presently, many studies on CNT forest reported 7 – 10 years ago and some predictions concerning potential fields of application seem to be somewhat naive; however, they were a necessary step on the way to knowledge and in the evolution of technology. An example of CNT field emitters shows that the period of time from feasibility demonstration to engineering development and industrial application is often very long.

A severe problem is to certify CNT forests and to fabricate reference samples. This was pointed in a study [333] whose authors developed a semi-industrial method of growth of CNT forest on wafers 50 – 100 mm in diameter with simultaneous quality control.

I clearly understand that, taking into account the state of the art of research and development in Russia, the materials presented above can be out of demand for some time. However, I hope that information concerning world-recognized research will favour attempts to change the situation for better.

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