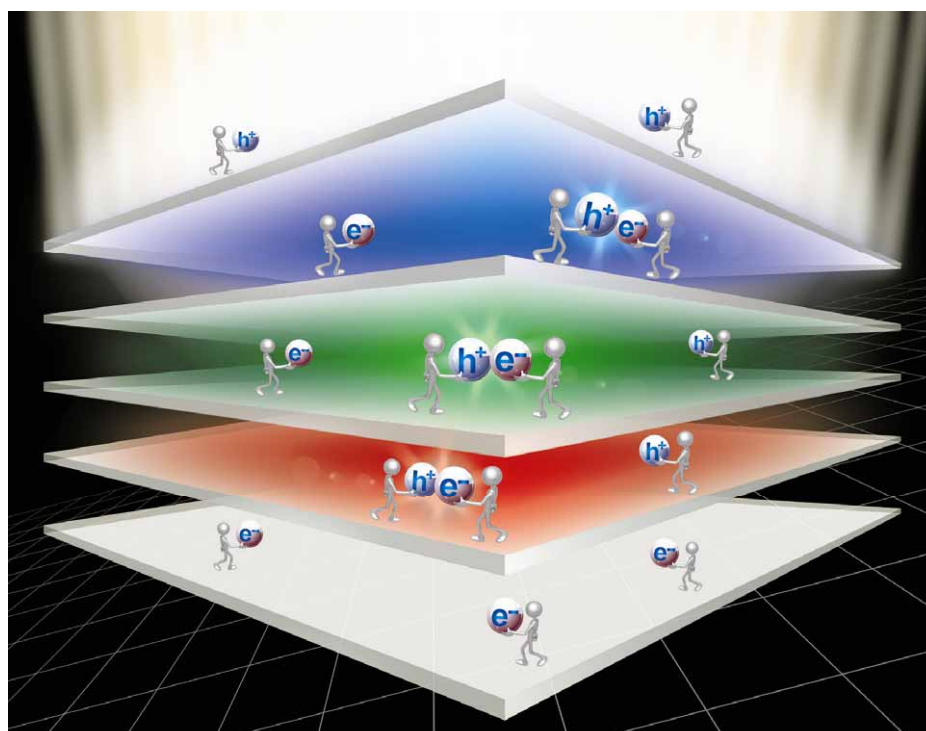


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Nanocarbons transparent conductive films†

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This tutorial review discusses the contradictory material properties of electrical conductivity and optical transparency for the examples of graphene films and carbon nanotube networks. It is argued that for homogeneous films both properties are linked by basic laws of physics and that for perfect monoatomic layers conductivity and transparency can be calculated from the fine structure constant. To beat these limitations, inhomogeneous films are required, such as graphene with an array of holes or nanotube networks. An overview is given on literature values of transparency and conductivity, both for graphene films and for nanotube networks.

1. Introduction

Whenever we encounter a contradiction, science becomes exciting. Conducting plastics are exciting^{1,2} but is plasticity and conductivity a contradiction? Aren't both typical metallic properties? Isn't it plastic flow we use to draw wires and don't we think of wires when we discuss electrical conductivity? Synthetic metals, is that a contradiction? Synthesis means putting together—and alloys like bronze and brass are put-together metals. Of course, nowadays plastics or synthetics remind us of nylon, nylon reminds us of silk, and silk is neither metallic nor electrically conducting. But transparent conducting films are a real contradiction, not only based on connotation but based on arguments of physics.

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Electrical conductivity requires mobile electrons, which move and create a current if we apply voltage to a wire. But these mobile electrons also move if we shine light on to them. Light is electromagnetic radiation and mobile electrons respond to electric fields, no matter whether these fields are created by batteries, power plants, or light. Flowing currents, *i.e.* moving electrons, dissipate energy, they absorb light, and if a metal absorbs light it is not transparent. (We don't discuss superconductivity here—lossless electrical currents—and we don't discuss the reflection of light. But in any case, electrical conductivity and optical transparency are contradictory properties of matter. You can have one or the other, but you cannot have both, see ref. 3). After all, the “optical conductivity” is related to absorption, and in most materials the conductivity increases with frequency. So we expect the absorption to be particularly high for the high frequencies of visible light.^{3–5}

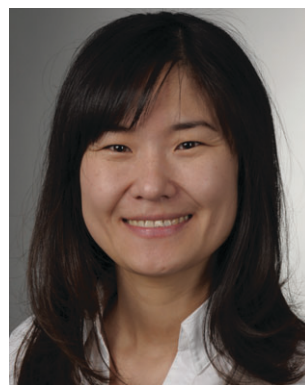
Of course, there is an evident compromise: chicken wire. Look at Fig. 1. This could be a piece of chicken wire. The size of the meshes is about 1 cm, light can easily pass through the holes, the wire is conductive, the whole sheet is conductive,



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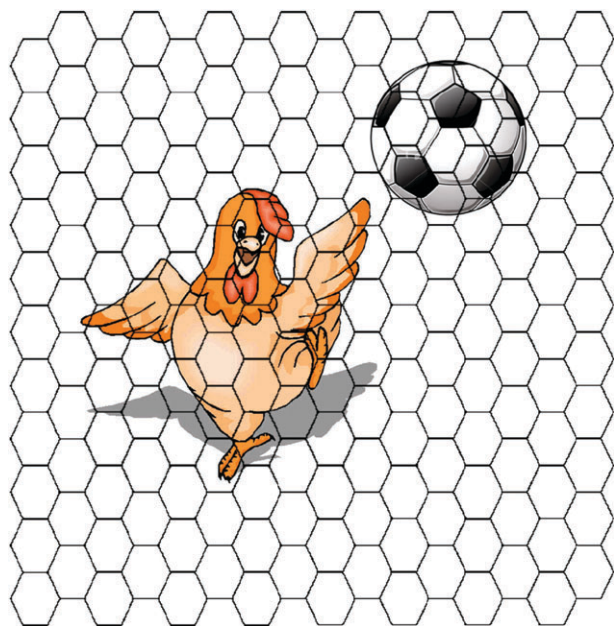


Fig. 1 Net behind a soccer goal, chicken wire, or the hexagonal lattice of graphene (a monolayer of graphite). If interpreted as graphene lattice, there is a carbon atom at each lattice point. Carbon normally is four-valent, but here each carbon atom has only three neighbours, leaving one electron to be shared with the lattice as a whole, pretty much like the electrons in alkali metals (except that in alkali metals the delocalized electrons have a σ wave function and in graphene they have a π wave function). For more details on the conductivity of carbon systems, see ref. 5.

and it is transparent. We can see the chicken through the fence. Normally, we don't want to make use of electrical conductivity of chicken wire. We keep the chicken inside the fence by mechanical forces. But for cattle, we often use electrical fences—and, alas, for human prisons, too. How small must the holes be in a chicken wire? Apparently they must be smaller than a chicken. And if the film is supposed to be used as the top layer of a solar cell, the holes must be smaller than the electron recombination length, so that the photogenerated charge can be efficiently collected. How large must the holes be? If they are much larger than the wavelength of light, light will have no problem to pass. The wavelength of visible light is about $1\ \mu\text{m}$. The chicken wire meshes are about $1\ \text{cm}$. So the ratio is four orders of magnitude. But even if the meshes were a little bit smaller than the wavelength of the light, light might be able to pass. Th. Ebbesen,⁶ for example, has shown that copper sheets become transparent if we drill an array of nanoholes into them, say with $100\ \text{nm}$ diameter. (Light will excite surface plasmons on the walls of the holes, the light will pass “in the near field”, and on the backside of the sheet light will form electromagnetic waves again.)

We look again at Fig. 1: Actually, this is not a photograph of chicken wire. It is the lattice structure of graphene, a hexagonal (honeycomb-like) crystal lattice. At each corner of the hexagons, there is a carbon atom and the distance between two carbon atoms is about $1.4\ \text{\AA}$ ($0.14\ \text{nm}$)—now four orders of magnitude smaller than the wavelength of light. Would light still be able to pass? There are two reasons for NO: Firstly,

there is a minimum hole size for surface plasmons to form.^{7,8} This is much larger than the lattice constant of graphene, therefore surface plasmons will not be able to form in the holes of graphene. Secondly, the hexagons are not empty holes. The edges of the hexagons symbolize the chemical bonds between the carbon atoms, these bonds are not tiny wires, but π -electron wave functions of the polyaromatic graphene system. These wave functions fill the total space quite well.^{9,10}

2. Graphene

When the field of synthetic metals started, in the 1970's, intercalated graphite was one of the materials of interest.^{11,12} Normal graphite is metallic, or nearly metallic, with a fairly high conductivity along the (a,b) planes and a much lower conductivity in the c direction (perpendicular to the planes). If acceptor molecules are intercalated between the graphite planes, the in-plane conductivity increases considerably, and the perpendicular conductivity decreases, leading to a highly anisotropic material ($\sigma_{ab} \approx 1000\sigma_c$). Already at that time the term “graphene” was used for a monoatomic layer of graphite. Fig. 2 shows how most of the conducting polymers are related to graphene.⁵ If we had a method of cutting with atomic precision, we could cut the conducting polymers out of the graphene sheet (and in some cases we would have to replace some carbons by heteroatoms).

In 2004 A. Geim¹³ and only a little bit later Zhang *et al.*¹⁴ succeeded in isolating small graphene flakes, contacting them and carrying out electrical transport measurements. Meanwhile quite large graphene films can be prepared, several cm^2 in size¹⁵ or even 30 inch rolls.¹⁶ Most of them are not really monolayers, they consist of “a few” layers, say 2 to 10, and are called oligographene (oligo = a few). Are these layers

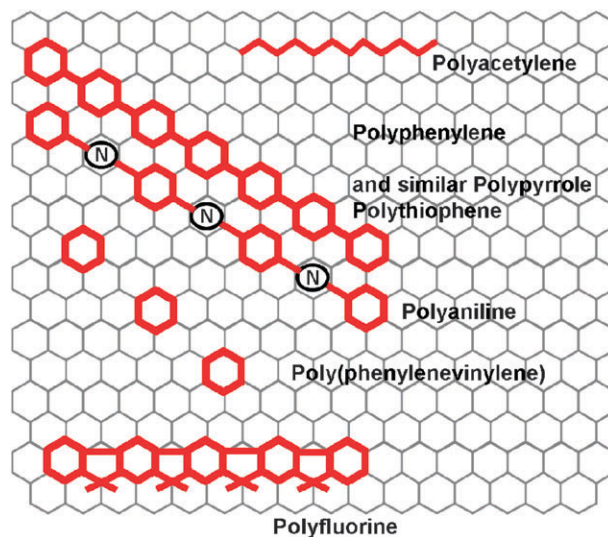


Fig. 2 Relation between graphene and conducting polymers. The conducting polymers can be envisaged as cut out from graphene (with the occasional replacement of carbon by nitrogen, sulfur, or oxygen). Graphene and conducting polymers have in common that most carbon atoms have only three neighbors instead of four, so that there are some electrons shared by the whole structure, pretty much like in ordinary metals.⁵

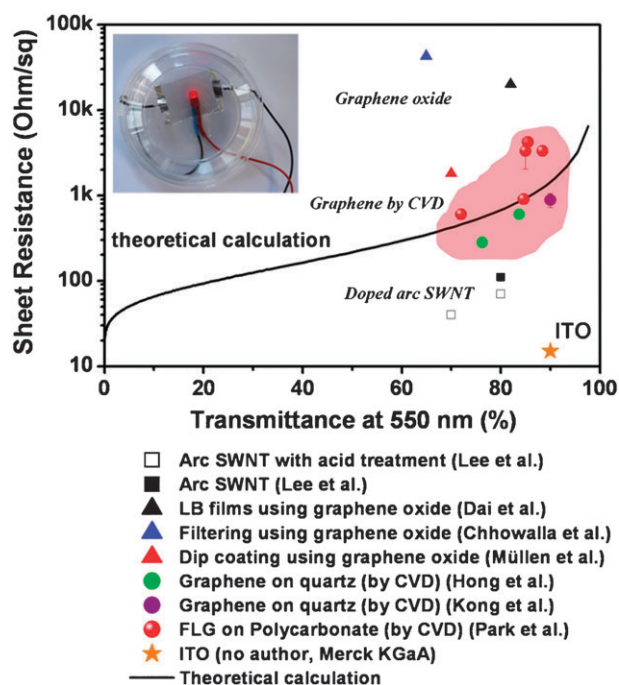


Fig. 3 Sheet resistance *versus* transparency for few-layer graphene, carbon nanotube networks and conductive transparent metal oxides. The solid curve is based on ref. 17.

conducting and transparent? We can easily measure and some of the experimental data are compiled in Fig. 3. Here the sheet resistance is plotted *versus* the transparency of thin films. “Sheet resistivity” (or “surface resistivity”, or “square resistivity”) is a measure of the conductivity of films when the film thickness or the thickness of the conductive surface coating is not known or not relevant. It is defined as the resistance of a square of any size (one can easily convince oneself that all squares have the same resistance, independent of the size: increasing the square will make the sample longer = increase in the resistance, and it will make it wider at the same time = decrease in resistance). In these plots the transmittance (transparency) plays the role of something like an effective sample thickness. Evidently, samples with a high transparency have a high surface resistance and *vice versa*. This is the contradiction between conductivity and transparency mentioned above.

We can measure transparency and conductivity in graphitic monolayers and in “few-layer graphene”, but we don’t really have to. We can easily calculate these quantities from general laws of physics, just using universal constants like the velocity of light, Planck’s constant and the elementary charge. These combine to the famous “fine structure constant”, which has the value of $1/137$. Plugging this into respective formulas¹⁷ leads to about 2.3% of light absorption per monolayer and to a square resistivity of about 6 k Ω . If we want to reduce the resistance we have to take several layers, but this will also reduce the transparency. These theoretical values (based on ref. 17) are plotted in Fig. 3 as a solid line.

Our theoretical considerations are not restricted to graphene, they would—with some changes—apply to any metallic monolayer or oligolayer. But we cannot make monolayers of

ordinary metals. Ordinary metals like to be three-dimensional and monolayers disintegrate into islands or droplets. It is due to the directional valance forces of graphitic carbon that we can form layer structures (the keywords here are “conjugated double bonds”, “polyaromaticity”, and “sp² hybridisation”).^{5,9,10}

Fig. 3 also contains experimental values.^{18–20} Several of them fit quite well to the theoretical curve. Some are below the curve. How can that be? Firstly, the theoretical curve is for “undoped” graphene. We might change the conductivity by doping. Doping means oxidation or reduction of the sample, *i.e.* changing the carrier density by removing or adding electrons. Some samples might have been doped without the intention of the experimenters, by contamination from laboratory air or from solvents during the process of film preparation. But, as outlined above, increasing the conductivity will inevitably increase the absorption of light and hence it will reduce the transparency (perhaps not exactly following the curve in Fig. 3). Secondly, there might be inhomogeneities in the films, some parts might be more transparent, others more conductive. Extreme inhomogeneities are holes (see the chicken wire model!). We will deal with “holey graphite” in Section 4. Other values below the curve in Fig. 3 are from carbon nanotube networks. Such networks are *per se* inhomogeneous. Transparent conducting carbon nanotube networks will be treated in Section 5.

There is a data point in the lower right hand corner of Fig. 3 marked “ITO”. ITO stands for indium tin oxide, a conducting metal oxide, which is the bench mark material of today for transparent conductors. For a review on inorganic transparent conductors, see ref. 21. Conducting metal oxide layers are fairly thick and brittle. Moreover, the price of indium has dramatically increased during the last years, “as the world seems to run out of indium”. That is why materials scientists urgently search for a replacement of ITO. Metal oxides normally are wide-bandgap semiconductors. They are transparent and not conducting. But these oxides are off-stoichiometric (with oxygen vacancies), there will be localized electronic states in the band gap and a compromise between conductivity and transparency can be obtained. Why is ITO transparent and conducting? In the view of the above sections, there are spacial fluctuations in the defect concentration, rendering some spots more conducting than others, so that the materials exhibit some aspects of chicken wire.

3. Preparation of graphene layers

The inset in Fig. 3 is a photograph of a light emitting diode behind a graphene film. This is just a catchy demonstration to show that few-layer graphene is really transparent and that large films of such a material can be made.

Small samples of graphene can be made by a method now dubbed “micromechanical cleavage”. This technique is quite simple and has already been employed when intercalated graphite was *en vogue*: A small natural crystal of graphite or a piece of highly oriented pyrolytic graphite (HOPG) is sandwiched between two strips of scotch tape. By separating the tapes the graphite crystal is thinned and the process is repeated so often till only graphite stacks a few layers thick are left. Andrei Geim and others have improved the method so

far, that monolayers can be obtained and deposited on silicon substrates or on electron microscope grids.^{13,22,23} Similarly, one can just write a line with a pencil (the core of which is graphite!) onto a silicon chip and look with an atomic force microscope at the ribbed off graphite flakes until one finds a monolayer (experienced experimentalists can even tell from the interference color in an optical microscope, whether a flake is a monolayer). Graphene flakes from micromechanical cleavage have typical dimensions of 10 or 100 micrometres. This is large enough for electrical measurements, but not for front layers of solar cells.

If silicon carbide is heated in vacuum, the vapor pressure of silicon is larger than that of carbon. Therefore a carbon-rich layer forms on the surface.^{24–26} Sometimes this layer is a graphene monolayer, and sometimes it can even be transferred to other substrates. Again only fairly small samples are obtained.

There are various methods for chemical, electrochemical, or sonochemical (ultrasound) exfoliation of graphite or combinations thereof. These methods yield a suspension of few-layer or monolayer graphene flakes, graphite oxide flakes, or some other chemical derivatives. Transparent substrates can be coated with these suspensions (dip coating, spray coating, spin coating, Langmuir–Blodgett technique) and after an annealing step to remove oxygen, other functional groups, or dispersion agents, transparent conducting films can be obtained. The methods of suspension coating can easily be scaled up for industrial production of large-area films.

An alternative method for producing large transparent conducting graphene films is chemical vapor deposition. The photograph in Fig. 3 shows a graphene film originally deposited on a silicon chip onto which a 300 nm nickel layer had been evaporated.¹⁵ Methane (mixed with argon and hydrogen) was used as carbon feedstock and the CVD reaction was carried out at about 1000 °C. After cooling to room temperature a thin polycarbonate layer was spin-coated onto the graphene layer and the nickel film was etched away chemically, so that finally the graphene film had been transferred to polycarbonate. Li *et al.*^{27,28} have shown that CVD of graphene also works on copper films, and finally Sukang Bae *et al.*¹⁶ have scaled this process up to 30 inch rolls.

Table 1 shows a compilation of resistance and transparency values of various transparent conducting graphene films reported in the literature.

4. Graphene layers with holes

To beat perfect graphene (or any other homogeneous layer) we will have to drill holes into our sample and to turn it into some form of chicken wire. Can we do that? There is a material known as “holey carbon”. A special brand thereof is Quantifoil. This is carbon with 2–3 µm wide holes. We have not measured the conductivity of Quantifoil, but we use it as a support for few-layer graphene and to look at graphene through the holes. A typical example is shown in Fig. 4.¹⁵ For many applications, *e.g.* for top electrodes of organic solar cells, the holes in holey carbon would be too large. (They should be smaller than the mean free path of photogenerated charge carriers in the photoactive semiconductor under the electrode). Perhaps smaller holes can be “drilled” into graphene by first introducing point defects, say replacing some carbon atoms by nitrogen atoms, and then chemically building onto the defects. The first step in this direction is shown in Fig. 5. This figure is a special high resolution electron transmission microscope (HRTEM)

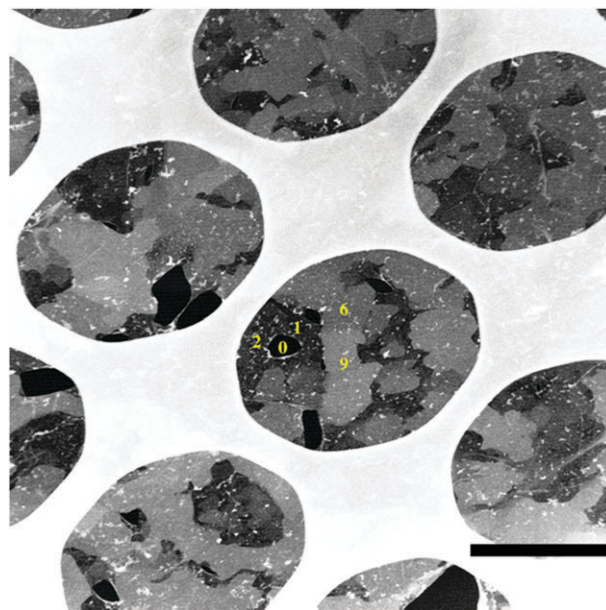


Fig. 4 Quantifoil (holey carbon): Thin carbon films with micrometre-sized holes as support for samples in transmission electron microscopy. In this case Quantifoil has been used to look through the holes to graphene (scale bar: 2 µm).¹⁵

Table 1 Comparison of sheet resistances of graphene based transparent conductive films

Year	Author	Preparation of graphene	Method of transparent conductive film	Sheet resistance	Transmittance at 500–550 nm
2007	Wang ²⁹	Graphene oxide/reduction	Dip coating	1.8 kΩ/sq	> 70%
2008	Wu ³⁰	Graphene oxide/reduction	Spin coating	5 kΩ/sq ~ 1 MΩ/sq	> 80%
2008	Li ¹⁹	Exfoliated graphene by molecule/annealing	Langmuir–Blodgett (LB)	8 kΩ/sq	80%
2008	Blake ³¹	Exfoliation in DMF with sonication	Spray deposition	5 kΩ/sq	90%
2008	Blake ³¹	Microcleavage/chemical doping	Transfer	400 Ω/sq	98%
2010	Lee ³²	Exfoliation in liquid/nitric acid treatment	Filtration/transfer	3 kΩ/sq	80%
2010	Yamaguchi ³³	Graphene oxide/reduction	Spin coating/transfer	1 kΩ/sq	70%
2009	Reina ³⁴	CVD/Ni	Etching/transfer	770 Ω/sq ~ 1000 Ω/sq	90%
2009	Park ¹⁵	CVD/Ni	Etching/transfer	600 Ω/sq	85%
2009	Kim ³⁵	CVD/Ni	Etching/transfer	280 Ω/sq	90%
2009	Li ²⁸	CVD/Cu	Etching/transfer	350 Ω/sq	90%
2010	Bae ¹⁶	CVD/Cu	Etching/transfer	40 Ω/sq	90%

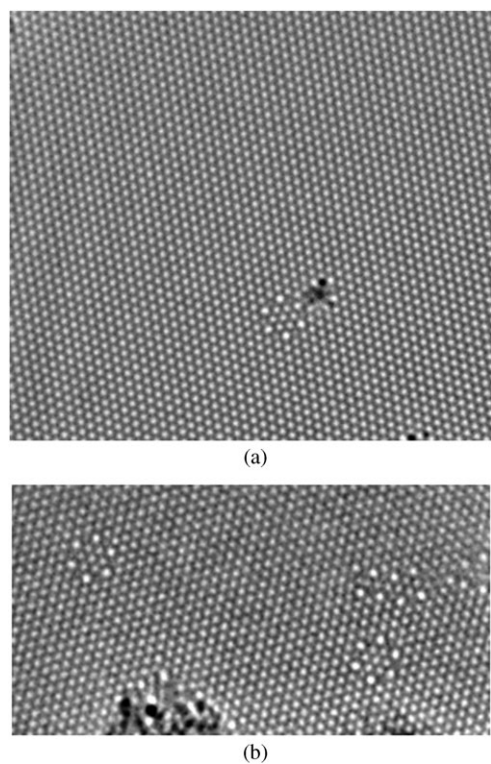


Fig. 5 High resolution transmission electron micrographs of nitrogen-doped graphene: Individual point defects of substituted nitrogen are seen, as well as a flower defect consisting of several pentagons and heptagons and rotated hexagons (a) low concentration (b) high concentration.³⁶

image of “nitrogen-doped” graphene.³⁶ We see various points of nitrogen substitution and a so-called flower defect, consisting of several pentagons and heptagons and rotated hexagons. Can we attack this defect with further chemistry, so that it acts like a hole for the passage of light?

Recently, Nemes-Incze *et al.*³⁷ reported graphene with hexagonal holes which have zigzag edges using a simple carbothermal reaction between SiO₂ and carbon. They generated regular point defects on graphene located on a SiO₂/Si substrate using the tip of an AFM probe as an indentation tool. By annealing the sample, hexagonal pits can be grown starting from the puncture holes introduced by the AFM tip. The generated hexagonal holes have zigzag edges which don't act as a scattering point of light. The results can inspire large graphene films on glass having nano-sized hexagonal holes as a new type of transparent conductive electrode.

5. Carbon nanotube networks

But why bother with chemistry on graphene, if we have carbon nanotube networks? Carbon nanotubes are rolled-up graphene. A recent monograph on carbon nanotubes is given in ref. 39. There are two types of carbon nanotubes depending on the number of rolled-up graphene layers. A single-walled carbon nanotube (SWNT) has one rolled-up graphene layer and a multi-walled carbon nanotube (MWNT) has more than two rolled-up graphene layers as a wall. In this article, we focus on SWNT, because networks of SWNTs allow for a

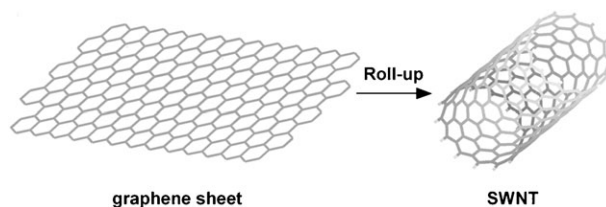


Fig. 6 Hypothetical rolling of graphene into a single-walled carbon nanotube—or unrolling a nanotube into a graphene ribbon.³⁸

better compromise between electrical conductivity and optical transparency.

Fig. 6³⁸ illustrates how one can imagine rolling graphene into nanotubes (of course, the real synthesis is different: In most synthetic routes a carbon plasma condenses in the presence of catalytic metal nanodroplets^{39,40}). Nanotubes conduct electricity as well as graphene, and they can easily form networks with holes for the light to pass. Usually these networks are formed by first preparing a suspension of carbon nanotubes (using a detergent and strong ultrasonic agitation) and then coating a substrate with the suspension, just as in the case of films from graphene suspensions (dip coating, spray coating, spin coating). Alternatively, transparent conducting nanotube networks can be grown by *in situ* chemical vapor deposition, *i.e.* by evaporating islands of catalyst particles (usually of transition metals like Ni, Fe, or Co) onto the substrate, and then growing nanotubes onto the islands, which finally will connect the islands to form a highly conducting network. Since we need a temperature of about 1000 °C to grow single-walled nanotubes by the CVD method this procedure cannot be applied to polymer substrates. One could think, however, of developing techniques of transferring nanotube networks from one substrate to another.

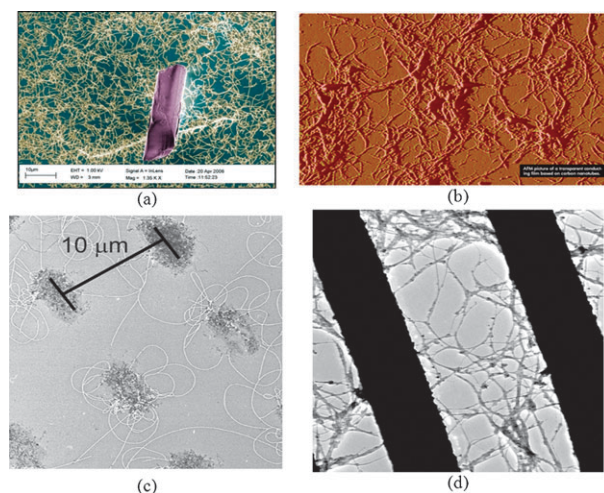


Fig. 7 (a) A carbon nanotube network made by casting a droplet of a nanotube suspension onto a silicon chip. SEM image. The large particle is the debris of a human hair.⁴¹ (b) Nanotube network sprayed on a silicon chip. AFM image (atomic force micrograph).⁴² (c) CVD grown carbon nanotubes connecting dots of metallic catalyst on a silicon-nitride membrane. SEM image.⁴³ (d) CVD grown nanotubes between metal stripes acting both as catalyst for nanotube growth and as electrical leads.⁴⁴

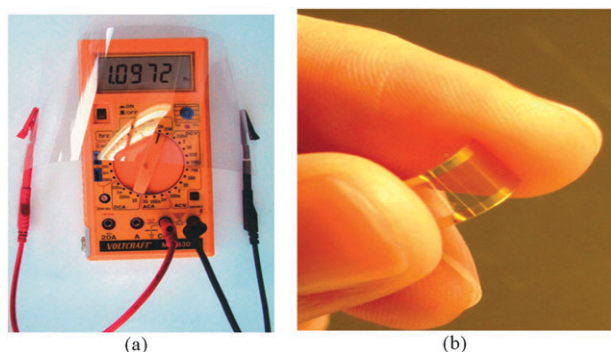


Fig. 8 (a) Ohmmeter seen through conducting transparent nanotube network spray-coated to a polycarbonate film, contacted with alligator clips.⁴² (b) Large-area transparent field-effect transistor consisting of two nanotube films, one semiconducting and the other metallic, separated by a parylene dielectric.⁴⁵

In Fig. 7(a)⁴¹ we see a SEM (scanning electron microscope) image of a nanotube network made by casting a nanotube suspension onto a silicon chip (as “scale” we have left the fragment of a student’s hair in the image, which incidentally had fallen onto the sample). Fig. 7(b–d) are further SEM images of nanotube networks. Fig. 7(b):⁴² tubes sprayed onto a chip, Fig. 7(c):⁴³ CVD grown nanotubes between catalyst dots on a silicon-nitride membrane, and Fig. 7(d):⁴⁴ CVD grown tubes, now between metal stripes which serve both as catalyst for nanotube growth and as leads for electrical conduction.

In Fig. 8(a)⁴² we present a photograph of a spray-coated polycarbonate film contacted by alligator clips for conductivity measurements. We see the ohmmeter behind the transparent film. It reads 10.9 MΩ. More sophisticated is the device shown in Fig. 8(b):⁴⁵ a large-area carbon nanotube-based field-effect transistor. Here there are two transparent nanotube networks, a semiconducting and a metallic one. They are separated by a thin dielectric of parylene. The semiconducting and metallic properties of the networks are controlled by adjusting the density: normal SWNT samples consist of a 2:1 mixture of semiconducting and metallic nanotubes. If the network is so dense that the metallic tubes form continuous paths, the overall behavior is metallic. At lower density, interrupted metallic paths are connected by semiconducting tubes, the

network is overall semiconducting and can serve as the active channel of a field-effect transistor.⁴⁵

In Table 2 we have compiled selected data on transparent conducting nanotube networks from the literature.

6. Summary and outlook

We have pointed out that, when looking for transparent conducting films, we have to make a compromise between transparency and conductivity. These two properties are linked by basic laws of physics and one cannot have high conductivity and high transparency at the same time. For homogeneous monoatomic films like graphene these quantities can be calculated from the fine structure constant. To beat these limitations we have to go to inhomogeneous films such as graphene with an array of holes or to nanotube networks. Fig. 3 and Tables 1 and 2 give an overview of the transparency and conductivity values obtained today. There are already several data points below the theoretical limit for homogeneous films. We expect that in the near future further progress will be made, both in graphene films and in nanotube networks. As for nanotube networks, the major bottleneck is the resistance from tube to tube. When two tubes cross in a network they touch at one point only, which certainly is not the best situation for electrical conductivity. A possible solution might be networks consisting both of nanotubes and of graphene flakes. In the ideal case a graphene flake would squeeze between the tubes and increase the contact area, as indicate in Fig. 9.

Presently the conductivity (at same transparency) of the best nanotube networks is still by an order of magnitude lower

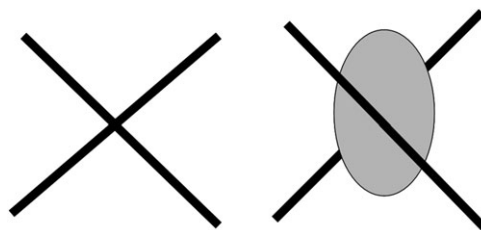


Fig. 9 Graphene chiplet between nanotube cross to improve electrical contact from tube to tube.

Table 2 Comparison of sheet resistances from SWNT based transparent conductive films

Year	Author	Preparation of SWNT	Method of transparent conductive film	Sheet resistance	Transmittance at 500–550 nm
2004	Wu ⁴⁶	Dual pulsed laser vaporization	Filtration/transfer	30 Ω/sq	> 70%
2006	Zhang ⁴⁷	HiPCO or arc discharge (P3)	Filtration/transfer	HiPCO (7200 Ω/sq)	87%
			SOCl ₂ doping	P3(380 Ω/sq)	
2007	Geng ¹⁸	Arc discharge	Spray coating/HNO ₃ doping	40 Ω/sq	70%
				70 Ω/sq	80%
2007	Andrade ⁴⁸	HiPCO	Dipping coating	186 Ω/sq	86%
2008	Green ⁴⁹	HiPCO	Filtration/transfer	140 Ω/sq	> 70%
		Laser-ablation			
		Arc discharge			
		Sorting metallic SWNTs			
2008	Jackson ⁵⁰	Arc discharge	Filtration/transfer	170 Ω/sq	80%
			Doping (SOCl ₂ or HNO ₃)	140 Ω/sq	
2010	Kim ⁵¹	CVD	Spray coating/densification (D)	259 Ω/sq (before D)	80%
				150 Ω/sq (after D)	

than that of the inorganic benchmark material ITO. But nanotube networks are less brittle, chemically more stable, and in the near future less expensive, so that transparent conducting graphene layers and carbon nanotube networks are already of high technological relevance today.

Acknowledgements

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