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Review

Phemenology of Filling, Investigation of Growth Kinetics and Electronic Properties for Applications of Filled Single-Walled Carbon Nanotubes

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Abstract: This review discusses the phemenology of filling, the investigation of kinetics, and the electronic properties for applications of filled single-walled carbon nanotubes (SWCNTs), and summarizes five main achievements that were obtained in processing the spectroscopic data of SWCNTs filled with metal halogenide, metal chalcogenide, metal and metallocenes. First, the methods of processing kinetic data were developed to reveal precise trends in growth rates and activation energies of the growth of SWCNTs. Second, the metal-dependence of kinetics was revealed. Third, metallicity-sorted (metallic and semiconducting) SWCNTs were filled with a range of substances and the electronic properties were investigated. Fourth, new approaches to processing the data of spectroscopic investigations of filled SWCNTs were developed, which allowed more reliable and precise analysis of the experimental results. Fifth, the correlation between the physical and chemical properties of encapsulated substances and the electronic properties of SWCNTs were elucidated. These points are highlighted in the review.

Keywords: carbon nanotube; filling; kinetics; electronic properties; Raman spectroscopy; near edge X-ray absorption; fine structure spectroscopy; photoemission spectroscopy; optical absorption spectroscopy

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1. Introduction

Single-walled carbon nanotubes (SWCNTs) are promising materials for a wide range of applications, such as molecular electronics, nanocomposites, solar cells, sensors, and electrochemistry lithium-ion batteries (Figure 1) [1–9].

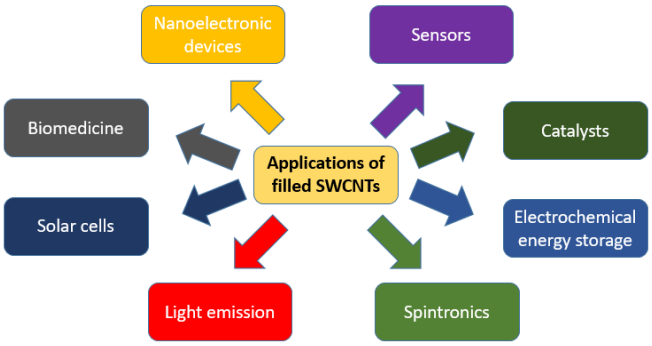

Figure 1. Applications of filled SWCNTs.

SWCNTs are modified in different ways to perform the functionalization of the inner, outer surface and insert substances in inner channels, i.e., perform filling. These methods

include covalent functionalization of the outer surface, noncovalent functionalization, substitution of atoms in the walls of SWCNTs, electrochemical doping, filling and nanochemical reactions within the nanotube channels [10]. The growth rates and activation energies of the growth of carbon nanotubes inside SWCNTs are calculated [11,12]. The kinetics of growth of the inner tubes are studied in detail by the annealing of metallocene-filled SWCNTs with a resolution of several minutes. It has been shown that nickel catalyst leads to higher growth rates at lower temperatures than cobalt catalyst. The activation energies are different for the two metals. Understanding the growth mechanism of SWCNTs leads to a better understanding of growth and electronic properties. The electronic properties of filled SWCNTs are studied mainly by Raman spectroscopy, near-edge X-ray absorption fine structure spectroscopy (NEXAFS), photoemission spectroscopy (PES) and optical absorption spectroscopy (OAS) [13–44]. Raman spectroscopy shows that there is a charge transfer in nanotubes filled with 3d, 4d, 4f, 5s and 6p-metal halogenides. We observed changes in the spectra shapes and shifts of peaks. NEXAFS reveals the formation of local chemical bonds. Photoemission spectra reveal shifts in the peaks. i.e., direct confirmation of doping with a charge transfer. The optical absorption spectroscopy confirms the charge transfer. These studies, which have been performed for almost two decades, are very important. They can lead to many excellent applications, in many different countries, including Russia.

In this review we discuss the phemenology of filling, the investigation of growth kinetics, and the electronic properties for the application of SWCNTs (Figure 2).

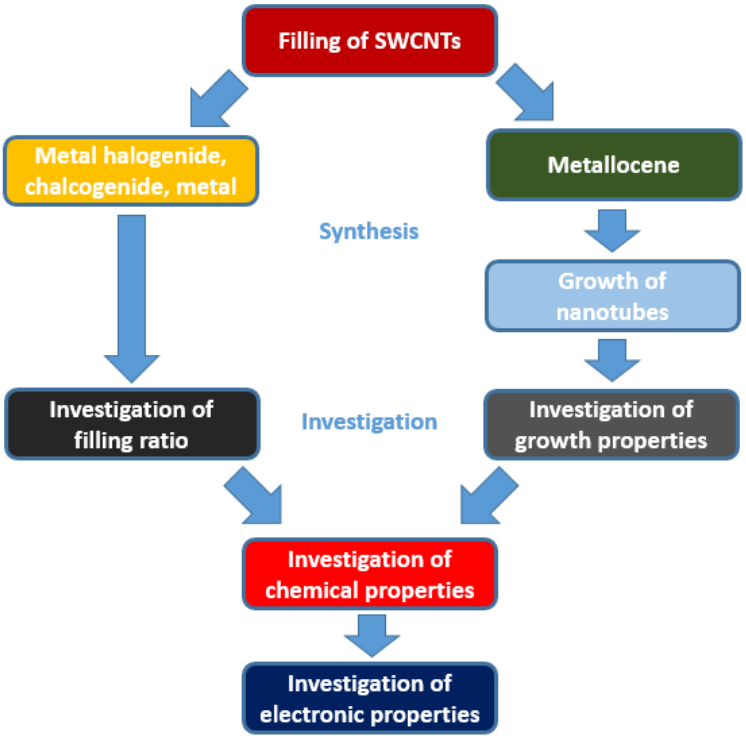

Figure 2. The schematics of filling of SWCNTs with metal halogenides, metal chalcogenides, metals, metallocenes, and investigation of growth properties, chemical properties and electronic properties.

The SWCNTs were filled with molecules [45–119], metals and nonmetals, such as Ag, Cu, P, As, and I [120–153], metal halogenides, such as MHal (M=Ag, Cu, Hal=Cl, Br, I), $MHal_2$ (M=Fe, Co, Ni, Mn, Zn, Cd, Pb, Hg, Hal=Cl, Br, I), and $MHal_3$ (M=Pr, Tb, Tm, Lu, Hal=Cl, Br, I) [154–207], and metal chalcogenides, such as MX (M=Ga, Sn, X=S, Se, Te), and $M_{2\times3}$ (M=Bi, X=Se, Te) [208–214]. They are filled inside SWCNTs by the gas or melt methods; the synthesis process involves thermal treatment with cooling, and an investigation with spectroscopic techniques for applications.

In Section 2 of this review, we consider the kinetics of growth of carbon nanotubes, and trends in the metal-dependence of growth rates and activation energies. In Section 3, we carry out an investigation into the doping effects in metallicity-sorted SWCNTs filled with inorganic compounds. In Section 4, we produce a comparison of the doping effect of different inorganic compounds on SWCNTs. Section 5 comprises a comparison of the doping effect of inorganic compounds on different diameter SWCNTs. In Section 6, a discussion of the influence of different encapsulated substances on the electronic properties of SWCNTs is performed.

2. Studies on Filled SWCNTs

Molecules have been encapsulated inside SWCNTs in 74 papers [45–119], and the filled SWCNTs have been investigated for applications in spintronics, construction material, magnetic storage, and magnetic recording. These applications were reviewed by us recently [214]. They were also reviewed by the groups of Jeremy Sloan [215], Marc Monthioux [216], and Ferenc Simon [217]. The following studies are available:

- The filling process of fullerenes and derivatives, as well as applications were studied by Hiromichi Kataura, Kazu Suenaga, Sumio Iijima, and Hisanori Shinohara [49,51,52,58,70–72,75,76,79,80,116].
- Molecule-filled SWCNTs were synthesized for the investigation of growth kinetics of carbon nanotubes. The fullerenes coalesce and form an inner nanotube. The mechanism was studied by our group [218].
- The synthesis process and catalysis properties of endohedral fullerene-filled SWCNTs were studied by Andrey Khlobystov and Thomas Chamberlain [48,53–55,81,83–86,112].
- The filling process of SWCNTs with other molecules was analyzed by Ákos Botos and Katalin Kamaros [46].
- The carbon nanotubes were also filled with molecules other than fullerenes, e.g., metallocenes, by Hidetsugu Shiozawa [59,95–98,109–111] and Marianna Kharlamova [105–108]. The growth properties of molecule-filled SWCNTs were reviewed in Ref. [219] where the growth mechanisms of carbon nanotubes were also discussed extensively.

Metals and nonmetals have been encapsulated inside SWCNTs in 33 papers [120–153]. These papers are dedicated to the filling of SWCNTs with iodine, phosphorous, arsenic, sulfur, selenium, silver, copper, and europium.

- Extensive studies of the microstructure of filled SWCNTs have been performed.
- The filling processes of SWCNTs have been analyzed, and the procedures have been optimized for the filling.
- The electronic properties have been investigated by spectroscopy.

Among Russian scientists, Elena Obraztsova [123], Alexander Okotrub [129], and Marianna Kharlamova [141,142,147] filled SWCNTs with simple substances.

Metal halogenides and metal chalcogenides were filled inside SWCNTs in 60 papers [154–214]. They are listed above. Here, we present an overview of the novelties in the investigation of the kinetics and electronic properties of filled SWCNTs. The following was achieved [10].

- The methods of processing of the kinetic data were developed to reveal precise trends in growth rates and activation energies of the growth of SWCNTs.
- The metal-dependence of kinetics was revealed.
- Metallicity-sorted (metallic and semiconducting) SWCNTs were filled with a range of substances. Indeed, only metallicity-mixed SWCNTs have previously been used for the filling [10]. The filling of metallicity-sorted SWCNTs allowed for the unambiguous assessment of the influence of encapsulated substances on the electronic properties of SWCNTs.
- New approaches to processing the data of spectroscopic investigations of filled SWCNTs were developed, which allowed for a more reliable and precise analysis of the

experimental results and for the drawing of clear conclusions about the influence of different fillers on the electronic properties of SWCNTs.

- The correlation between the physical and chemical properties of encapsulated substances and their influence on the electronic properties of metallicity-sorted and mixed SWCNTs with different diameters was elucidated.

3. Kinetics of Growth of SWCNTs

The growth kinetics of SWCNTs are important, because it allows control of the synthesis process of carbon nanotubes. The parameters, such as the growth rates and activation energies of the growth of SWCNTs, allow us to choose synthesis conditions that lead to the highest yield of SWCNTs. The investigation of the growth mechanism of carbon nanotubes allows for the determining of the growth model and appropriate mathematical model for describing the experimental growth.

In Refs. [11,12], the kinetics of growth of carbon nanotubes inside nickelocene- and cobaltocene-filled SWCNTs were investigated. The synthesis was performed from the gas phase, followed by an investigation into the atomic structure, growth properties, chemical properties and electronic properties for applications.

Figure 3 shows the Raman spectroscopy data of cobaltocene-filled SWCNTs annealed at a temperature of 580 °C for 2–4168 min, acquired at laser wavelength of 633 nm and 568 nm [11]. The spectra demonstrate the increase in intensity of peaks of the inner tube at 175–325 cm^{-1}, which corresponds to a growth of inner SWCNTs inside filled SWCNTs.

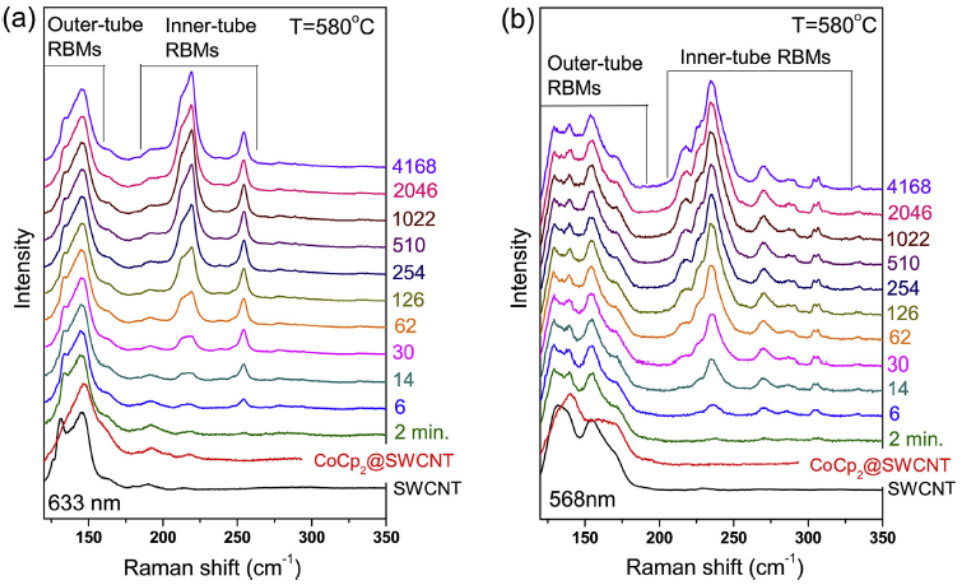

Figure 3. The Raman spectra of cobaltocene-filled SWCNTs annealed at temperature of 580 °C for 2–4168 min, acquired at laser wavelength of 633 nm (**a**) and 568 nm (**b**). Reprinted from M. V. Kharlamova et al. Chiral vector and metal catalyst-dependent growth kinetics of single-wall carbon nanotubes, Carbon, 2018, V. 133, pp. 283–292, Copyright 2018, with permission from Elsevier [11].

The growth rates and activation energies of the growth of inner SWCNTs were calculated for cobaltocene- and nickelocene-filled SWCNTs. Figure 4 compares the activation energies of the growth of nanotubes on cobalt and nickel clusters, formed upon the heating of metallocene-filled SWCNTs [220]. The activation energies E_α and E_β of an initial fast and subsequent slow growth on the purely metallic Ni and Co particles equal ~1.5–1.9 eV and ~0.8–1.8 eV, respectively. For large tubes with a diameter above ~0.95 nm on the Ni catalyst, they are significantly larger than on the Co catalyst, and the values of the smaller tubes are equal. The difference in the Ni and Co is due to different diffusion rates.

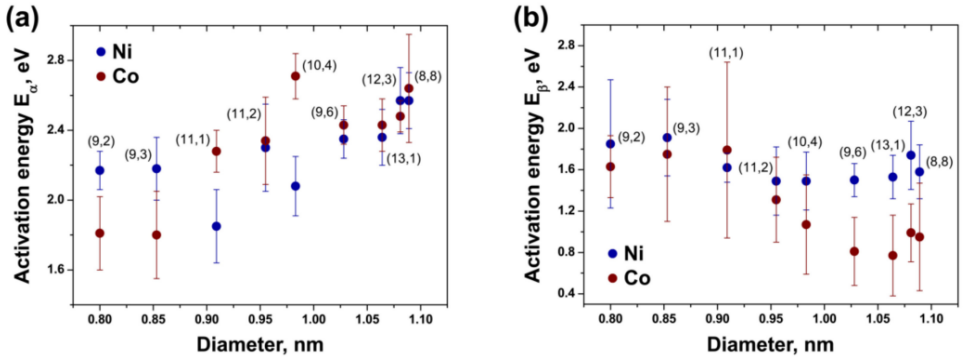

Figure 4. The dependence of activation energy E_α (**a**) and E_β (**b**) of growth of inner nanotubes on their diameter. Reproduced from [215]. Copyright 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

4. Investigation of Doping Effects in Metallicity-Sorted SWCNTs Filled with Inorganic Compounds

Recently, new reports on the filling and investigation of metallicity-mixed and metallicity-sorted nanotubes were published (Figure 5).

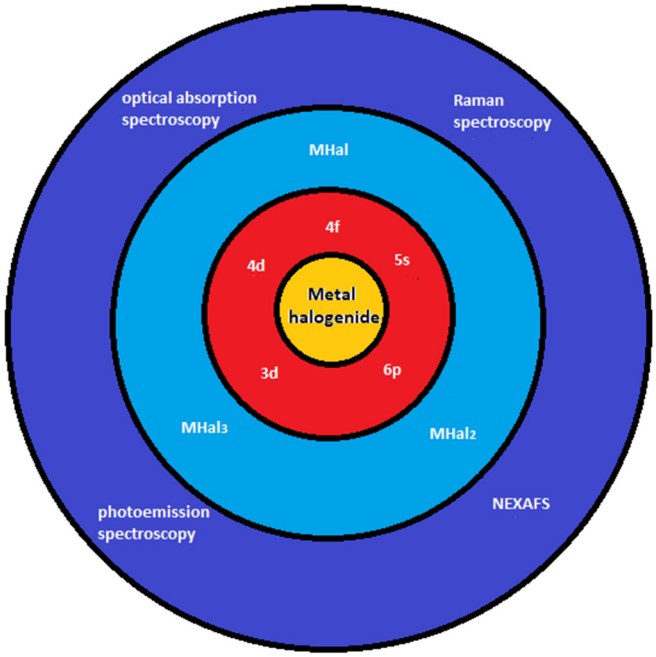

Figure 5. The schematic of metal halogenide-filling and investigation of SWCNTs.

Table 1 summarizes the encapsulated inorganic compounds, diameter and conductivity types of the host SWCNTs, methods of the investigation of the electronic properties, types of doping and the observed Fermi level shifts of SWCNTs.

Table 1. The encapsulated inorganic compounds, diameter and conductivity types of the host SWCNTs, methods of the investigation of the electronic properties, types of doping and the observed Fermi level shifts of SWCNTs.

Encapsulated Metal Halogenide	Diameter and Conductivity Type of SWCNTs [a]	Methods of the Investigation of the Electronic Properties [b]	Type of Doping	Fermi Level Shift, eV	Reference
AgCl	1.4 nm m-SWCNT	RS, XPS, UPS	p	−0.36	[164]
AgCl	1.4 nm s-SWCNT	RS	p	n/a	[163]
$PrCl_3$	1.4 nm m-SWCNT	OAS, RS, XAS, XPS	p	−0.42	[182]
	1.4 nm s-SWCNT			−0.28	
CuCl	1.4 nm m-SWCNT	OAS, RS (+ EC charging), XAS, XPS, WF, VB	p	−0.57	[156]
	1.4 nm s-SWCNT			−0.37	
CuBr	1.4 nm m-SWCNT	OAS, RS (+ EC charging), XAS, XPS, WF, VB	p	−0.60	[156]
	1.4 nm s-SWCNT			−0.36	
CuI	1.4 nm m-SWCNT	OAS, RS (+ EC charging), XAS, XPS, WF, VB	p	−0.35	[156]
	1.4 nm s-SWCNT			−0.25	
$MnCl_2$	1.4 nm mix-SWCNT	RS, XPS	p	−0.43	[175]
$MnBr_2$	1.4 nm mix-SWCNT	RS, XPS	p	−0.37	[175]
$FeCl_2$	1.4 nm mix-SWCNT	OAS, RS, XAS, XPS	p	−0.30	[170]
$FeBr_2$	1.4 nm mix-SWCNT	OAS, RS, XAS, XPS	p	−0.30	[170]
FeI_2	1.4 nm mix-SWCNT	OAS, RS, XAS, XPS	p	−0.30	[170]
$CoBr_2$	1.4 nm mix-SWCNT	OAS, RS, XPS	p	−0.38	[190]
$NiCl_2$	1.4 nm mix-SWCNT	OAS, RS, XAS, XPS	p	−0.58	[167]
$NiBr_2$	1.4 nm mix-SWCNT	OAS, RS, XAS, XPS	p	−0.29	[167]
$ZnCl_2$	1.4 nm mix-SWCNT	OAS, RS, XAS	p	n/a	[169]
$ZnBr_2$	1.4 nm mix-SWCNT	OAS, RS, XAS, XPS, WF, VB	p	−0.28	[169]
ZnI_2	1.4 nm mix-SWCNT	OAS, RS, XAS, XPS	p	−0.25	[169]
RbI	1.4 nm mix-SWCNT	RS, XPS	n	+0.20	[200]
$RbAg_4I_5$	1.4 nm mix-SWCNT	OAS, RS, XAS, XPS	p	−0.35	[207]
AgCl	1.4 nm mix-SWCNT	OAS, RS (+ EC charging), XAS, XPS, UPS	p	−0.38	[155]
AgBr	1.4 nm mix-SWCNT	OAS, RS (+ EC charging), XAS, XPS, UPS	p	−0.37	[155]
AgI	1.4 nm mix-SWCNT	OAS, RS (+ EC charging), XAS, XPS, UPS	p	−0.30	[155]
$CdCl_2$	1.4 nm mix-SWCNT	OAS, RS, XAS, XPS	p	−0.36	[173]
$CdBr_2$	1.4 nm mix-SWCNT	OAS, RS, XAS, XPS	p	−0.36	[173]
CdI_2	1.4 nm mix-SWCNT	OAS, RS, XAS, XPS	p	−0.39	[173]
$PbCl_2$	1.4 nm mix-SWCNT	RS, XPS	p	−0.15	[177]
$PbBr_2$	1.4 nm mix-SWCNT	RS, XPS	p	−0.07	[177]
PbI_2	1.4 nm mix-SWCNTs	RS, XPS	p	−0.17	[177]
$TbCl_3$	1.4 nm mix-SWCNTs	RS	p		[183]
$TbBr_3$	1.4 nm mix-SWCNTs	RS	p		[183]
TbI_3	1.4 nm mix-SWCNTs	RS	p		[183]
$TmCl_3$	1.4 nm mix-SWCNTs	RS	p		[211]
$LuCl_3$	1.4 nm mix-SWCNTs	RS (+ EC charging)	p		[184]
$LuBr_3$	1.4 nm mix-SWCNTs	RS (+ EC charging)	p		[184]
LuI_3	1.4 nm mix-SWCNTs	RS (+ EC charging)	p		[184]
$HgCl_2$	1.7 nm mix-SWCNT	XAS, XPS	p	−0.20	[172]

[a] m-SWCNT = metallic SWCNTs, s-SWCNT = semiconducting SWCNTs, mix-SWCNT = metallicity-mixed SWCNTs; [b] OAS = optical absorption spectroscopy, RS (+ EC charging) = Raman spectroscopy (with electrochemical charging), XAS = X-ray absorption spectroscopy, XPS = X-ray photoelectron spectroscopy, UPS = ultraviolet photoelectron spectroscopy, WF = work function measurements, VB = valence band photoemission measurements.

The electronic properties of SWCNTs filled with manganese halogenides [175], iron halogenides [170], cobalt halogenides [190], nickel halogenides [167], zink halogenides, [169], cadmium halogenides [173], luthetium halogenides [184], mercury halogenies [172], and

lead halogenides [177] were studied by Raman spectroscopy, near-edge X-ray absorption fine structure spectroscopy, photoemission spectroscopy, and optical absorption spectroscopy and p-doping with the Fermi level shift of −0.05–0.5 eV was revealed.

OAS was applied to investigate the direction of the charge transfer in filled SWCNTs in Refs. [155,156,167,169,170,173,182,190,207]. The OAS spectra demonstrated the suppression of the peaks of Van Hove singularities. This was attributed to the doping of SWCNTs with a lowering or increasing of the Fermi level of nanotubes. However, the direction of the charge transfer cannot be deduced from the OAS data (Figure 6) [212].

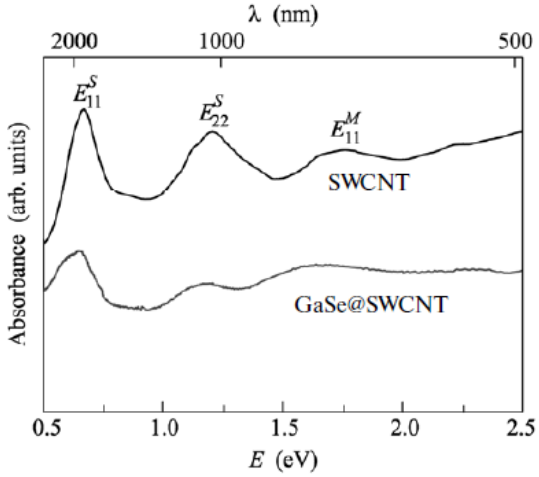

Figure 6. The OAS spectra of the pristine, and GaSe filled SWCNTs. The peaks are denoted. Reprinted from Kharlamova M.V. Novel approach to tailoring the electronic properties of single-walled carbon nanotubes by the encapsulation of high-melting gallium selenide using a single-step process. JETP Letters. V.98. N.5. P.272–277, 2013, Springer Nature [212].

Raman spectroscopy was also used to study the electronic properties of filled SWCNTs in Refs. [155,156,163,164,167,169,170,172,173,175,177,182–184,190,200,207,211]. Raman spectra revealed the changes in the radial breathing mode and G-mode, such as shifts in the peaks and modification of the band profile. This testifies to the doping of SWCNTs by encapsulated compounds (Figure 7) [177].

XPS was employed to evaluate the direction and value of the Fermi level shift of the filled SWCNTs in Refs. [155,156,164,167,169,170,172,173,175,177,182,190,200,207]. The C 1s XPS spectra showed the shift in the peak and change in its width. The shift is caused by the charge transfer between SWCNTs and introduced compounds due to the Fermi level variation. The up- and downshift of SWCNTs corresponds to n- and p-doping of nanotubes by the encapsulated substances, respectively (Figure 8) [175]. UPS was used a direct method of investigation of the Fermi level shift in filled SWCNTs in Refs. [155,164,169]. The UPS spectra showed the shifts in the peaks of Van Hove singularities. This is evidence of the Fermi level shift in filled SWCNTs. The down- and upshift of the peaks corresponds to the lowering and increasing of the Fermi level of nanotubes.

NEXAFS was applied to analyze local interactions between encapsulated substances and SWCNTs in Refs. [155,156,167,169,170,172,173,182,207]. The modifications in the spectra may testify to the formation of chemical bonds between the nanotube walls and introduced substances (Figure 9) [212].

Authors of Refs. [161,162] filled the separated metallic and semiconducting SWCNTs with copper chloride. Using optical absorption spectroscopy and Raman spectroscopy, they showed that the encapsulated salt leads to p-doping of nanotubes. The efficiency of doping for the metallic-enriched SWCNT fraction was higher than that for the semiconducting SWCNTs. The functionalization of both semiconducting and metallic SWCNT films by filling nanotubes with CuCl led to a significant increase in optical transmittance (above

90% in the near-infrared field). The best absolute optical transmittance was observed for filled metallic SWCNT films.

In Ref. [163], high-purity semiconducting SWCNTs were filled with silver (I) chloride, and the multifrequency Raman spectra of filled and unfilled semiconducting SWCNTs were compared. Raman spectroscopy at five laser wavelengths allowed the investigating in detail of the electronic properties of the filled SWCNTs. Using different laser wavelengths allowed exciting electronic transitions of different semiconducting nanotubes and the study of the influence of the incorporated silver (I) chloride on their electronic properties. The RBM and G-bands of the Raman spectra of the pristine and filled SWCNTs were fitted with individual components. The peak positions and relative intensities of individual components of the RBM and G-bands of the pristine and filled SWCNTs were compared. Changes in peak intensities and appearance of new peaks in the RBM-band of the filled SWCNTs as compared to the pristine SWCNTs were revealed (Figure 10a). This testified to the filling-induced alteration in the resonance excitation conditions of nanotubes due to a charge transfer in the filled nanotubes that causes exciting optical transitions in smaller or larger diameter nanotubes using the laser with the same wavelength. Additionally, the prominent shifts in the components of the G-band of the filled SWCNTs as compared to the pristine SWCNTs as well as no changes in the component intensities were revealed (Figure 10a). The shifts in the components of the G-band were found to be independent of laser wavelength. These shifts were attributed to p-doping of SWCNTs by the inserted silver (I) chloride accompanied by the charge transfer from nanotubes to the salt.

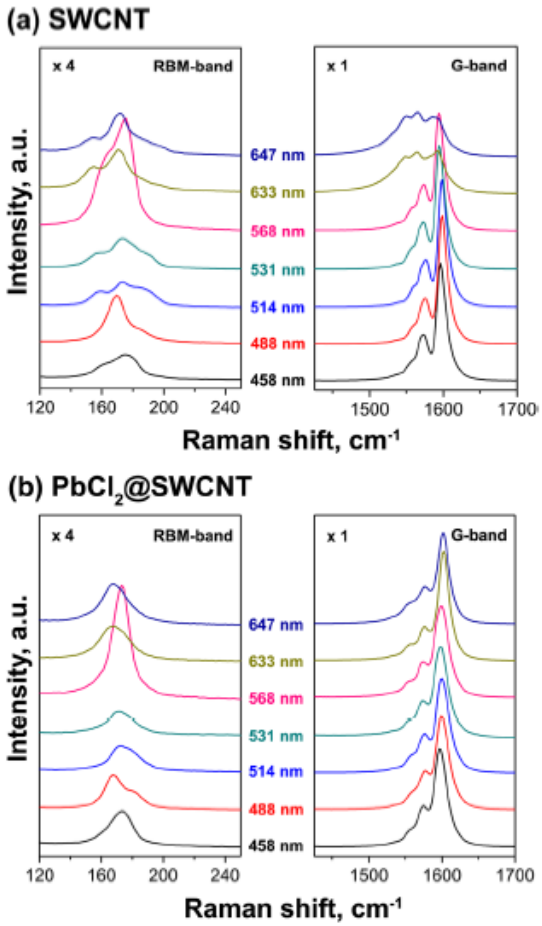

Figure 7. The Raman spectra of the pristine SWCNTs, and $PbCl_2$—filled SWCNTs acquired at different laser excitation wavelengths. Reprinted from Kharlamova M.V., Kramberger C., Rudatis P., Pichler T., Eder D. Revealing the doping effect of encapsulated lead halogenides on single-walled carbon nanotubes. Appl. Phys. A. 2019. V.125. N.5. article number 320, Springer Nature [177].

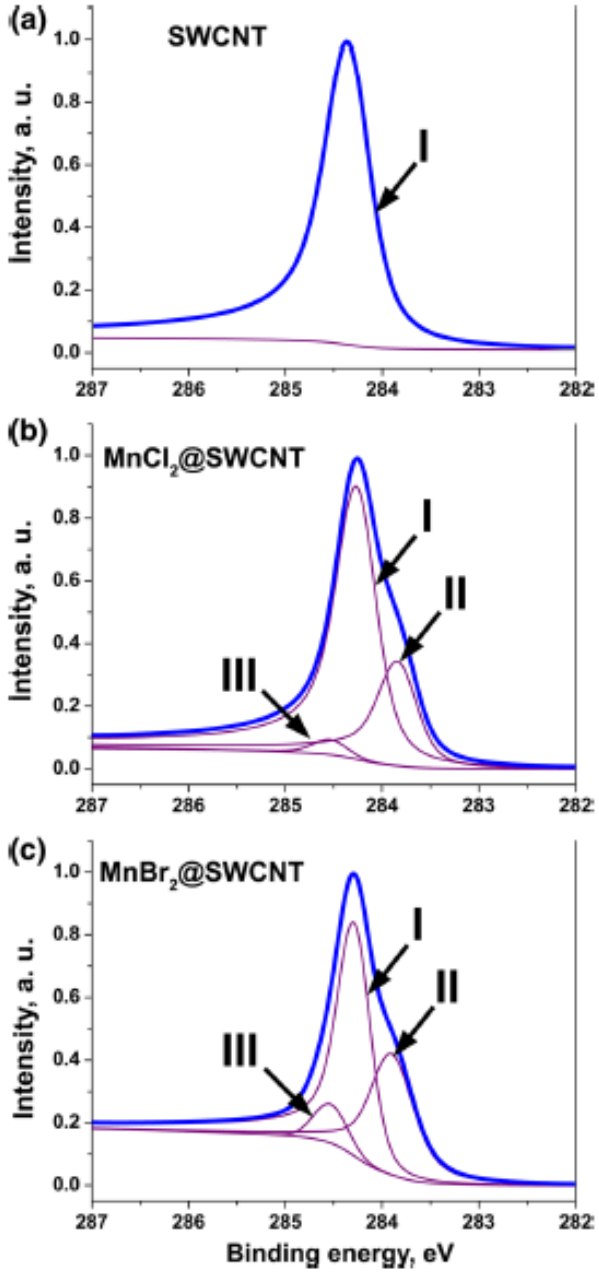

Figure 8. The C 1s XPS spectra of the pristine, and $MnCl_2$, $MnBr_2$ filled SWCNTs. The inset shows the shift in the peak. Reprinted from Kharlamova M.V. Electronic properties of single-walled carbon nanotubes filled with manganese halogenides. Appl. Phys. A. 2016. V.122. N.9. article number 791, Springer Nature [175].

Authors of Ref. [164] filled high-purity metallic SWCNTs with silver (I) chloride. The filled and unfilled SWCNTs were investigated by Raman spectroscopy, XPS and UPS, which were combined to study comprehensively the modified electronic properties of the filled SWCNTs. XPS confirmed the chemical composition of the encapsulated compound. A detailed analysis of Raman spectra of the pristine and filled SWCNTs allowed the investigation of filling-induced changes in the electronic properties of SWCNTs. The RBM and G-bands of the Raman spectra were fitted with individual components. The analysis of the RBM-band testified to the disappearance of the peak in the largest-diameter filled metallic SWCNTs, which was attributed to the changes in their resonance conditions. The analysis of the G-band revealed upshifts in the components of the filled SWCNTs and the

changed profile of the band. This indicated doping of SWCNTs by the encapsulated silver (I) chloride and gap opening in the band structure of the filled SWCNTs, resulting in a transition of metallic nanotubes into semiconducting SWCNTs. The direction of the charge transfer in the filled SWCNTs was determined by XPS. The observed downshift of the C 1s XPS peak of the filled SWCNTs of 0.36 eV as well as its broadening were attributed to p-doping of SWCNTs accompanied by the charge transfer from the nanotubes to the inserted silver (I) chloride (Figure 10b). The detailed information on the doping-induced shift of the Fermi level of SWCNTs upon their filling was obtained by UPS. The UPS spectra of the filled SWCNTs demonstrated the downshift of the π-resonance by 0.36 eV, which was a direct confirmation of the downshift of the Fermi level of nanotubes (Figure 10c).

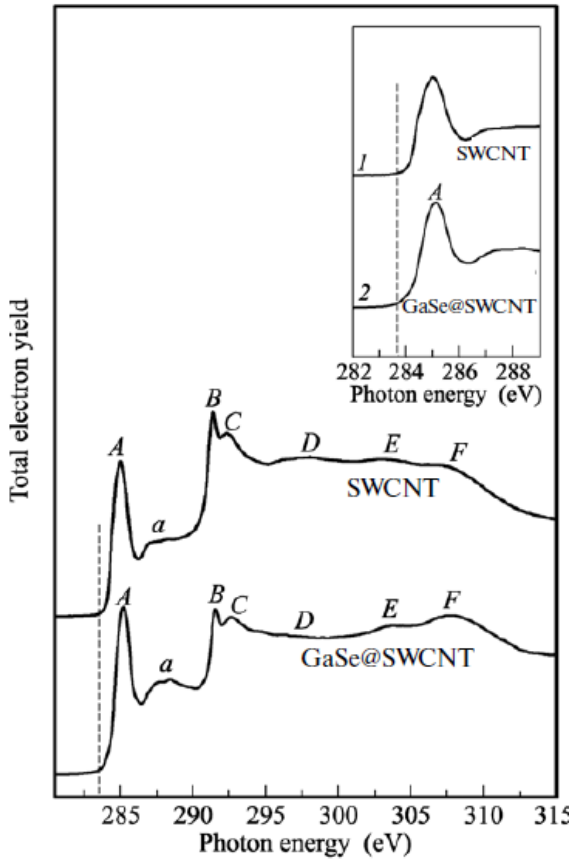

Figure 9. The NEXAFS spectra of the pristine and GaSe filled SWCNTs. Features of the spectra are denoted. The inset shows the π*-peak. Reprinted from Kharlamova M.V. Novel approach to tailoring the electronic properties of single-walled carbon nanotubes by the encapsulation of high-melting gallium selenide using a single-step process. JETP Letters. V.98. N.5. P.272–277, 2013, Springer Nature [212].

The above-mentioned reports demonstrate the effect of the encapsulated materials on the electronic properties on either purely metallic or semiconducting SWCNTs. They also showcase the potential of precise Fermi level engineering of SWCNTs by filling their channels and achieving high doping levels. This control over the electronic properties will be the key to design the next generation of SWCNT-based nanoelectronic devices.

Different encapsulated inorganic compounds have a different influence on the electronic properties of host SWCNTs. Several authors investigated the correlation between the chemical nature of the incorporated inorganic compound and their doping effect on nanotubes.

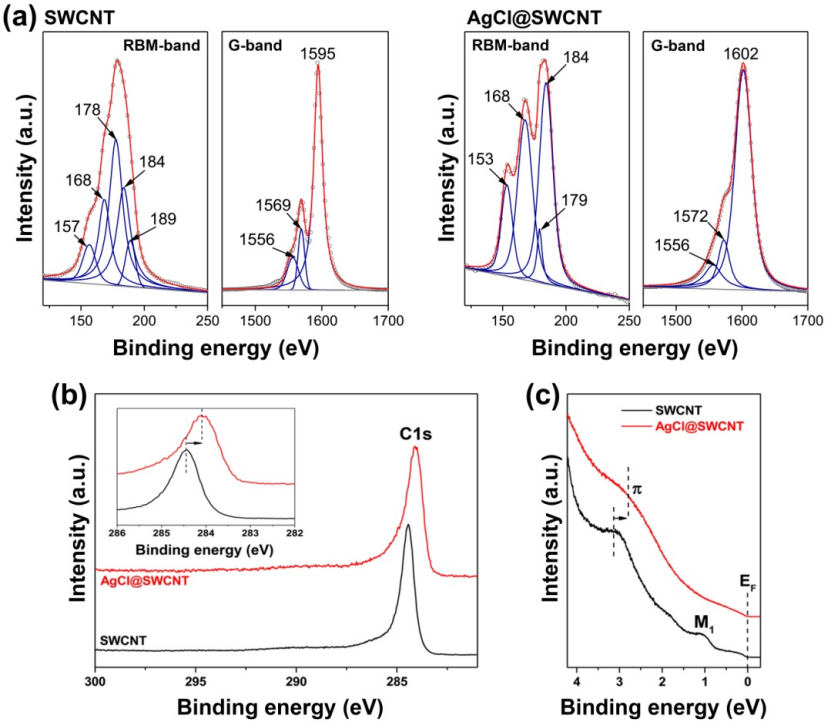

Figure 10. (**a**) The RBM and G-bands of Raman spectra of the pristine and silver (I) chloride-filled semiconducting SWCNTs acquired at a laser wavelength of 514 nm fitted with individual components. The RBM-band is fitted with the components belonging to the nanotubes of different diameters. The G-band is fitted with G^-_{LO}, G^+_{TO} and G^+_{LO} components [163]. Copyright 2018 M. V. Kharlamova et al. This is an open access article distributed under the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited. (**b**) The C 1s XPS spectra of the pristine and silver (I) chloride-filled metallic SWCNTs. The inset zooms in the shift in the C 1s peak, as shown by the arrow. The dashed vertical lines denote the peak positions. Reproduced from Kharlamova M.V. et al. Fermi level engineering of metallicity-sorted metallic single-walled carbon nanotubes by encapsulation of few-atom-thick crystals of silver (I) chloride. Journal of Materials Science. V.53. N.18. P.13,018–13,029. 2018, Springer Nature [164]. (**c**) The valence band spectra of the pristine and silver (I) chloride-filled metallic SWCNTs. The dashed vertical lines denote the positions of the π-peak. The shift in the π-peak is indicated by the arrow. The peak labeled M_1 corresponds to the first Van Hove singularity of the pristine metallic SWCNTs. The Fermi level (E_F) is indicated. Reproduced from Kharlamova M.V. et al. Fermi level engineering of metallicity-sorted metallic single-walled carbon nanotubes by encapsulation of few-atom-thick crystals of silver (I) chloride. Journal of Materials Science. V.53. N.18. P.13,018–13,029. 2018, Springer Nature [164].

In Ref. [191], a systematic comparison of the doping effect of encapsulated nickel (II) bromide, cobalt (II) bromide and iron (II) bromide on SWCNTs was conducted using Raman spectroscopy. The authors compared the relative intensities of and shifts in the peaks of the RBM and G-bands of the Raman spectra of the filled nanotubes (Figure 11). The RBM-band was fitted with two components corresponding to nanotubes of different diameters. In the spectrum of the pristine SWCNTs, the first component corresponding to the larger-diameter SWCNTs had a larger intensity. The relative intensity of two RBM peaks was changed upon the filling of nanotubes. In the spectra of the filled SWCNTs, the second component corresponding to the smaller-diameter SWCNTs had the larger intensity. The largest changes as compared to the pristine SWCNTs were observed for the SWCNTs filled with iron (II) bromide (Figure 11a). On the basis of these data, it was concluded that iron (II) bromide has a larger doping effect on SWCNTs than either cobalt (II) bromide or nickel (II) bromide.

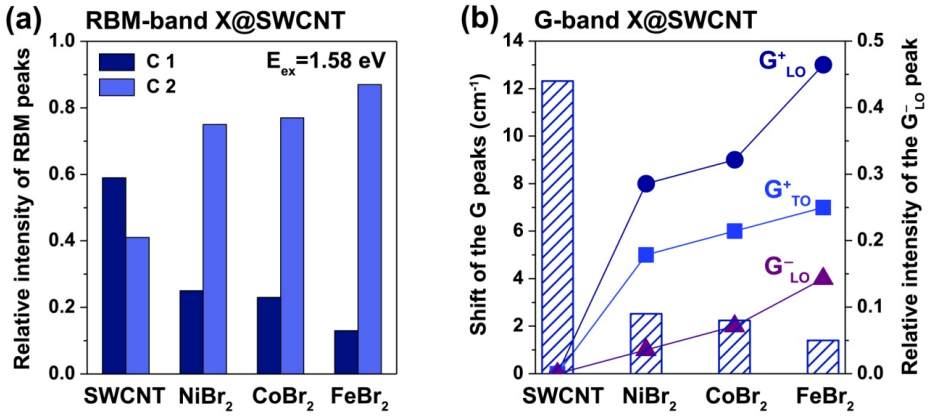

Figure 11. The results of the analysis of Raman spectra of the pristine SWCNTs and nanotubes filled with iron (II) bromide, cobalt (II) bromide and nickel (II) bromide. The comparison of the relative intensity in the RBM peaks (**a**), shifts of the G-band peaks and relative intensity of the G^-_{LO} peak (**b**) [191]. Copyright 2015 Marianna V. Kharlamova. This is an open access article distributed under the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

The results of the line shape analysis of the G-band of the Raman spectra of the filled SWCNTs were in agreement with the results of the fitting of the RBM-band. Figure 11b compares the shifts in the components of the G-bands and the relative intensities of the Breit−Wigner−Fano component (G^-_{LO}) for the pristine SWCNTs and filled nanotubes. It can be seen that the upshifts of all components of the G-band increase in line with nickel (II) bromide-cobalt (II) bromide-iron (II) bromide. In addition, the relative intensity of the metallic G^-_{LO} component decreases significantly in the spectra of the filled SWCNTs as compared to the pristine nanotubes. The largest difference is again observed for the SWCNTs filled with iron (II) bromide. Taking into consideration these data, authors concluded that the iron (II) bromide has the largest p-doping effect on SWCNTs, whereas nickel (II) bromide has the smallest effect. This is explained by the different ionization potentials of iron, cobalt, and nickel. It is the minimal for iron, and it is maximal for nickel. Here the anion is the same, but the size of the anion and electron affinity of halogen plays an important role.

In Ref. [183], a systematic comparison of the doping effect of the encapsulated terbium (III) chloride, terbium (III) bromide and terbium (III) iodide on SWCNTs was conducted using Raman spectroscopy. The fitting of the RBM and G-bands of the Raman spectra of the filled SWCNTs and comparison of the relative intensities of and shifts in the peaks showed that the p-doping effect on SWCNTs increases in line with terbium (III) iodide–terbium (III) bromide–terbium (III) chloride.

Combining the OAS, Raman spectroscopy and photoemission data allowed the authors of Refs. [155,156,177] to investigate in detail the influence of the anion type of the encapsulated metal halogenides on the doping level of SWCNTs. For halogenides of silver and copper, it was revealed that the p-doping level of SWCNTs increases in line with metal iodide–metal bromide–metal chloride [155,156]. For halogenides of lead, the strongest p-doping effect on SWCNTs was observed for lead (II) iodide, whereas the weakest p-doping effect was revealed for lead (II) bromide [177].

In Ref. [208], a comparison of the doping effect of the encapsulated tin, gallium and bismuth chalcogenides was performed using the data of OAS, Raman spectroscopy and XPS. It was shown that the encapsulation of tin sulfide inside the SWCNTs does not cause significant changes in the electronic structure of semiconducting SWCNTs, whereas there is a slight influence on metallic nanotubes. The introduction of gallium telluride inside the SWCNTs results in a downshift in the Fermi level of SWCNTs, i.e., p-doping of the

nanotubes. The filling of SWCNTs with bismuth selenide does not lead to the modification of the electronic properties of nanotubes.

5. Comparison of Doping Effect of Inorganic Compounds on Different Diameter SWCNTs

In Ref. [165], the authors performed a comparison of the doping levels of silver (I) chloride-filled metallicity-mixed SWCNTs with diameters of 1.4 and 1.9 nm synthesized by the arc-discharge and chemical vapor deposition methods, respectively. The electronic properties of the filled SWCNTs were investigated by Raman spectroscopy and XPS. The analysis of the Raman spectroscopy data acquired at laser wavelengths of 514 and 633 nm revealed the shifts and changes in the relative intensities of the components of the RBM-bands of the filled 1.4 nm-diameter SWCNTs, which are a result of the alteration in resonance excitation conditions of nanotubes upon their filling [165]. The RBM-band of the Raman spectrum of the filled 1.9 nm-diameter SWCNTs was completely suppressed; this was probably caused by the presence of large-diameter silver (I) chloride nanocrystal inside the SWCNT channel that distorted the radial symmetry of nanotubes. The G-band of the Raman spectra of the filled 1.4 and 1.9 nm-diameter SWCNTs showed significant upshifts, which were attributed to p-doping of SWCNTs by the encapsulated silver (I) chloride. In addition, the data indicated different filling ratios of 1.4 and 1.9 nm-diameter SWCNTs [165]. The XPS data confirmed that the filling of SWCNTs with silver (I) chloride led to the p-doping of nanotubes and downshift of their Fermi level. The C 1s XPS peaks of the filled 1.4 and 1.9 nm-diameter SWCNTs showed downshift values of 0.40 and 0.23 eV as compared to the pristine nanotubes, which may testify to the different doping levels of SWCNTs. Larger doping levels of the arc-discharge 1.4 nm-diameter SWCNTs as compared to the CVD 1.9 nm-diameter nanotubes were explained by the different filling ratios of SWCNTs, which are probably caused by differences in synthetic protocols of the pristine SWCNTs [165].

6. Discussion of the Influence of Different Encapsulated Substances on the Electronic Properties of SWCNTs

The analysis of the literature shows that metal halogenides inside SWCNTs (Figure 12a), whose work function (WF) is larger (Figure 12b) or smaller (Figure 12c) than the value of SWCNTs lead to the downshift or upshift in the Fermi level of SWCNTs, accordingly. These metal halogenides are MHal (M=Ag, Cu, Hal=Cl, Br, I), $MHal_2$ (M=Fe, Co, Ni, Mn, Zn, Cd, Pb, Hg, Hal = Cl, Br, I), $MHal_3$ (M=Pr, Tb, Tm, Lu, Hal=Cl, Br, I) [154–207]. Metal chalcogenides, whose work function is larger (Figure 12d) or equal (Figure 12e) to the value of SWCNTs lead to the downshift in the Fermi level of SWCNTs, or no changes in the electronic structure of nanotubes, accordingly. These metal chalcogenides are MX (M=Ga, Sn, X=S, Se, Te), $M_{2\times3}$ (M=Bi, X=Se, Te) [208–214]. Elementary substances–metals (Figure 12f) and non-metals (Figure 12g), whose work functions are smaller or larger than the values of SWCNTs, or molecules (Figure 12h) result in the upshift or downshift of the Fermi level of SWCNTs, accordingly. These substances are Ag, Cu, Eu, P, As, I [45–153]. The discussion is as follows:

- Regarding kinetics of growth of carbon nanotubes, the growth mechanism of SWCNTs was revealed. The application of Raman spectroscopy allowed us to calculate the growth rates and activation energies. They are in the range of 0.5 to 3.0 eV. No activation energies for growth of individual carbon nanotubes were reported thus far.
- The authors showed the metal-dependence of growth kinetics, and that the use of different metal catalyst precursors allowed analyzing the influence of metal on the growth mechanism of SWCNTs. It was shown that different metals lead to different growth mechanisms of carbon nanotubes. More TEM investigations are required to analyze the mechanism in detail.
- Regarding the filling of metallicity-sorted SWCNTs, separated metallic, semiconducting SWCNT samples were proved to be a viable tool to unravel the effects of the filler on the electronic properties of the compound material. The investigation of

the macroscopic electronic properties by a variety of spectroscopic methods (OAS, RS, XPS, UPS and XAS) allowed us to thoroughly address the influences on the electronic properties that are caused by encapsulated elementary substances, inorganic compounds and molecules.

- The authors developed new approaches to processing the data of spectroscopic investigations of filled SWCNTs. There have been developments in the evaluation of spectroscopic data. The precise and reliable data analysis supported drawing clear conclusions on the quantitative charge transfer present in differently filled SWCNTs.
- Regarding the correlation between the physical and chemical properties of encapsulated substances and the electronic properties of SWCNTs, the combined spectroscopic studies on filled SWCNTs allowed us to determine the charge transfer quantitatively. It can be evaluated as elementary charges per SWCNT surface atom, or additionally as elementary charges per SWCNT length. With the filling ratio and the interconversion of encapsulated substances, the doping level can be varied in a wide range. It is even possible to tune the doping level across the charge neutrality point and switch from n to p type doping. This powerful control of the electronic properties in heterogeneously filled SWCNTs enabled the demonstration of many different applications. The applications covered in this review include nanoelectronics, thermoelectric power generation, electrochemical energy storage, catalysis, sensors, spintronics, magnetic recording and biomedicine. This review also addresses current issues and likely contenders for a breakthrough in the near future, namely photovoltaics and light emission.

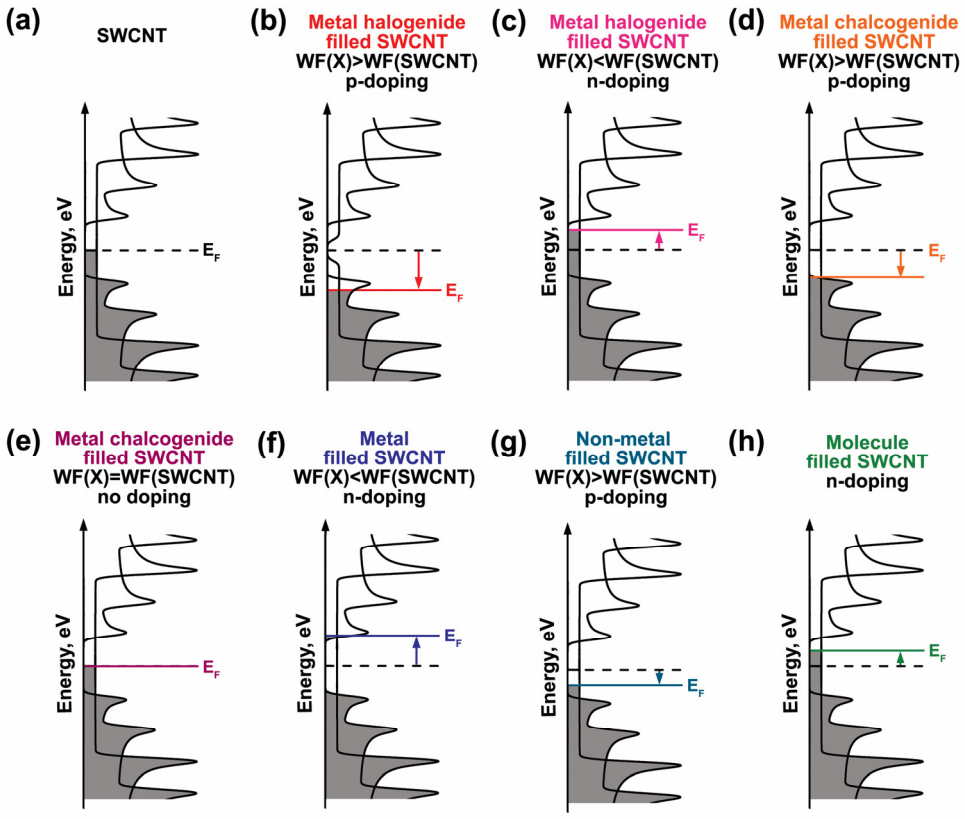

Figure 12. The schematics of the Fermi level shift in SWCNTs (**a**) filled with inorganic compounds–metal halogenides, whose work function (WF) is larger (**b**) or smaller (**c**) than the value of SWCNTs, and metal chalcogenides, whose work function is larger than (**d**) or equal (**e**) to the value of SWCNTs; elementary substances–metals (**f**) and non-metals (**g**), whose work functions are smaller or larger than the values of SWCNTs, respectively; and molecules (**h**).

7. Conclusions

In this paper, we reviewed the kinetics of growth and the electronic properties of single-walled carbon nanotubes for applications. The use of methods of investigations allowed us to analyze in detail the properties of filled SWCNTs. New techniques of data processing were developed that allowed us to conclude about growth mechanisms and charge transfer mechanisms for filled SWCNTs toward applications. New samples were obtained, and their analysis confirmed the reported trends. Further progress is expected in controlled electronic properties of filled SWCNTs, which will, in conjunction with the wide range of demonstrated and envisaged applications, stimulate further advancements toward viable technologies built on them.

Author Contributions: The manuscript is written solely by M.V.K. who contributed as leading author to every reviewed paper. C.K. edited the manuscript. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Data are available on request to the first author (M.V.K.) of every reviewed paper.

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