

Thesis Changes Log

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PhD Program: Materials Science and Engineering

Title of Thesis: Assembling Networks OF Single-Walled Carbon Nanotubes for Electronic and Optical Applications

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Co-supervisors: Professor Tanja Kallio, Assistant Professor Dmitry Krasnikov, Assistant Professor Fedor Fedorov

The thesis document includes the following changes in answer to the external review process.

I am very grateful to the Jury Members for their time for reviewing my doctoral thesis work as well as for their general positive feedback and valuable comments and suggestions for thesis improvement. Below, responses to the remarks of each Jury Member are represented.

Response to Prof. Dmitry Gorin

1. Page 39, Figure 3, I recommend to add an additional figure 3b from an article, where such type of dependences has been presented and please add the references to figure caption.

I thank Prof. Gorin for this suggestion. **Figure 3** was complemented with an additional image of a theoretical representation of a percolation curve adapted from [1].

2. Page 62, Figure 8, Usually, we have one RBM peak. In Figure 8 we see some peaks. Could you explain this peculiarity?

Indeed, every SWCNT with a specific chirality has its own characteristic frequency of a RBM peak interconnected to its diameter [2]. However, often an ensemble of SWCNTs with a broad distribution of chiralities and diameters, such as a SWCNT film or a fiber, is measured, which result in a series of RBM peaks (for instance, such a typical Raman spectrum is presented in [3]). As the Thesis devoted to the random networks of SWCNTs, such a spectrum was shown in **Figure 8**. This comment was added to the Thesis text.

3. Page 82, Figure 13, b,e and Page 84, Figure 14, e. These experimental dependences have a maximum. What do you think about applicability of experimental design techniques to this type of study?

I thank Prof. Gorin for this interesting question. Indeed, pronounced maxima are observed for nanotube yield, conductivity, and length when H₂ is added to CO atmosphere. The current task was to find an optimum hydrogen concentration for each of this parameter. Meanwhile, all these parameters are measured simultaneously at a single synthesis experiment (that is, there is no need in carrying out a new experiment for a new parameter measurement).

Since the study is devoted rather to the investigation of general effect of H₂ as a growth promoter, the main goal was to find an optimum range in the one-parameter task, which is quite simple from this viewpoint. Thus, there was no need in performing a lot of experiments, when additional optimization would be useful.

Nevertheless, as the next step, when several input parameters could be considered (for instance, temperature and H₂ concentration – to find optimal conditions for the low-temperature regime), the use of experimental design techniques, such as Box-Wilson method, could significantly speed up the search for optimal points. Typically, in our research groups, for such tasks different machine learning algorithms, such as artificial neural networks and support vector regression were applied [4,5].

4. Page 111, table 5, I would like to ask about a physical explanation of the small magnitude of percolation thresholds for such type of nanocomposite. Did you compare your data with the early published ones for the similar SWNT containing composites?

I thank Prof. Gorin for raising this question. Indeed, obtained values for both SWCNT/TPU and SWCNT/epoxy nanocomposites appeared to be very low. It is more than one order of magnitude lower than the predicted value of ~ 0.035 wt% from the classical percolation theory.

First, I would like to note that the results were obtained using impedance response data, namely, the DC values (at the lowest frequency) and the characteristic features of the frequency dependencies of the impedance response (presence or absence of a frequency-independent region indicating ohmic behavior of conductivity achieved by formed percolative network of nanotubes). Besides, for higher-conductivity SWCNT/epoxy nanocomposites, the results were confirmed by direct measurements of sample resistance. Thus, the obtained results are confirmed by several methods and by reproductivity of the measured data among different samples.

The ultralow values of percolation threshold might be explained by the balance between high dispersion degree and, at the same time, moderate agglomeration between nanotubes [1]. It is also often referred to as a kinetic percolation threshold due to filler movement and re-aggregation [6,7]. The origin of this phenomenon could be in the formation of small-scale chain-shaped aggregates which allows formation of a percolative conductive network at concentrations noticeably lower than it would be reached at the uniform distribution. This behavior was repeatedly observed in experimental and simulation studies [1, 6-9]. Besides, it is worth noting, that the classical percolation theory is quite limited model where such parameters, as a filler curvature and an agglomeration degree are out of consideration. The corresponding discussions were added to the Thesis for a better representation of the achieved results.

Regarding the second question, indeed, it would be great to compare the results achieved here with the published data for similar nanocomposite systems since it would demonstrate relevancy and novelty of the approach. New **Table 5** was added where percolation values of CNT/TPU nanocomposites as well as CNT type, aspect ratio, and fabrication techniques are shown. Values of percolation threshold achieved in this work turned out to be at least one order of magnitude lower, which verifies the method developed in the current study.

5. Page 114-115, I recommend to add the reference on Ilya's articles to every statement that is presented in Chapter 6.

I followed these recommendations from Prof. Gorin added the references to the conclusions presented in Chapter 6.

Response to Prof. Alexander Korsunsky

1. Overall, the thesis is written well. In the beginning the introductory part is devoted to the overview of work in the field carried out to date, as well as summary of the key relationships that are further

employed in the analysis. This is done in a fairly terse and sketchy manner that I feel is sufficient for readers closely acquainted with the scope of problems being addressed but could be made better grounded in the physics of CNT's for broader reader base.

I thank for evaluation of my Thesis work. I agree that some conclusions and regularities regarding CNT physics could be given in quite a brief manner. Nevertheless, I needed to cover all topic related to conductivity of nanotubes and their ensembles together with a brief review of experimental techniques for both nanotube synthesis and nanocomposite fabrication (in addition to the description of their conductivity features) in the introduction part, so it took more than 25 pages. Therefore, too deep consideration of the physical aspects of nanotubes could distract a reader from the main focus of the work devoted rather to chemical engineering problems.

Nevertheless, some additional explanations and grounds for especially complex aspects in the texts were added (primarily, those based on the further questions from Prof. Korsunsky and other Jury Members) as well as references to the papers with detailed explanations of specific phenomena.

2. The discussion of matters of fabrication and characterisation is presented well and reflects the substantial amount of work carried out by the candidate. Furthermore, the reader gets the impression that the work was mostly or exclusively done by the author, although some doubts arise when the phrases appear such as "we carried out investigation" etc. In this relation, the candidate is advised that in a Thesis it is best to avoid using "we", because the candidate's personal contribution is being assessed.

I thank Prof. Korsunsky for this remark, statements with "we" were reformulated to avoid such an impression.

3. Generally, the work reported suggests that the project has been successful. However, I found it difficult to ascertain how novel the author's achievement has been. Since novelty of results is a principal measure of success for assessment, the author is advised to seek to make it abundantly clear exactly which results are being offered for degree defense as novel.

I thank Prof. Korsunsky for indicating this drawback in Thesis. The novelty of the results was emphasized in the conclusions of each part of the work.

4. The physical meaning and units of all constants and parameters that appearing in Eq.(7) should be explained.

Explanations and units of the parameters and constants describing tunnelling conductivity between CNTs in eq. (7) were added to the text.

5. There appears to be no explanation / justification given of the statistical aspect of SWCNT population distribution: why certain proportion is m, s, and hence why the proportion of junctions m-m, m-s and s-s, etc.

I thank Prof. Korsunsky for pointing to this shortcoming in the Thesis. In the case of random distribution of SWCNT chiralities, it is believed that $\sim 1/3$ of them possess metallic conductivity (m), and $\sim 2/3$ are semiconductors (s). This indeed results in the specific proportion of junctions: $\sim 1/9$ m-m contacts, $\sim 4/9$ m-s contacts, and $\sim 4/9$ s-s contacts.

These proportions for random chirality distribution of SWCNTs are come from the selection rules for determining nanotube conductivity type. Cylindrical shape of nanotubes imposes periodic boundary conditions for circumferentially directed wavevector (perpendicularly to the nanotube axis). This results in the fact the nanotube bandgap depends on its chirality, namely, on the divisibility of the chirality coordinate difference $n - m$ by 3: if $(n - m) \div 3$, bandgap is zero, the dispersion relation is analogous to graphene and SWCNT is nominally metallic; otherwise, (if $n - m$ is not multiple of 3), SWCNT is semiconductive with a bandgap of a few hundreds of meV. Thus, at random distribution,

that is, any possible pair of chirality coordinates n and m , one third of nanotubes will be metallic, and two third – semiconductors. Similar statistics are achieved for a typical broad chirality distribution.

Meanwhile, even at the narrow nanotube diameter distributions, there are plenty chirality possibilities, so that this m/s ratio would be preserved. At the same time, in the synthesis and postprocessing methods aimed to obtain a narrow chirality distribution, m/s proportions of nanotubes could be significantly altered. Nevertheless, consideration of such systems was beyond the scope of this work.

These discussions were added to Thesis.

Response to Prof. Ayrat Dimiev

1. Page 20, Fig. 1. In Figure caption, the copyright is shown incorrectly. It is 2015, not 2023.

The figure caption was corrected.

2. Page 48, line 1: Two Figure numbers (1 and 4) are given; old Figure # is not removed.

I thank for this remark, this inaccuracy was fixed.

3. Page 60; the role of the excitation laser frequency is missing in describing the G-band shape. Either list all the factors in details (preferred), or none of them.

I thank Prof. Dimiev for raising this question. Indeed, G-band shape is determined not only by features of carbon nanotubes but excitation laser frequency too. Frequency-dependent response of G-band shape is due to the fact that laser excitation wavelength causes a resonance scattering of nanotubes with specific chiralities (similarly to RBM peaks' position effect). Meanwhile, G' shape fits by a Lorentzian function for semiconducting SWCNTs and a BWF function (with a specific asymmetrical shape) for metallic one. Thus, since different excitation frequencies result in different nanotube ensembles appeared to be resonance (that is, metallic/semiconducting ratio) and, in turn, in different contribution of Lorentzian/BWF functions in G'-band shape.

The corresponding discussions were added to the text.

4. Page 67. Here, and at several occasions throughout the text, the English grammar needs to be improved.

I thank for pointing to this drawback. The text grammar was carefully revised and corrected.

5. Page 101. The conclusions, formulated here (kinetic nature of percolation threshold, etc.) inspired by ref [92] are not typical for the field. Flocculation between nanotubes cannot lead to "formation of a nanotube network within the whole host materials" since this leads to filler-free spaces between the flocculates; the situation is opposite. This needs to be either further detailed, or removed/reformulated.

I thank Prof. Dimiev for raising this essential question. Indeed, flocculation between particles, which would lead to their better dispersion, is quite controversial. Nevertheless, I would like to mention that a moderate degree of aggregation was shown to lead to a lower values of percolation threshold by both experimental [8] and simulation [9] studies. The reason of such counterintuitive behavior may be explained by the formation of small-scale chain-like agglomerates, which would allow formation of percolative conductive pathways at the lower nanotube concentration compared to the case of evenly distributed particles. However, such aggregates were not shown directly in the current work, thus, in this work, it is considered as a main hypothesis behind the ultralow values of percolation threshold, which appeared to be significantly lower than that predicted by the classical percolation theory.

Nevertheless, I agree with the remark and conclusions were reformulated for a more clear representation.

Response to Prof. Yutian Zhu

1. There are still some grammatical or format errors in the thesis. For example, in page 3 (Abstract), the sentence “Reactor chemical engineering approaches were developed: controllable adjustment of residence time (τ , by flow rate control) allows maintaining catalyst activation stage and varying nanotube growth, this way optimizing R_{90} (51 Ω/\square was achieved for doped films) (i), and the introduction of H₂ as a reducing agent, which was demonstrated to increase synthesis yield by 15 times (ii).”, the forms of the tenses in this sentence is not consistent. In addition, “ Ω/\square ” is not correct.

In page 99, the sentence of “Besides, magnified SEM images (Figure 18c1-d1) illustrate the gradual thickening of bundles approaching the TPU matrix, which might indicate high TPU wetting of nanotubes, which is attributed to the high affinity of TPU to nanotubes and is in a good agreement with the reported data [167,168].” is not correct from its structure.

In page 107-108, “powders (Table 1)” is dissected into two pages.

In page 108-109, “ magnitude (Table 1)” is dissected into two pages.

In page 106, the title “5.2. SWCNT/thermoset nanocomposites. Nanotube bulk density optimization.” is not suitable. It seems there are two sentences for one title.

I thank Prof. Zhu for the careful revision of Thesis text. All the inaccuracies were fixed.

2. Did the author make modification of the commercial SWCNTs for the section of SWCNTs/polymer composites? It seems there are good compatibility between SWCNTs and polymer matrix. Why?

Commercial SWCNTs used for fabrication of SWCNT/TPU nanocomposites (the study devoted to elastic nanocomposites, **Section 5.2**) were used without any modifications. Meanwhile, for investigation of SWCNT powder bulk density (consolidation degree) effect in SWCNT/thermoset (epoxy) nanocomposites, SWCNT powders were subjected to (1) pneumatic compression (“briquettes”) and (2) rapid expansion in the supercritical fluid (“RESS”). Also, masterbatches (MB) were used to compare SWCNT powders with different bulk densities. It is described in detail in **Section 3.1.2** (summarized in **Table 1**). However, neither functionalization of nanotubes, nor addition of binders, surfactants, stabilizers, *etc.* was applied.

Good compatibility between SWCNTs and TPU can be explained by high affinity of TPU to nanotube walls resulting in high wetting of the polymer. It is supported by SEM images (where some thickening of nanotubes/bundles can be observed, **Figure 18** and new **Figure A9**) and relatively low conductivity of nanocomposites compared to SWCNT/epoxy ones (**Figures 19** and **24**). Besides, it agrees with the literature data [10,11].

3. For SWCNTs/polymer composites, there show ultralow percolation thresholds based on the curves of Dc conductivity plotted versus SWCNT concentration. What is the volume fraction of SWCNTs in polymer matrix? Since here the composites are prepared by the coprecipitation of SWCNTs and TPU in water. SWCNTs should be randomly distributed in TPU matrix. Therefore, SWCNTs can only form a random conductive network in TPU. It is strange that SWCNTs can form the percolated network at a concentration of 0.006%.

I thank Prof. Zhu for raising this important question. Volume fraction of SWCNTs is interrelated with weight fraction according the ratio $wt\% = vol\% \cdot \frac{\rho_{SWCNT}}{\rho_{TPU}}$, where ρ_{SWCNT} can be estimated as 1.9 g/cm³ and ρ_{TPU} is 1.16 g/cm³ according manufacturer’s data. Thus, percolation threshold of 0.006 wt% is equivalent to volume fraction of 0.0037 vol%.

It is also worth noting that such low percolation threshold values were found for both SWCNT/TPU and SWCNT/epoxy nanocomposites (the latter ones are even lower) by using the impedance response data. Meanwhile, both DC values (impedance response at the lower frequency) and characteristic features of the frequency dependencies of conductivity (presence/absence of frequency-independent region indicating ohmic response of percolative nanotube network) were taken into account. Besides, for higher-conductivity SWCNT/epoxy nanocomposites, the results were confirmed by direct measurements of sample resistance. Thus, the obtained results are confirmed by several methods and by reproductivity of the measured data among different samples.

The ultralow values of percolation threshold might be explained by the balance between high dispersion degree and, at the same time, moderate agglomeration between nanotubes [1]. It is also often referred to kinetic percolation threshold due to filler movement and re-aggregation [6,9]. The origin of this phenomenon could be in the formation of small-scale chain-like aggregates which allows formation of a percolative conductive network at concentrations noticeably lower than it would be reached at the uniform distribution. This behavior was repeatedly observed in experimental and simulation studies [1, 6-9]. Besides, it is worth noting, that the classical percolation theory is quite limited model where such parameters, as filler curvature and agglomeration degree are out of consideration.

The corresponding discussions were added to the text to represent the results achieved more illustratively.

4. For the strain sensing properties of SWCNT/TPU composites, how about their stability and repeatability? Since SWCNTs are high aspect ratio and rigid, the conductive SWCNTs networks can not be completely recovered after stretching.

I thank Prof. Zhu for this important remark. Indeed, in addition to their sensitivity, repeatability and aging stability of strain sensors is a key parameter for their applications in soft electronics. In this study, cycling tests, which could demonstrate the stability of the samples, were not conducted since it was beyond the scope of the research objectives. The goal of this work was to investigate fundamental possibilities for piezoresistivity (strain sensing) and EMI shielding, *i.e.*, prerequisites for applications in soft electronics.

Nevertheless, I agree that it is a crucial indicator for applications and definitely worth studying in the further investigations of the elastic SWCNT-based nanocomposites, fabrication method of which was developed in the current work. The corresponding remark was added to the manuscript.

Response to Prof. Il Jeon

1. CO₂ and H₂, the growth promoters and CO, the carbon source are all gases. Therefore, the ratio of the injected amounts of each component is important while they vary easily with even a small change in temperature. Because MFC (Mass Flow Controller) is a device that controls the flow rate per minute and at different temperatures, the number of molecules vary. Hence, how did you manage to maintain constant temperature? From the illustrations you provided there was no contraptions or equipment that help maintain the temperature.

Indeed, MFCs control the volume of gas passed per minute, which can be affected by the environment temperature. Nevertheless, it should be noted that the reactor and the gas supply system are located in the thermostated laboratory room. Thus, temperature deviations do not exceed 1-2 °C. Thus, as the system works under room temperature, these deviations result in volume and, hence, flow rate inaccuracy not exceeding 0.7%, which is comparable with the accuracy of MFC operation.

Meanwhile, even such temperature deviations could be crucial for ferrocene vapor pressure (for instance, it is 1.00 Pa at 25 °C and 1.34 Pa at 28 °C, so the difference in catalyst concentrations could reach 30%). This is why the cartridge with ferrocene is kept in the thermostat at the fixed temperature of 28 °C.

The corresponding discussions were added to the Thesis text.

2. Growth parameter, H₂ is recognised for their role in high yield and quality production of CNTs owing to their small size which give arise to high mobility and reactivity at high temperatures. As such, SWCNTs (single-walled carbon nanotubes) of higher yield and quality are typically synthesized at high temperatures only. Then, my question is that why CO exhibits optimal efficiency at a relatively low temperature of 880 °C. Alternatively, what results can we expect when the synthesis temperature is higher than 880 °C when using H₂? If the optimal temperatures for the two promoters are different, more detailed explanation is called for.

Indeed, temperature has a major impact on the synthesis process. Typically, two aspects of temperature effect can be considered - thermodynamics and kinetics. According to the thermodynamical calculations (**Appendix 2** in Thesis), both CO disproportionation (the Boudouard reaction) and hydrogenation – the reactions leading to carbon release – become suppressed at temperatures above ~ 700 °C, *i.e.*, the reverse reactions are dominant. Thus, from thermodynamical viewpoint, lower temperatures are beneficial for synthesis productivity. Moreover, in early papers devoted to the aerosol CVD synthesis of nanotubes, the optimum temperature range for the Boudouard reaction was found as 470 – 820 °C (based on online detection of CO₂ as a reaction product) [12]. Nevertheless, the key reason for selecting a temperature above 800 °C is in Arrhenius's dependency of nanotube growth rate, *e.g.*, in overcoming activation energies of rate-limiting stages for nanotube growth. Thus, an optimum of 850 – 880 °C for nanotube growth in the current aerosol CVD method due to the opposite effects of thermodynamics and kinetics was found [13,14].

Nonetheless, since, indeed, in terms of kinetics, increase in temperature could positively affect the synthesis process. Moreover, recently in the works of our group, two different temperature regimes in nanotube growth were found: low-temperature one before 900 °C and high-temperature one above 900 °C). Thus, for deeper understanding of H₂ effect in the CO-based synthesis, SWCNTs were synthesized at 1000 °C. It was found that H₂ influences catalyst activation stage in the low-T regime leading to the enormous increase in the yield. In the high-T one, it affects the nanotube growth stage resulting in nanotube length increase (Figure and, as a result, increase in both SWCNT film conductivity and yield. These in **Section 4.1.2** (on pages 78-86) and are reflected in **Figures 13-15**.

An optimum temperature for CO/H₂ mixture can be selected in dependency on the synthesis tasks being solved. As for conductivity applications, since in the low-T regime (at 880 °C), H₂ increases the yield by ~ 15 times without deterioration of film conductivity, this temperature can be considered as optimal in terms of conductivity/productivity balance (which was found to be one of the highest in this work).

Nevertheless, discussion of the temperature choice and its effect on the synthesis process are crucial indeed, so it was added to the Thesis.

3. In chapter 5, the candidate said the bundles get thicker from the SEM images (Fig 21), but it is difficult to agree from the provided images. (I highlighted it yellow. It would help a lot if you provide SWCNTs/bundles value over SWCNT/TPU nanocomposites)

Figure 18 compiles SEM images of SWCNT/TPU nanocomposites fabricated at different SWCNT concentrations. The idea if this figure was to demonstrate SWCNT dispersion in TPU matrix as well as to show SWCNTs/bundles approaching surface of TPU (region of bundles pullout off the polymer). Some thickening of bundles approaching TPU is believed to take place since it is in good agreement with the idea of high affinity of TPU to nanotubes, which is supported by relatively low nanocomposite conductivity because of high polyurethane wetting of nanotube walls (for instance, compared to the conductivity of the SWCNT/epoxy nanocomposites (**Section 5.2**) and agrees with the literature data [10,11]. Nevertheless, indeed, it could be difficult to observe by these images. Unfortunately, statistics on bundle thickness near the TPU surface would be ambiguous because of complex and rough surface of nanocomposite resulting in irregular image perspective.

For support of these observations, additional images were added to the Thesis text (**Figure A9, Appendix 5**) where bundle thickening can be observed clearer.

4. Please check references. (I highlighted them red) There seems to be a technical error

The technical errors of references were fixed.

5. The graphs provided need to be improved greatly, especially the figure legends

The figures were revised and updated according the recommendations.

6. There are many grammar, unit, capital/small letter mistakes, commas, punctuality and so on. (I highlighted them blue)

I thank Prof. Jeon for such a thorough review of my Thesis work. All the highlighted issues were corrected.

References

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