

Jury Member Report – Doctor of Philosophy thesis.

Name of Candidate: Mariia Zhiliaeva

PhD Program: Materials Science and Engineering

Title of Thesis: A novel straightforward wet pulling technique to fabricate carbon nanotube fibers

Supervisor: Professor Albert Nasibulin

Name of the Reviewer: Prof. Alexei Buchachenko

I confirm the absence of any conflict of interest	
April	Date: Nov 17, 2021

The purpose of this report is to obtain an independent review from the members of PhD defense Jury before the thesis defense. The members of PhD defense Jury are asked to submit signed copy of the report at least 30 days prior the thesis defense. The Reviewers are asked to bring a copy of the completed report to the thesis defense and to discuss the contents of each report with each other before the thesis defense.

If the reviewers have any queries about the thesis which they wish to raise in advance, please contact the Chair of the Jury.

Reviewer's Report

Reviewers report should contain the following items:

- Brief evaluation of the thesis quality and overall structure of the dissertation.
- The relevance of the topic of dissertation work to its actual content
- The relevance of the methods used in the dissertation
- The scientific significance of the results obtained and their compliance with the international level and current state of the art
- The relevance of the obtained results to applications (if applicable)
- The quality of publications

The summary of issues to be addressed before/during the thesis defense

The thesis presented by Mariia Zhiliaeva in completion of the Skoltech doctoral program in Materials Science and Engineering targets two goals. First, it advocates the novel wet-pulling method for the fiber production from the carbon nanotube films. Second, it explores a wide range of applications of these fibers, mostly for mechanical sensing. Thus, both parts of the discipline qualification, materials science and materials engineering, are represented in the thesis. Important and inherent feature of the thesis is the consideration of use of the product supported by the proofs of application principle. The thesis material is featured in two publications, both in topically relevant and high profile (Q1-level) scientific journals, plus one manuscript submitted to the journal of the same level. These accomplishments fully satisfy the requirements set by the criteria of the Skoltech doctoral program in Materials Science and Engineering and leave no doubts in the PhD qualification of Mariia in the corresponding scientific field.

The thesis text consists of four chapters. Introductory section 1 sets the goals of the work and provides short overview of the state of the art in the fields of carbon nanotube fiber production and force sensing devices. Though the coverage skips many details, it is enough to advocate the problem setting and approaches to the solutions, as well as to let the reader orient in the material that follows. Experimental section 2 describes the methods used for of carbon nanotube fiber preparation, including carbon nanotube synthesis, special treatments of the films and fibers for altering their properties and the techniques used to characterize the structure, optical, electrical, mechanical and acoustic properties of the fibers. Section 3 presents and discusses the research results. As I see it, it consists of two logical parts: subsections 3.1-3.3, devoted to materials synthesis, manufacturing, characterization and modification, and subsections 3.4, 3.5, which expose various sensing applications of the fibers. To my opinion, presentation of the latter suffers from uneven level of exploration achieved for each application. The force sensing is evaluated in details, to the stage close to prototyping, while only third a page with video is dedicated to electrical diode circuit. Vibration and heart rate monitoring applications are just demonstrated, without attempts to optimize the sensitivity and design. It might be instructive to combine subsections 3.4.1 and 3.5 to give a coherent presentation of the force sensors and combine other, less explored applications in another subsection or even section. However, I admit that this suggestion is more a matter of taste and the current structure has its own logic and advantages. The final concluding chapter correctly summarizes the main findings of the thesis.

I have quite a few comments on the work, ranging from `for curiosity` questions to recommendations to revise the thesis text. Below they follow, with indications of the preferred way to handle them.

1. I feel a missing connection between the materials sections (3.1-3.3) and application sections (3.4, 3.5). While the former characterize a range of fibers manufactured in slightly different ways and possessing different properties, the latter stress more on the design. It would be instructive to point out what particular fibers were used (at least) for force sensor and how improvement of the fiber properties affects the sensor performance. And learn the experience on what is more

important and beneficial – optimization of the fiber properties or sensor design? [The author may wish to consider revision of the thesis text in response.]

2. Various sensing applications with fibers require quite distinct temperature regimes. Even wearable electronics should operate at temperatures higher than the room one, not to mention outdoor force or vibration monitoring. It would be interesting to learn on the stability range of fibers and how their useful properties vary with temperature. [I understand that this question is beyond the scope of the thesis, but feel that it may add an interest to in vivo discussion at the defense.]

3. It can be more instructive to merge Tables 1 and 2 and give a short statement on the recommended way of manufacturing (perhaps depending on application targeted, see comment 1 above) [The author may wish to consider revision of the thesis text in response.]

4. Subsection 3.1.3 proposes a scheme for large-scale fiber production technology. I wonder whether or not some more solid background behind (preliminary project, prototype, cost estimation, commercialization plan, etc.) does exist. [This is perhaps for in vivo discussion at the defense.]

5. The majority of the proposed applications lacks the comparison with alternative solutions. [Perhaps rightly, as instructive comparison would require optimization and quantitative testing. Take it as a comment for a future.]

6. Layout of the thesis text should be improved (broken tables, page breaks between the figures and figure captions, empty page, etc.). [This is perhaps the only obligatory revision to be made.]

These comments and questions by no means undermine the value of the research work done and very reasonable quality of its presentation. To my opinion, the thesis reports on general, flexible and cheap approach for carbon nanotube fiber production, potentially scalable. Wide range of applications of so produced fibers is considered. The most developed one, force sensor, is brought to the stage of optimized prototype. The others are less explored, but one should understand that the corresponding efforts go far beyond the scope and time line of a single PhD work. I therefore have no doubts to recommend the thesis submitted by Mariia Zhiliaeva for public defense after minor revision.

Provisional Recommendation

I recommend that the candidate should defend the thesis by means of a formal thesis defense

I recommend that the candidate should defend the thesis by means of a formal thesis defense only after appropriate changes would be introduced in candidate's thesis according to the recommendations of the present report

The thesis is not acceptable and I recommend that the candidate be exempt from the formal thesis defense