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Jury Member Report – Doctor of Philosophy thesis.

Name of Candidate: Hassaan Ahmad Butt

PhD Program: Materials Science and Engineering

Title of Thesis: Carbon nanotube fibers as embedded electrodes for the dual-stage monitoring of multi-functional carbon nanotube nanocomposites

Supervisor: Professor Albert Nasibulin

Co-supervisor: Assistant Professor Dmitry Krasnikov

Name of the Reviewer: Prof. Oleg Tolochko

I confirm the absence of any conflict of interest (Alternatively, Reviewer can formulate a possible conflict)	**Date: 22-11-2023**

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

Dissertation work of Mr. Hassaan Ahmad Butt dedicated to the development of technology for producing high-quality nanocomposite materials and in-situ studying their properties during synthesis and exploitation by using carbon fiber electrodes. Thesis shows the possible novel application of carbon fibers made from the Carbon Nanotube (CNT) through the wet pulling technique as embedded electrodes for the one-step manufacturing and lifecycle monitoring of carbon nanotube nanocomposites. The CNTFs are shown to be able to detect various parameters; manufacturing defects, lifecycle damage and some functional properties of CNT nanocomposites.

During the manufacturing cycle, the CNTF electrodes showed sensitivity to the concentration of both types of CNTs, proving that they may be utilized for the detection of different concentrations of different CNTs within a nanocomposite matrix. The electrodes showed almost identical resistance readings with negligible variance regardless of whether 2- or 4-point measurements were made, providing the advantages of reducing the number of electrodes needed for monitoring, reducing chances for regions of inhomogeneity which may cause later mechanical failure and allowing an inexpensive material-based manufacturing monitoring solution. Comparatively, metallic embedded electrodes have the disadvantage of high noise, contact resistance and inconsistency between batches, with measured values 1-2 magnitudes higher.

Overall structure of the dissertation looks well organized and logical. It consist of 5 Chapters and bibliography. The topic of the dissertation is relevant to its actual content. There are 4 publication of high quality, which were included in thesis, and 2 more papers submitted.

The experimental and theoretical studies carried out made it possible to significantly deepen and expand knowledge about the processes occurring during synthesis and exploitation of heterophase systems with CNT; a new concept for obtaining homogeneously desperation of CNT in nanocomposites was proposed. Suggested method has a strong potential to affect industrial production techniques for smart, self-diagnostic and multifunctional nanocomposites.

However, a number of questions, which can be discussed during the defense, are the following:

It is not clear how the concentration of nanotubes in the composites for study was selected. In general, composites with nanotube concentrations of up to 2% were prepared, but only two concentrations of CNTs (0.25% and 0.75%) were used for the study, which were used to determine the sensitivity of CNTFs to the properties and concentration of CNTs.

In general, for each type of filler there is an optimal value of its concentration, at which the maximum value of the strain sensitivity coefficient is achieved. Increasing the filler concentration may lead to two different effects. Firstly, the range of deformations in which the composite retains its conductive properties increases, which should lead to a decrease in the value of GF, and secondly, the value of the resistance of the composite in the absence of deformations decreases, which should lead to its increase. The lower the filler concentration in the matrix, the less stretching the sample begins to change its resistance. A significant increase in filler concentration leads to a decrease in the sensitivity of composites to tension: in order to noticeably change the resistance, such samples had to be stretched to a greater length. This effect of filler concentration on tensile sensitivity can be explained by the fact that when stretched, the distance between particles increases, which, from the point of view of conductivity values, is equivalent to a decrease in filler concentration. In addition, stretching the composite leads to a change in the value of the critical conductivity index t, which indicates a change in the dimension of the structure, and hence the value of the percolation threshold, because The percolation threshold generally depends on both the dimension of the structure and the ratio of the sizes of conducting clusters and non-conducting sections

So, some important properties of composites should be studied for more details, such as percolation limit, localization radius of the wave function (jump length), features of the three-dimensional network presumably formed by nanotubes, etc.

Tensile test showed that samples incorporating CNTFs show a piezoresistive responses similar to the samples measured with standard silver electrodes (gauge factor between ~2-12), whereas the samples with metallic electrodes showed higher responses at lower force values. However, it is not clear what variations of metallic electrodes had been used. What was the main principles of metallic electrode selection?

Author suggests that his concept, which is cheaper when compared to alternative techniques, has a strong potential to positively impact industrial production techniques for smart, self-diagnostic and multifunctional nanocomposites. However, this is not clear and also there are no any economical calculation.

The work contains many references to the functional or multifunctional properties of composites, but in general, only electrical conductivity and strain sensitivity were studied.

At the same time, the noted shortcomings are not of a fundamental nature and therefore do not reduce the value of the work performed and the results obtained and cannot have a significant impact on the positive assessment of the work as a whole. The reliability of the results obtained and the validity of the provisions and conclusions formulated in the work are due to mutually complementary modern research methods. The conclusions drawn are in line with the ideas of modern theories and the data of other researchers.

Assessing the work as a whole, I believe that in terms of the volume of data obtained, the level of generalization of experimental and theoretical material, and the novelty of the results, publication level it meets all requirements for PhD dissertations.

Provisional Recommendation

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