Review Report on PhD Thesis

Faculty: **Central European Institute of Technology**
Brno University of Technology in Brno

Academic year: **2024/2025**

Student: **Ing. Martin Kovařík**

Doctoral study program: **Advanced Materials and Nanosciences**

Supervisor: **Prof. Ing. Miroslav Kolíbal, Ph.D.**

Reviewer: **RNDr. Martin Ledinský, Ph.D.**

PhD thesis title:

Fabrication of functional nanostructures and their analysis by surface-sensitive techniques

Topicality of doctoral thesis:

The topic of the dissertation is highly relevant, with a clear connection to microelectronics. Characterization using atomic force microscopy represents one of the key approaches for locally probing the electronic properties of nanomaterials. Moreover, the investigation of defects in GaN-based materials is an essential and critical issue for their large-scale application. Equally important is the demonstration and explanation of the influence of SEM measurements on the electrical properties of nanomaterials. A particularly suitable and promising alternative for studying these samples is the newly developed constant-current AFM method, which enables defined and reproducible measurements of local treshold voltages, thereby allowing the detection of potential shunts in the active layer.

Meeting the goals set:

The specific objectives or formal assignment of the dissertation are not explicitly stated in the thesis, which makes it somewhat difficult to assess them directly. Nevertheless, it is clear that the student focused on the electrical characterization of semiconducting materials at the nanoscale. The work has successfully advanced both the methodological aspects of characterization techniques and the practical implementation of measurements. As a result, the electrical properties of the studied materials can now be measured in a more reliable, reproducible, and interpretable manner, allowing meaningful comparison with other characterization methods. It is evident that the student has developed a very good understanding of measurement techniques and has been able to build upon this knowledge to establish new experimental approaches as well as the subsequent evaluation and interpretation of data. The fundamental goal, namely, to educate and train an independently working researcher, has been clearly achieved.

Problem solving and dissertation results:

The submitted dissertation presents a substantial body of high-quality and field-relevant results. Having previously evaluated the student's pre-dissertation thesis four years ago, I am in a position to clearly compare the outcomes from the beginning of the doctoral studies with the final results. The progress is remarkable, particularly in the depth of discussion, the clarity of interpretation, and the student's ability to place the findings into a broader scientific context.

The thesis also shows that the student had to overcome several fundamental obstacles that initially made local current measurements extremely challenging. He developed systematic approaches for cleaning both probes and samples, which enabled reproducible measurements over sufficiently long periods of time to allow meaningful characterization. Furthermore, he successfully addressed the issue of large conductivity contrasts between defects and the bulk material by developing the ccAFM method. This technique not only solved a major experimental difficulty, but also provided a gentler approach for both the sample and the probe. In addition, he managed to correct for AFM drifts and established synchronization of repeated measurements at the same location, ensuring higher reliability of the data.

The scientific quality of the results is convincingly demonstrated by a first-author publication in ACS Applied Energy Materials, together with two additional co-authored publications, which further underline significance of the work.

Importance for practice or development of the discipline:

It is evident from the above that the developed measurement techniques are clearly focused on addressing real and pressing challenges, like exploring the effect of SEM imaging on electrical properties of nanomaterials and detecting properties of GaN-based optoelectronic materials, particularly those related to their large-scale fabrication. The dissertation not only advances and refines standard AFM techniques but also demonstrates their application for a deeper understanding of defects in these materials. Such insights may ultimately contribute to simplifying characterization through faster, but less selective, methods such as SEM.

Formal adjustment of the thesis and language level:

The submitted dissertation thesis fully meets all formal requirements expected of a doctoral thesis. The published manuscript in ACS Applied Energy Materials clearly demonstrates the high quality of the achieved results, and their application potential. The language level of the dissertation is good; the text is well structured and provides a clear and comprehensive documentation of the applied AFM methodologies. The figures and graphical material are carefully prepared, and effectively illustrate the topics under discussion.

The dissertation, however, is not entirely free of errors and typographical issues. For instance, the bandgap of GaN lies in the UV region rather than in the blue region (p. 30), and the reference to "clasical band theory" on p. 69 is presumably intended to mean quantum theory. Nevertheless, these minor shortcomings do not significantly distract from the reading of the thesis.

Questions and comments:

1. The cAFM results presented on page 22 are interpreted as indicating reduced conductivity at the graphene grain boundaries. However, in the corresponding image, the grain boundaries appear to exhibit higher negative currents. Does this imply that the grain boundaries are in fact more conductive? What could be the underlying mechanism responsible for this behavior?
2. An unsuccessful attempt to correlate AFM techniques with EBSD crystallographic orientation in the characterization of CVD graphene is mentioned in the thesis. Could you explain in more detail how this correlation was attempted, and whether it would be feasible to design the measurements in such a way that the same location could be characterized?
3. In Figure 4.13, you discuss a sharper image obtained at higher setpoint current. How exactly is "sharpness" defined here, and where can this be observed? From my perspective, the measurements look rather similar. I am also intrigued by the very low cutoff voltage of –2.1 V at 70 pA setpoint. I would normally expect this threshold to increase with the set current. Could we have a look at the histograms of these measurements?
4. In Figure 4.15, I do not observe any clear correlation between the ECCI and ccAFM images. Is it possible that electrically active defects were not etched away? Could their spatial size be smaller compared to other electrically inactive defects?
5. Is the effect shown in Figure 4.16 dependent on the direction of the flowing current (applied bias)? Did you attempt to perform discharging using UV illumination?
6. The SEM clearly affects the sample. Do you have an explanation for the square-shaped boundary artifact observed? Do you see any changes in the sample topography? Could SEM electron irradiation also structurally affect the nanotube itself?

Conclusion:

In summary, the dissertation thesis represents a substantial and original contribution to the field of nanoscale electrical characterization of GaN-based optoelectronic materials. The candidate has successfully advanced AFM-based techniques, overcoming fundamental methodological obstacles such as tip and sample cleaning, conductivity variations between defects and bulk layers, and measurement drifts. The introduction of the constant-current AFM approach has provided a more reliable, reproducible, and less invasive method, enabling the collection of meaningful data suitable for comparison with other characterization techniques.

CEITEC
VYSOKÉ UČENÍ TECHNICKÉ V BRNĚ

The research outcomes are of high scientific value, as demonstrated by first-author and co-authored publications in leading international journals. Importantly, the work demonstrates clear applicability to real-world challenges, provides explanations for possible SEM measurement artifacts, addresses the pathways toward more efficient defect characterization of large-area GaN films.

Overall, the dissertation confirms the candidate's ability to conduct independent, high-level research. The methodological advances achieved, the depth of analysis, and the scientific maturity displayed in the work leave no doubt that the essential goal of doctoral training – the development of an independent and capable researcher – has been fully accomplished.

In my opinion, the reviewed thesis fulfill all requirements posed on theses aimed for obtaining PhD degree. This thesis is ready to be defended orally, in front of respective committee.

In Prague, date 20th august 2025

....

RNDr. Martin Ledinský, Ph.D.