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REVIEW OF DOCTORAL THESIS

of Mrs. Ameneh Mikaeeli, titled:

"Thermal and Electrical Properties of Nanolayer Polymers"

under supervision of dr hab. Michał Pawlak, prof. NCU, and Prof. Andreas D. Wieck.

The doctoral thesis was conducted as part of a double degree program between the Nicolaus Copernicus University in Toruń, Poland, and the Ruhr University in Bochum, Germany.
The report on the thesis of Mrs. Ameneh Mikaeeli was done due to the request of the Chairman of the Physical Sciences Discipline council, dr hab. Piotr Maslowski, prof. NCU.

Assessment of topic of the thesis, its timeliness and originality

The thesis concerns characterization of thermal and electrical properties of conductive poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) - PEDOT:PSS thin films. The topic is timely and contributes to expanding knowledge on thermal and electronic transport in polymer materials. Mrs. Ameneh Mikaeeli is co-author of 6 international articles, in four of them as the firs author and other articles as a second and third author, which confirms her important role in the work. The results were published in the international journals, i.e. Nanomaterials, Materials, Applied Sciences, The Journal of Physical Chemistry C. PhD student presented results at international conferences in the form of oral and poster presentations. The quantitative and qualitative output of Mrs. Mikaeeli meets typical requirements for doctoral candidates.

The originality of the thesis lies primarily in the systematic analysis of thermal and electrical properties of conductive polymers in the form of thin films. The Author elaborated technological procedure of electric field assisted deposition with emphasis on modification of process parameters, like application of a lateral electric field and real time current monitoring during film deposition. Further, the application of electric field resulted in anisotropy of electrical properties of the film. The ability to manipulate the electric or thermal conductivities is important for materials dedicated for flexible electronic and energy conversion applications. The author correctly identified that thermal transport in such systems is far less studied than electrical transport. The strengths of the work lie in a clearly defined, up-to-date topic, a visible component of originality and good embedding in the international literature. Weaknesses are in limited discussion in conclusions of each chapter, there is a lack of final conclusions of the thesis.

Structure and composition of the thesis

The dissertation comprises a total of 130 pages, the actual work fits on 111 pages, with 61 figures and 17 tables. The thesis is divided into seven main chapters, regarding introduction and literature review, description of materials and samples preparation, experimental techniques to study thermal and electrical transport properties, investigation of influence of substrate and thickness on properties, and analysis of anisotropy thermal and electrical conductivities.

The first chapter sets the context of intrinsically conducting polymers, narrows to PEDOT:PSS, and states the research hypothesis, aim and objectives, scope, and the original scientific contributions. In the second chapter materials and sample, the preparation procedures are described, among them two PEDOT:PSS formulations: FHC Solar; 3–4 wt% aqueous dispersion, with and without $CuCl_2$, and different substrates applied like glass, ITO-coated glass, and GaAs:Si. The fabrication routes, and the electric field assisted deposition setup is also discussed. In the Chapter 3 measurement methods are presented. The chapter describes methods for thermal and electrical parameters determination, discusses theoretical models, a sensitivity analysis, and the Wiedemann–Franz law. The methods are generally described to a level that would allow replication, which is a strength. In the next chapter the theoretical background with Mott's VRH, fluctuation induced tunneling, disordered metal models and numerical simulations are presented. This separation of theory and experiment is good practice. The results from electrical measurements, comparison between the tunneling and VRH models, and the substrate, thickness influence on electrical properties are discussed in the Chapter 5. Next Chapter reports results from thermal measurements focusing on PTR derived thermal conductivity, the thermal anisotropy, the thermal and electrical correlation built on the Wiedemann–Franz framework. It is comparatively short (~10 pp) given the central role of its conclusions.

The last Chapter is the longest one and contains the most original material, the lateral electric field assisted deposition, real time voltage/current monitoring during samples fabrication, anisotropy analysis, C-AFM results, and the comparison with field free and doped samples. The overall conclusion of the thesis is presented in this short chapter (Sec. 7.10, p. 108) rather than standing as an independent concluding chapter of the thesis. Weaknesses can be referred to some parts that are repeated in the text, some paragraphs in chapters partially overlap with the discussion, which unnecessarily extend the text.

Research methodology and experimental reliability

The thesis investigates the electrical and thermal transport properties of conductive polymers deposited by solution based method on three different substrates. The doctoral candidate fabricated films with controlled thickness, morphology, and conductivity, then characterized them using complementary techniques. Electrical properties were tested by the Van der Pauw method integrated with the cryostat system to investigate the temperature dependent electrical conductivity. In addition to macroscopic methods, local electrical properties were investigated using C-AFM. Thermal properties were determined using photothermal infrared radiometry (PTR), and thermoreflectance method (TR). The surface morphology of thin films was studied by AFM (surface roughness Sq, Sa), and structure was investigated SEM and by X-ray diffraction method. There is only one exemplary XRD pattern for layer deposited on GaAs:Si substrate and one cross-sectional SEM single image in the Fig. 2.5, page 14 and Fig. 2.6, page 15. The XRD results could be better presented, e.g. by subtracting the background and describing the peaks. The thermal and electrical conductivities were then correlated and electronic contribution to heat propagation was derived using the Wiedemann–Franz law. A particularly interesting and valuable part of the thesis is the paragraph discussing charge transport mechanisms in conducting polymers supplemented with numerical simulations. The Mott's variable-range hopping (VRH), the fluctuation-induced tunnelling, and the disordered-metal models were tested for interpretation of the electrical measurement results. The author of the thesis stated that the Lorenz number in conducting polymers is not necessarily constant, thus the classical Wiedemann–Franz law may not be strictly applicable. The work does not convincingly resolve how electronic thermal conductivity is separated from phonon thermal conductivity when the very premise a constant Lorenz number is in doubt.

Another doubt concerns the reproducibility of results. The thesis reports that conductivity variation is below 3% (Section 5.2, page 55), but it does not clearly state how many samples were measured, how many batches were used, or provide error bars or confidence intervals for the electrical and thermal conductivity values. For a thesis that claims the anisotropy is reproducible, a clearer and more complete statistical analysis is expected. Similar situation is for AFM imaging. The results from surface roughness measurements were shown only for thin films deposited on glass, not on GaAs:Si (Sec. 5.4.3), thus the claimed substrate comparison cannot be verified morphologically.

Results and their interpretation

The thesis presents a thorough study of transport properties in conductive polymers based on PEDOT:PSS system using several complementary techniques and measurement directions. This makes it possible to reliably correlate electrical and thermal data. The work includes a careful sensitivity analysis with numerical modelling for estimating the thermal and electrical conductivity. The reviewer concern is that the electric field reorganizes PEDOT-rich domains, this is not directly supported by structural data. So far, this reorganization is suggested only by electrical measurements, not shown with imaging or other techniques that resolve the material morphology. The very high conductivity of the layer deposited on ITO (~8000 S/cm) and its attribution to PEDOT:PSS are not convincingly separated from ITO substrate/interface contributions or contacts influence. Another comment concerns the thermal conductivity and cross-plane electrical conductivity measurements. The only PTR measurements of thermal signal for one polymer layers on glass substrate were depicted in the Fig. 6.2, similarly to cross-plane electrical conductivity measurements performed only for one layer deposited on the GaAs:Si substrate. The discussion on influence of substrate on thermal conductivity or cross-plane electrical conductivity should be completed by measurements for layers deposited on different substrates, and layers of different thicknesses. The thesis discusses also the influence of incorporation of $CuCl_2$ on the electrical properties of polymer films. However, this conclusion is supported by only one brief paragraph. The author should clarify what results or observations support the statement that adding $CuCl_2$ did not improve the electrical conductivity and led to poor solution homogeneity. It would be highly beneficial to provide a comprehensive table summarizing the thermal conductivity, electrical conductivity, and morphological parameters for all tested samples. At present, the results are scattered in separate sections, which makes their direct comparison difficult. I would like to ask the doctoral candidate to comment on following questions:

1. Why does the electrical conductivity enhance perpendicular rather than parallel to the applied field, and how do your results reconcile with Mahajan (2015) and Kim et al. (2024)?
2. The Table 5.4 (page 61) shows results from electrical measurements of PEDOT:PSS thin films at three different temperatures: 0 K, 25 K, 50 K, I kindly ask the candidate to comment on:

   - temperature scale and what is the reason for such large difference in sheet resistance and conductivity values in Tables 5.4 and 7.5.
   - how did you calculate the electrical conductivity values in Table 5.4.
3. Could you comment on the statement, that PEDOT:PSS layers deposited on ITO-coated glass substrates exhibited significantly higher effective electrical conductivity compared to films deposited on insulating substrates (Page 66). For layer deposited on ITO/glass substrate with electrical conductivity ~8000 S/cm, what evidence shows that, this is the intrinsic PEDOT:PSS conductivity rather than an effective response dominated by the ITO layer or interface?

Language, style and formal aspects

The doctoral thesis is written in correct English and includes many technical elements, such as equations, figure captions, and parameter names. The style is generally clear, concise, and appropriate for a scientific work. The figures, graphs, and tables are generally easy to read, labelled, and well placed in the text, which helps the reader follow the argument. The author uses clear symbols and physical notation that follow standard conventions in solid-state physics and materials engineering. Overall, these features give the dissertation a professional appearance and support the reader's understanding of the experiments and their physical interpretation.

However, there are some issues that detract from the overall quality. Several sections contain repeated descriptions of the same methods or results across different chapters, which makes the text unnecessarily long and can confuse the reader about what is new in each chapter. In addition, some of the AFM images are of low quality: they appear noisy, have poor contrast, or lack of clear scale bars and labels. This makes it difficult to assess the morphology and nanostructure that is compared with electrical parameters. Improving the clarity and resolution of AFM images, and removing redundant textual descriptions, would strengthen the analysis of results and make the key findings more convincing.

Weaknesses: as mentioned before, there are minor typos and inconsistencies in the text, and a lot of repeated fragments that make selected chapters unnecessarily long, for example:

- figures are blurry with too small fonts, the AFM image shows artifacts, e.g. in the Chapter II: Materials and Sample Preparation Fig. 2.3, Chapter III: Measurement Methods, Fig. 3.13, Chapter V, Chapter VI and Chapter VII, Fig. 5.8, Fig. 6.1, Table 6.1, Fig. 7.10 and Fig. 7.11.
- page 12, repeated sentence: " To validate the thickness values estimated from the Ossila thickness-spin-speed relationship ..." and "validate the overall thickness trend predicted by the Ossila thickness-spins-peed relationship."
- page 29, Fig. 3.10 is from Ref. Nanomaterials 2024, 14(21), 1711 and not from Ref. arXiv preprint arXiv:2602.18840, 2026.

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- page 30, Eq (3.17), $k_{\perp n}$ in the legend should be the cross plane thermal conductivity and not thermal diffusivity.
- page 42 and 45, repeated sentences: "For $n = 3$, the model corresponds to three dimensional hopping…, However, interpreting the dimensionality parameter requires caution."
- page 46 and 47, Eq. (4.5) and (4.6) - conflict of symbol designations for σ_0.
- page 48, Fig. 4.7, page 51, Fig. 4.13, characteristics marked in black and blue, not fully visible on the graph.
- page 57, Fig. 5.2, results for layer of thickness 300 nm on GaAs substrate missing? It would be better measure layers with the same thickness, e.g. 240 nm, 320 nm, 480 nm deposited on different substrates - no results for the 240 nm layer on glass and the 320 nm layer on GaAs. There is incomplete description of Figure 5.2. "The thickness dependence of $T1$ and $T2$ for PEDOT:PSS (F HC Solar) thin films coated on glass substrate."
- page 58 and 59, repeated descriptions of Table 5.2 in the text: "Table 5.2 summarizes the fitting parameters obtained… The consistently high fitting quality demonstrates that the tunneling model accurately describes the temperature-dependent electrical conductivity for all investigated samples."
- Page 61, Fig. 5.4, AFM results presented only for layers deposited on glass, lack of results for layers deposited on GaAs:Si substrates. What is the size of the images?
- page 62, Table 5.4 - wrong temperature scale, missing symbols in the last column.
- page 63, Fig. 5.6 temperature dependence of sheet resistance – lack of fitting for graph a)
- page 73, Table 6.1 - poor quality screenshot, it is very difficult to read the data from the table.
- page 11 and page 91 repeated figures Fig. 2.1 and Fig. 7.3. – this is the same graph.
- page 68, Fig 5.8 - poor quality graphs, misleading labeling of the theoretical models used above the graphs.
- page 106, Fig. 7.11 poor quality of C-AFM graphs, lack of scale of graph g). What is the size of images. It is difficult to compare results from images of different size.

Final conclusion

In summary, I conclude that the doctoral thesis is an original achievement of the author, and clearly indicate that the PhD student has demonstrated the necessary knowledge, the ability to use various research techniques, and has the ability to independently design and conduct scientific research. The author correctly emphasizes that thermal transport in conductive polymers remains significantly less explored than electrical transport. The thesis therefore addresses an important research gap by providing a simultaneous and correlated analysis of thermal and electrical transport properties in the same PEDOT:PSS system. Despite critical comments, the thesis fulfills the statutory requirements set forth by the currently binding Act on Academic Degrees and Academic Title. Therefore, I hereby request that it be admitted to a public defense before the Council of the Discipline of Physical Sciences at the Nicolaus Copernicus University.