Review report on doctoral thesis

Title: **Plasma-Assisted Deposition of Functional Bioactive Nanostructured Films**
Author: **Petr Sezemský**
Reviewer: prof. Ing. Petr Zeman, Ph.D.

The submitted doctoral thesis deals with preparation and optimization of transparent conductive oxide films, namely indium tin oxide (ITO) films, on optical fibers, and with characterization of their structure and properties with the aim to develop biosensors with multiple interrogation, and high sensitivity and selectivity. It presents very interesting interdisciplinary research into the fields of plasma science, surface engineering, optical and electrochemical characterization, and biosensing.

The ITO films were deposited by the physical vapor deposition technique of magnetron sputtering utilizing benefits of non-equilibrium low-temperature and low-pressure plasma. For the deposition, radiofrequency (RF) or high-power impulse magnetron sputtering (HiPIMS) were used or a combination of both of them. HiPIMS, as a new advanced deposition technique, has attracted great attention not only of scientists but also of coating companies in the last 15 years. This technique makes possible to generate high fluxes of ionized atoms of sputtered material during very short high-power pulses applied to the sputtered target and thus to tune and control the microstructure and physical and functional properties of the prepared films. For these reasons, the deposition technique was chosen correctly.

For the sensing of biomolecules, a simultaneous multiple interrogation based on optical and electrochemical detection with crossover verification was proposed and tested. The optical detection builds on the lossy-mode resonance (LMR) phenomenon, in which specific wavelengths of the incident light propagated in high-n and low-k films such as ITO are attenuated. In parallel, the electrical conductivity of ITO provides the possibility of using it as a working electrode during the electrochemical detection. Based on these facts, the approach of combining the optical and electrochemical detection using ITO is very appropriate.

The thesis is written in very good English with almost no typos and errors and the reader can easily follow it. Only the figures could be printed in a higher quality to be more legible. The structure of the thesis is divided into three main parts, which are the theoretical section (50 pages), the research section with conclusions (28 pages), and 17 publications (270 pages). The theoretical section provides an introduction into the area of nanostructured surfaces, low-temperature plasma-assisted deposition techniques (especially magnetron sputtering), optical refractometry methods and cyclic voltammetry. Here, I appreciate mainly the description of basic principles and theory of the optical and electrochemical methods. On the other hand, I miss information how much the multiple interrogation based on simultaneous optical and electrochemical detection is original and if it has already been studied or applied to biosensing. I suggest that this be addressed during the thesis defense.

The research reported in the thesis is experimental and is a teamwork of several researchers from different institutions, as follows from the authorship of the publications. Although the contribution of

the author of the thesis is quantified and briefly mentioned on the first pages of the thesis, I would appreciate if the author could be more specific during the thesis defense and could describe his participation in preparation and characterization of the films in more detail. I would also have expected that in addition to the main aim of the thesis (listed on page 52), the individual tasks of author's work would be defined in the thesis as well.

The author's publishing activity is high, above the current standard for a doctoral thesis. It amounts to 12 articles in peer-reviewed impacted scientific journals indexed in the WoS database and 5 papers in conference proceedings. It should be, however, noted that he is the first author of two impacted articles and in most of the others, his contribution as a co-author is minor, especially in the papers dealing with optical or electrochemical testing. Therefore, in my opinion, it would be better and sufficient to include only the most important articles in the thesis. As for the conference contributions, I positively value his participation in 3 international conferences and 3 international workshops, where he always gave a talk, of which 1 was invited.

In conclusion, the submitted thesis represents high-quality research interconnecting several scientific disciplines and meets the requirements for a doctoral thesis in physics related fields. The main aim of the thesis was successfully achieved. The author has shown good expertise in experimental work and theory, and the ability to independently work in the field of science and publish his results in scientific journals and at international conferences. Therefore, **I do recommend** the thesis for the defense.

I have a few questions and remarks to the thesis:

1. What do you exactly mean by the fact that the prepared ITO films are "nanostructured", and did you investigate the effect of the nanostructure on the film properties and their sensing sensitivity?
2. You state that the crystallinity of the films is a crucial parameter determining electrical properties of the ITO films and can be improved by HiPIMS due to the energy influx to the substrate. How did you measure the crystallinity and quantify it?
3. One may expect that the elemental composition of the ITO films will be also a crucial parameter that will change as the process parameters (e.g., the total pressure, the composition of the gas mixture) are varied. Did you try to measure it, and can you comment on its effect on the optical and electrical properties of the ITO films?
4. Did you find any relationship between plasma parameters and properties of the ITO films?

Plzeň, January 27, 2022

prof. Ing. Petr Zeman, Ph.D.