



**80<sup>th</sup> International Scientific  
Conference of the  
University of Latvia 2022**

**University of Latvia  
Institute of Chemical Physics**

# **NANOTECHNOLOGIES AND RADIATION PROCESSES**

**ONLINE MEETING**

**February 3–4, 2022**



**UNIVERSITY  
OF LATVIA**



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*Nanotechnologies and Radiation Processes : Online meeting*. University of Latvia  
Institute of Chemical Physics, Riga, 3–4 February 2022. Riga: University of Latvia  
Press. 52 p.

Scientific Committee: *Dr. chem.* Gunta Kunakova

Editors:

Gunta Kunakova

Elīna Kerre

Prepared by the University of Latvia Press.

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ISBN 978-9934-18-799-5 (PDF)

<https://doi.org/10.22364/iscul.80.icp.nrp>

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# 80<sup>th</sup> International Scientific Conference of the University of Latvia 2022

## ONLINE MEETING PROGRAMME

February 3–4, 2022

[EET Time zone] <b>February 3, 2022</b>	
11:00 11:05	Opening
<b>KEY PRESENTATIONS</b>	
11:05 11:45	<p>The recycling of waste heat through the application of nanofluidic channels (TRANSLATE)  <b>Justin D. Holmes</b>  <i>School of Chemistry &amp; Environmental Research Institute, University College Cork, Cork, Ireland</i></p> <p><b>Steffen Hardt</b>  <i>Institute for Nano- and Microfluidics, Technische Universität Darmstadt, Darmstadt, Germany</i></p> <p><b>Satarupa Dutta</b>  <i>Institute for Nano- and Microfluidics, Technische Universität Darmstadt, Darmstadt, Germany</i></p> <p><b>Ievgen Nedrygailov</b>  <i>School of Chemistry University College Cork, Cork, Ireland</i></p>
11:45 12:25	<p>Astronomy-oriented THz detection with graphene  <b>Samuel Lara-Avila</b>  <i>Quantum Device Physics Laboratory, Department of Microtechnology and Nanoscience, Chalmers University of Technology, Gothenburg, Sweden</i></p>
Lunchtime break	
<b>ENERGY / MATERIALS / RADIATION PROCESSES</b>	
13:00 13:20	<p>Extraction and mass-separation of <sup>43</sup>, <sup>44</sup>, <sup>47</sup>Sc radionuclides from irradiated natural Ti targets at the CERN-MEDICIS facility  <b>Edgars Mamis</b>  <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i>  <i>CERN, Geneva, Switzerland</i></p>
13:20 13:40	<p>Blended vs. exfoliated Bi<sub>2</sub>Se<sub>3</sub>/CNT hybrid structure-based thermoelectric nanocomposites – properties and applications  <b>Krisjanis Buks</b>  <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i>  <i>3D Strong Ltd., Ulbroka, Latvia</i></p>
13:40 14:00	<p>The formation of solid electrolyte interface on Bi<sub>2</sub>Se<sub>3</sub> thin films from aqueous electrolytes  <b>Yelyzaveta Rublova</b>  <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i></p>

Break (10 min)	
14:10 14:30	<p>Porous anodized aluminium oxide thickness determination employing spectroscopic ellipsometry</p> <p><b>Aušrinė Jurkevičiūtė</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i></p>
14:30 14:45	<p>Application of PEM cells for hydrogen isotope separation</p> <p><b>Rudolfs J. Zabolockis</b> <i>Institute of Chemical Physics, Faculty of Chemistry, University of Latvia, Riga, Latvia</i></p>
14:45 15:00	<p>Seebeck coefficient and resistance of flexible carbon nanotube-bismuth selenide thermoelectric thin films</p> <p><b>Lāsma Bugovecka</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i></p>
15:00 15:15	<p>Changes in tritium concentration after sorption depending on storage conditions</p> <p><b>Elza Lagzdina</b> <i>Institute of Chemical Physics, Faculty of Chemistry, University of Latvia, Riga, Latvia</i></p>
Break (15 min)	
15:30 15:50	<p>Self-assembled gold nanoparticle arrays for antigen based biosensors</p> <p><b>Aleksandrs Dutovs</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i></p>
15:50 16:10	<p>Microfluidic mixers and magnetic particle capture chambers based on OSTE polymer</p> <p><b>Janis Cipa</b> <i>Faculty of Physics, Mathematics and Optometry, University of Latvia, Riga, Latvia Cellboxlab Ltd.</i></p>
16:10 16:25	<p>Automated dataset acquisition for nanoporous anodic alumina sensor substrate production monitoring and performance evaluation</p> <p><b>Vladislavs Perkaņuks</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i></p>
16:25 16:45	<p>Deep trapping states in cerium doped <math>Gd_3Ga_3Al_2O_{12}</math></p> <p><b>Toms E. Šusts</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i></p>

[EET Time zone] **February 4, 2022**

### 3D TOPOLOGICAL INSULATORS / RADIATION PROCESSES

10:00 10:20	<p>Obtaining quantum confinement in topological insulator nano-devices</p> <p><b>Xavier Palermo</b> <i>Quantum Device Physics Laboratory, Department of Microtechnology and Nanoscience, Chalmers University of Technology, Goteborg, Sweden</i></p>
10:20 10:40	<p>Synthesis and structural characterization of magnetically-doped <math>Bi_2Se_3</math> nanowires</p> <p><b>Andrei Felsharuk</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i></p>



10:40 11:00	Charge transport properties of encapsulated topological insulator nanoribbons <b>Gunta Kunakova</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i>
Break (10 min)	
11:10 11:30	Tritium retention in plasma facing materials of JET ITER-Like-Wall campaigns and factors influencing it <b>Anete S. Teimane</b> <i>Institute of Chemical Physics, Faculty of Chemistry, University of Latvia, Riga, Latvia</i>
11:30 11:50	Spectrometric analysis of agriculture residual fibers <b>Liga Avotina</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i>
11:50 12:05	Photoluminescence of lithium orthosilicate pellets prepared via solid-state synthesis <b>Mareks Senko</b> <i>Institute of Chemical Physics, Faculty of Chemistry, University of Latvia, Riga, Latvia</i>
12:05 12:20	Manufacturing and characterisation of flexible Bi <sub>2</sub> Se <sub>3</sub> /CNT heterostructure thermoelectric materials <b>Loreta Brauna</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i>
Lunchtime break (40 min)	
13:00 13:20	Physical vapor deposition synthesis of Bi <sub>2</sub> Se <sub>3</sub> nanoribbons on thermally dewetted Au nanoparticles <b>Raitis Sondors</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i>
13:20 13:40	Epitaxial growth of topological insulator thin films by physical vapor deposition technique <b>Kiryl Niherysh</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i>
13:40 14:00	Bottom-up synthesized Bi <sub>2</sub> Se <sub>3</sub> nanoribbons for applications in nano-electromechanical switches <b>Liga Jasulaneca</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i>
Break (10 min)	
14:10 14:25	Chemisorbed gaseous compounds on surface of lithium orthosilicate containing ceramic materials analysed by gravimetry and TG/DTA-FTIR spectrometry <b>Anna Ansonē</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i>
14:25 14:40	Method development of permeation studies for tritium-labelled water vapour for proton-exchange membranes <b>Patricija Kalnina</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i>

14:40 14:55	<p>Methods for fabricating networks from copper oxide nanowires synthesized by thermal oxidation</p> <p><b>Davis Gavars</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i></p>
14:55 15:10	<p>Study on formation of radiation-induced defects in advanced ceramic breeder pebbles using different types of ionising radiation and absorbed dose</p> <p><b>Madara Tomele</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i></p>
Break (10 min)	
15:20 15:40	<p>Characteristics of ionic transport in highly ordered nanoporous aluminium oxide membranes</p> <p><b>Valerii Malyshev</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i></p>
15:40 15:55	<p>Tungsten nanolayer gradual oxidation and oxide analysis by infrared spectrometry</p> <p><b>Annija E. Goldmane</b> <i>Institute of Chemical Physics, University of Latvia, Riga, Latvia</i></p>
15:55 16:00	Closing remarks

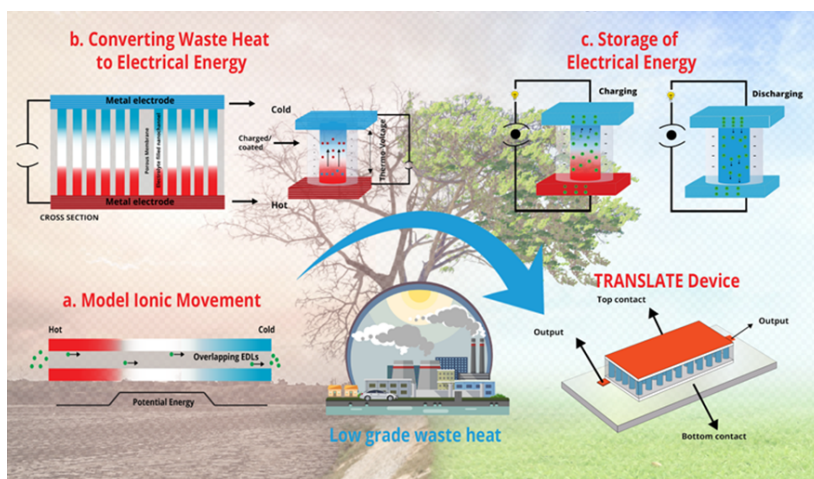
# THE RECYCLING OF WASTE HEAT THROUGH THE APPLICATION OF NANOFUIDIC CHANNELS (TRANSLATE)

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While climate change, increasing energy consumption, deteriorated air quality and exhaustion of natural resources represent central economic and social challenges today, the waste heat energy released into the atmosphere is a clean, fuel-free and inexpensive energy source. Seventy per cent of energy generated daily is waste heat. Even though thermoelectric and thermo-electrochemical cells are technologies used to convert waste heat into electrical energy, there does not yet exist a technology for the viable harvesting of low-grade waste heat [1]. This presentation will provide an overview of the EU-funded project ‘TRANSLATE’ which aims to develop a proof-of-concept nanofluidic platform technology based on the flux of ions in nanochannels (Fig. 1) [2]. The work targets innovation in versatile and sustainable energy harvesting and storage [3].



**Fig. 1.** Graphical Representation of the TRANSLATE project

## References

- [1] Agathokleous, R. et al. Waste Heat Recovery in the EU industry and proposed new technologies. *Energy Procedia*. **2019**, 161, 489–496.
- [2] Dietzel, M.; Hardt, S. Thermoelectricity in confined liquid electrolytes. *Phys. Rev. Lett.* **2016**, 116, 225901.
- [3] <https://cordis.europa.eu/project/id/964251>

# ASTRONOMY-ORIENTED THZ DETECTION WITH GRAPHENE

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Further leaps in THz astronomy demand new materials to implement devices capable of reaching fundamental detection limits [1]. In this talk we discuss the possibility to use graphene to implement THz detectors, owing to the small heat capacity and weak electron-phonon coupling of this two-dimensional material. With graphene doped to the Dirac point, its resistance is dominated by quantum localization effects, and the thermal relaxation of charge carriers is governed by electron diffusion [2]. This combination allowed us to demonstrate a graphene bolometric mixer with potential to outperform state-of-the-art detectors based on superconducting materials [3]. Given the scalability of the material and in conjunction with emerging quantum-limited amplifiers, we foresee the possibility to implement large arrays of quantum-limited sensors in the THz domain for radio astronomy, potentially surpassing superconductor-based heterodyne detectors.

## References

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- [2] He, H.; Kim, K. H.; Danilov, A.; Montemurro, D.; Yu, L.; Park, Y. W.; Lombardi, F.; Bauch, T.; Moth-Poulsen, K.; Yakimov, T.; Yakimova, R.; Malmberg, P.; Müller, C.; Kubatkin, S. & Lara-Avila, S. Uniform doping of graphene close to the charge neutrality point by polymer-assisted spontaneous assembly of molecular dopants. *Nat. Commun.* **2018**, 9, 3956.
- [3] Lara-Avila, S.; Danilov, A.; Golubev, D.; He, H.; Kim, K. H.; Yakimova, R.; Lombardi, F.; Bauch, T.; Cherednichenko, S. & Kubatkin, S. Towards quantum-limited coherent detection of terahertz waves in charge-neutral graphene. *Nat. Astron.* **2019**, 3, 983.

# EXTRACTION AND MASS-SEPARATION OF <sup>43,44,47</sup>Sc RADIONUCLIDES FROM IRRADIATED NATURAL TI TARGETS AT THE CERN-MEDICIS FACILITY

Edgars Mamis<sup>1,2</sup>, Elina Pajuste<sup>1</sup>, Thierry Stora<sup>2</sup>, Sebastian Rothe<sup>2</sup>, Eric Chevallay<sup>2</sup>, Charlotte Duchemin<sup>2</sup>, Maija Radzina<sup>3</sup>

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<sup>43,44g,44m,47</sup>Sc are very promising medical radionuclides in “matched theranostic pair” radiopharmaceutical development for cancer treatment. Use of natural titanium as target material for production of <sup>43,44g,44m,47</sup>Sc radionuclides is favourable in terms of cost and allow to produce these isotopes in sufficient amount for clinical applications. While previous investigations have directed towards the use of enriched targets with cyclotrons or nuclear reactors, we report here the recent studies, development, production and extraction of scandium radionuclides from irradiated high purity natural titanium rolls at an isotope mass separation facility, such as MEDICIS at CERN.

In this study, natural titanium target material was irradiated at the MEDICIS target irradiation station with 1.4 GeV protons delivered by the CERN Proton Synchrotron Booster (PSB). Although nuclear reaction cross-sections of <sup>43,44g,44m,47</sup>Sc computed for nat-Ti(p,x) confirm that enough production yield can be achieved to synthesize radio-bioconjugates for imaging studies [1], the isotope separation and target operating parameters were yet to be determined. Furthermore, the presence of contaminant isotopes such as Sc-46 in the final product is not acceptable, therefore produced Sc radionuclide separation step, according to their mass using the Isotope Separator OnLine (ISOL) technique and the MEDICIS mass-separator, is mandatory. The separation of Sc and Ti as pure radioactive beams is challenging due to their reactive nature, high boiling points and low vapour pressure. To overcome this difficulty, more volatile molecule formation and extraction as molecular beams was investigated.

Low intensity radioactive Sc<sup>+</sup> and ScF<sub>x</sub><sup>+</sup> (x = 1–2) beams with surface ion source have been reported [2]. In this work, ScF<sub>x</sub><sup>+</sup> (x = 1–2) and natTiF<sub>y</sub><sup>+</sup> (y = 1–3) molecular beams in natural Ti and natural Ta environment have been obtained with a Versatile Arc Discharge Ion Source (VADIS). We hereby report our results on the positive effect of volatile rare earth and refractory metal molecular beam formation for isotopically pure <sup>44,47</sup>Sc extraction and mass-separation. Although ISOL and mass-separation is mandatory, chemical separation from presence of isobars in the collection foil must be considered.

## References

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# BLENDED VS. EXFOLIATED $\text{Bi}_2\text{Se}_3$ /CNT HYBRID STRUCTURE-BASED THERMOELECTRIC NANOCOMPOSITES – PROPERTIES AND APPLICATIONS

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Preparation of thermoelectric nanocomposites with inorganic nanostructure fillers is a promising route of manufacturing power sources for wearable low-energy devices. However, there exists various methods of preparing flexible nanocomposites, such as physical blending, solution mixing, etc., each having its own advantages and limitations. [1]

In this work we compared a traditional physical blending preparation method to a more innovative approach of exfoliation. The flexible thermoelectric nanocomposites were based on hybrid structures of  $\text{Bi}_2\text{Se}_3$  and carbon nanotubes (CNTs) functioning as an active thermoelectric filler, as well as a non-conductive polymer. Hybrid nanostructures like these have recently attracted the attention of many research groups around the world. [2] [3]

$\text{Bi}_2\text{Se}_3$  nanostructures were synthesized on CNT networks via the catalyst-free vapour-solid deposition technique. [4] The obtained  $\text{Bi}_2\text{Se}_3$ /CNT hybrid structures were blended into or exfoliated by a non-conductive polymer matrix. The morphology and chemical composition of the obtained hybrid structures and nanocomposites were analysed by using scanning and transmission electron microscopy and energy dispersive X-ray analysis. Thermoelectric and electrical properties were measured with a laboratory-made experiment setup and a physical property measurement system. The properties of the blended and exfoliated polymer nanocomposites were compared and the differences between the two preparation methods are discussed.

## References

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# THE FORMATION OF SOLID ELECTROLYTE INTERFACE ON $\text{Bi}_2\text{Se}_3$ THIN FILMS IN AQUEOUS ELECTROLYTES

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The development of lithium-ion batteries (LiBs) is proceeding at a rapid pace. They have a lot of applications, for example to power mobile communications, computer equipment, vehicles, equipment for industrial and military purposes and so on. The most common types of LiBs are batteries in which lithium hexafluorophosphate is dissolved in an organic solvent. But such chemical power sources have a number of disadvantages, the main of which is the combustibility of organic electrolytes, which can lead to fires and explosions. The insecurity of organic electrolytes has led to attempts to replace them with safer aqueous electrolytes. The first attempt to use aqueous lithium-ion batteries was made in the early 1990s, and in the 2010s, such research became more systematic. Numerous studies have shown that the cathodes commonly used for non-aqueous electrolytes are also stable in aqueous solutions, but the choice of a suitable anode for aqueous media has proven to be a challenge due to the dissolution of the material in water. Bismuth chalcogenides, in particular  $\text{Bi}_2\text{Se}_3$ , are promising anodes for aqueous media, but there are still many questions about them. One of the most important parameters that can characterize the safety and stability of the anode is the formation of the solid-electrolyte interphase (SEI) layer. In an aqueous electrolyte, gaseous decomposition products such as  $\text{O}_2$  and  $\text{H}_2$  by themselves cannot form the SEI layer on the electrode surface. However, oxygen and carbon dioxide dissolved in the solution can react with lithium and form the SEI layer from reduction products. For aqueous lithium-ion batteries, it is necessary to use concentrated aqueous electrolytes that ensure the formation of a stable SEI layer, since this layer quickly hydrolyses and dissolves in diluted electrolytes.

We investigate the formation and electrochemical properties of SEI layer on  $\text{Bi}_2\text{Se}_3$  thin films in 5 M  $\text{LiNO}_3$  water-based solution. It was determined by cyclic voltammetry that the SEI layer is formed during the first cycling at a potential of 0.84 V (vs. Ag/AgCl) and consists predominantly of  $\text{Li}_2\text{O}$  and  $\text{Li}_2\text{CO}_3$ . At the same time, electrode impedance spectroscopy data showed that the resistance of the SEI layer gradually decrease (~ 10 times) from the 1st cycle to the 100th, which may indicate the instability of the formed SEI layer. Thus, the instability of the SEI layer is the main reason for the decrease in the charge-discharge capacity of the anode, which decreased by ~ 3 times during the first 30 cycles.

## Acknowledgements

This work was supported by project No. 1.1.1.2/VIAA/4/20/694.



# POROUS ANODIZED ALUMINIUM OXIDE THICKNESS DETERMINATION EMPLOYING SPECTROSCOPIC ELLIPSOMETRY

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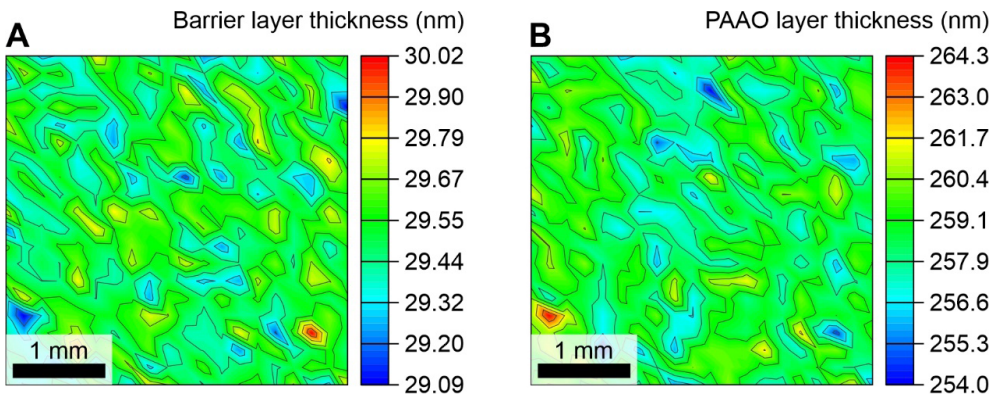
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Spectroscopic ellipsometry is a non-destructive technique for investigation of optical properties of materials as well as layer thicknesses of thin films. It is not affected by ambient light, intensity fluctuations of light source, and does not require special sample preparation. However, the desired results are obtained from the fitting of the model to experimental data [1].

This work presents the spectroscopic ellipsometry mapping of the thickness of porous anodised aluminium oxide (PAAO) sample. The measurements were performed employing rotating compensator GES5-E (Semilab) spectroscopic ellipsometer at 400 locations covering the sample surface ( $4.8 \times 4.8 \text{ mm}^2$ ) [2]. Ellipsometric parameters were acquired in reflection mode at  $55^\circ$ – $75^\circ$  incidence angles.

The analysis was carried out employing *Spectroscopic Ellipsometry Analyzer* (Semilab) software in wavelengths range of 250–950 nm. For optical model, 4-layer structure was described: aluminium, barrier layer ( $\text{Al}_2\text{O}_3$ ), PAAO ( $\text{Al}_2\text{O}_3$  mixture with air), and air. Optical properties of all materials were available in *n&k* database of employed software.

After the fitting of the data from all locations of the sample, the thickness values of barrier layer and PAAO were extracted. Results are presented in Fig. 1. Average thickness of barrier layer was 30 nm, while of PAAO – 259 nm.



**Fig. 1.** The topology (thickness maps) of PAAO sample, which was anodised for 217 s:  
A – barrier layer, B – PAAO layer

## Acknowledgements

This research is supported by European Regional Development Fund postdoctoral project “Patterned hybrid multilayer films for optical sensors” (No. 1.1.1.2/VIAA/4/20/615).

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# APPLICATION OF PEM CELLS FOR HYDROGEN ISOTOPE SEPARATION

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Hydrogen is the lightest chemical element with 3 naturally occurring isotopes – protium, deuterium and tritium, the latter being radioactive. Currently there are various methods of hydrogen isotope separation that is essential for applications in fields such as nuclear fusion. However, the main drawbacks of most methods are cost and low efficiency [1, 2]. Electrochemical separation can be considered as one of the most promising methods due to profound advances in the field of solid-state proton exchange membranes (PEM's) [3].

An electrolyser/fuel cell configuration was proposed for a liquid-gas-liquid type system to be used for hydrogen isotope separation from water. Ultrapure water (0,055  $\mu\text{S}/\text{cm}$ ) as well as tritiated water of the same purity was used for the electrolysis process. Electrolysis was performed at 2,1 V DC in *H-TEC* 1-Cell Rebuildable PEM Electrolyser. Produced hydrogen and oxygen gas was recombined in *H-TEC* 1-Cell Rebuildable PEM Fuel Cell and the produced water was collected. Tritium concentration in generated gas and water measured using tritium monitor TEM2100 with flow detector DDH 32 (TEM) and *PerkinElmer* Tri-Carb 2900TR liquid scintillation counter (LSC), respectively.

Electrolysis of water yielded hydrogen and oxygen gas that subsequently were recombined in the fuel cell to produce water. The liquid-gas-liquid concept appeared to function, however, factors such as the temperature of the water being electrolyzed as well as the pressure within the system have a great impact on the efficiency, achieving the highest electric current with warm water (40 °C). The hydrogen gas from tritiated water was collected and tritium concentration measured. Results show a difference in the ratio of tritium atoms to total hydrogen atoms, which further would indicate a difference in the generated water. This is to be studied in the future.

The electrolyser/fuel cell system operation was synced to facilitate liquid-gas-liquid transformation under 20 mbar pressure. The results obtained are to be implemented in future research regarding hydrogen isotope separation by electrolytic methods.

## Acknowledgements

This research has been performed within the framework of the European Regional Development Fund (ERDF) project No. 1.1.1.1/19/A/137.

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# SEEBECK COEFFICIENT AND RESISTANCE OF FLEXIBLE CARBON NANOTUBE-BISMUTH SELENIDE THERMOELECTRIC THIN FILMS

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Good thermoelectric (TE) materials could be one of possible resolutions for reducing global waste heat. These materials are building blocks for TE generators, which can transform thermal energy directly into electric power. Sources of waste heat are many hot equipment surfaces, industrial pipes, and even human skin. Given that many of these surfaces are non-linear and some are mobile, elastic and mechanically stable thermoelectric materials are needed.

Well-recognised high-performance TE materials at near-room temperatures are  $V_2VI_3$  type compounds such as  $Sb_2Te_3$  and  $Bi_2Se_3$ . However, materials from these chalcogenides and other inorganic TE materials are usually inflexible and brittle. At the same time, organic TE materials (polymers) are often very flexible, but with low efficiency. To combine good thermoelectric efficiency and mechanical stability at near-room temperatures, inorganic chalcogenides can be composited with carbon nanotubes. Thermoelectric composites usually have a functional thermoelectric filler, that is surrounded by an inert matrix, for protection from environment and better mechanical stability [1, 2].

In this work, carbon nanotube – bismuth selenide (CNT- $Bi_2Se_3$ ) thermoelectric composites with polyimide, polydimethylsiloxane (PDMS) and polyvinyl alcohol (PVOH) as an inert matrix were prepared. The resistance and Seebeck coefficient of these flexible TE thin films vs bending radius and bending cycles were studied. One of the 100 bending cycle results can be seen in Figure 1, which shows very little resistance change. Conclusions and comparison of the film mechanical stability is made.

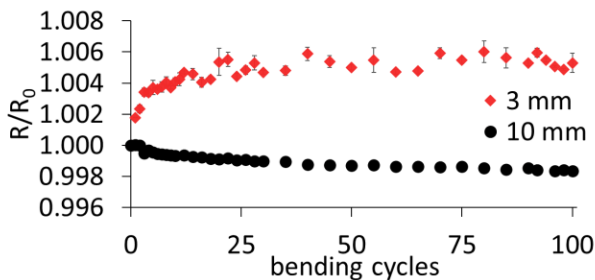


Fig. 1. Relative resistance change for a CNT- $Bi_2Se_3$  composite (CNTs = 3.2 wt%) encapsulated in PVOH vs bending cycles to 10 mm (black dots) and 3 mm (red squares) bending radius

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# CHANGES IN TRITIUM CONCENTRATION AFTER SORPTION DEPENDING ON STORAGE CONDITIONS

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Nowadays, as an alternative to traditional sources of energy, fusion energy is being developed and studied. Hydrogen isotopes – tritium and deuterium – are planned to be used as fuel in fusion reactors. Tritium is radioactive; therefore, its release should be kept to a minimum. At the same time, due to its radioactivity, tritium can be used for determination of behaviour of hydrogen in the materials. Therefore, experiments such as sorption and thermodesorption can be carried out.

In this study, tritium behaviour in SiO<sub>2</sub> based thermally insulating ceramics has been investigated

First, tritiated water vapour sorption was done in the studied material. Sorption took place at 577 °C in a hermetically sealed stainless-steel tube. Samples were exposed to air containing either tritiated water vapor HTO (60% relative humidity) or tritiated hydrogen gas HT (< 0.1%)

Two methods of tritium desorption analysis were selected to determine the behaviour of hydrogen isotope in the material. First, method of thermodesorption, during which release of tritium is measured as a function of temperature and time. This method was carried out immediately after sorption, samples were placed in a stainless-steel tube within a furnace, which was connected to a tritium monitor. During thermodesorption, gas passes through the system, as a result of which the desorbed tritium is carried to the measurement device. The second is the *Vance* method [1]. This method gives information on total amount of accumulated tritium and is based on tritium thermal release and its collection in a gas bubbler followed by the liquid scintillation measurement. For estimation of the effects of the storage conditions two approaches were used – storage in closed bags and open boxes. The same tritium measurement procedures were applied as for the samples that were analysed just after sorption experiments.

Thermodesorption spectra indicated two retention mechanisms of tritium in the studied material – most of the tritium is released already at the room temperature, whereas for the remaining tritium elevated temperature for detrapping and release is required.

It was estimated, that during the storage most amount of tritium is released within the first hours.

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# SELF-ASSEMBLED GOLD NANOPARTICLE ARRAYS FOR ANTIGEN BASED BIOSENSORS

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Antigen based biosensors of specific antigens present in human serum are widely used as a tool to diagnose various types of cancers, like leukaemia (acute and chronic), carcinoma (lung and prostate cancers) and other oncological diseases [1]. There are variety of diagnosis methods developed, such as electrochemical, fluorimetric and plasmonic biosensing, but many of them are time-consuming and require use of nanolithography and complex functionalizing agents, which are relatively expensive [2]. There is still necessity for developing rapid, relatively inexpensive, and still highly-sensitive methods for antigen detection. Due to localized surface plasmon resonances (LSPR) metal nanoparticles, such as gold nanospheres and their nanoarrays, are perspective type of sensor substrates to approach desired targets [3].

Here we show templated synthesis of dense gold nanoparticle arrays for plasmonic antigen detection. Au nanoparticles were deposited onto prepared template via dip-coating from a colloidal solution. Acquired nanoarray surface was functionalized with organic compounds and specific antibodies with antigens were incubated on modified Au nanoparticle surface. Sample was exposed to visible light under 45° angle and scattered light spectrum was collected and analysed.

Preliminary results show that Au nanoparticle surface functionalization tends to decrease scattered light intensity and slightly shift the maximum wavelength to blue spectrum region comparing to unmodified Au nanoparticle light scattering spectra. However, incubation of specific antigen on modified surface leads to noticeable scattered light intensity increase accompanied with wavelength shift to red spectrum region both comparing to unmodified and modified nanoparticles. The observed spectral change can be attributed to presence of specific antigens and antibodies on the sample surface as expected for LSPR sensing [3]. The high density of non-closely packed nanoparticle arrays results in a high signal to background ratio and enables measurements with widely available low-cost spectroscopy equipment.

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# MICROFLUIDIC MIXERS AND MAGNETIC PARTICLE CAPTURE CHAMBERS BASED ON OSTE POLYMER

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Early detection and diagnostics is the key towards increased prostate cancer (PC) survival [1]. Extracellular vesicles (EV) are a promising liquid biopsy marker for early cancer detection [2], however, EV detection efficiency is an ongoing issue due to the EV size and concentrations. As such, well known materials such as polydimethylsiloxane (PDMS) cannot be used due to small particle absorption [3]. Aim of the PROCEX project is to create microfluidic device for EV capture using functionalized magnetic nanoparticles and mixers. To achieve a working device, EV-compatible micro mixers, and magnetic particle capture chambers (MPCC) must be developed. In this work off-stoichiometry thiol-enes (OSTE) was used to fabricate planar accordion type microfluidic mixers, using soft lithography based on SU8 photoresist and PDMS molds. For MPCC soft lithography combined with 3D printed mold was utilised. Two OSTE filling approaches were tested: reaction injection molding, and casting technique.

Finally, microfluidic mixers and MPCC were created using casting technique as seen in Fig. 1., where mixing efficiency was evaluated using dyes such as tartrazine and methyl blue. For MPCC efficiency evaluation, iron nano particles were used in a permanent neodymium magnet flow-cell setup.

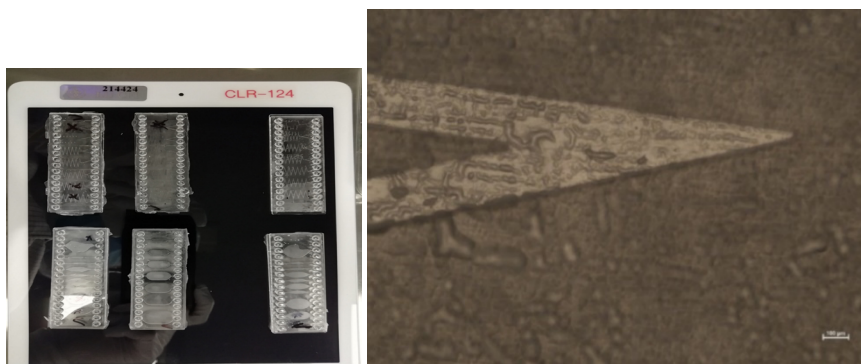


Fig. 1. Part of developed microfluidic mixer device, (left), magnification  $\times 5$  of a micro mixer channel (right)

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# **AUTOMATED DATASET ACQUISITION FOR NANOPOROUS ANODIC ALUMINA SENSOR SUBSTRATE PRODUCTION MONITORING AND PERFORMANCE EVALUATION**

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Nanoporous anodic alumina (NAA) is a self-organized material, that can serve as a template for low-cost production of nanostructured hybrid multilayer optical sensor substrates, for example, in metal-insulator-metal (MIM) configuration [1] or hosting zinc oxide nanorods [2] and other materials. However, production requires multiple steps and precise control of NAA geometry. In particular, the NAA thickness tuning (typically 300 nm) can be used to optimize the peak wavelength for localized surface plasmon resonance (LSPR) sensing using dense non-closely-packed (NCP) metal nanoparticle arrays on NAA surface [3].

We have developed an automated prototype technology line for rapid fabrication and testing of aluminum-NAA-gold nanoparticle array LSPR sensor substrates with an interferometric NAA thickness control system. It consists of a custom-built computer-controlled power supply unit with continuous anodization current monitor, optical setup for reflectance measurements inside electrolyte bath during anodization, computer-controlled dip-coating setup, automated spectral mapping system for quality and performance testing.

The control software is written in C++ and Python programming languages, whereas postprocessing of data is done in GNU Octave environment. It controls the process parameters, temperature, current, motorized parts, spectrometer, etc. and performs a real-time calculation of NAA thickness by fitting reflection spectra to a mathematical multilayer model based on propagation and matching matrices at each interface. In an ongoing development the source code is gradually being published in public repository [4].

The software includes functionality for uploading the structured datasets to open science repository Zenodo for traceability, transparency, review and indexing. The upload uses fairly simple application programming interface (API) functions. Briefly, in a first step an access token for user identification is generated. Next, a bucket URL is retrieved in JSON (JavaScript Object Notation) format for a new upload. The actual upload is done by sending a HTTPS PUT requests to the bucket URL and providing the access token as a parameter. Thereafter the user can edit metadata, elect to make the dataset private or public, reserve DOI number, or keep the upload open for adding new files at a later time.

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# DEEP TRAPPING STATES IN CERIUM DOPED $Gd_3Ga_3Al_2O_{12}$

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Cerium doped gadolinium aluminium gallium garnets (GAGG:Ce) possess a good light yield and energy resolution, fast scintillation response, high radiation stability and hardness [1]. Due to these properties, they have been proposed as potential scintillator materials for use in high luminosity collider detectors to mitigate the effects of event pile up [2]. For all radiation detection purposes the stability of radiation induced defects and glow curve peak properties must be considered.

In the present research the glow curve peak properties of two GGAG:Ce samples with differing after-synthesis conditioning were compared. After irradiation by X-rays thermostimulated luminescence (TSL) measurements were performed. The samples were heated from room temperature up to 620 K using a heating rate of 2 K/s. Radioluminescence (RL) measurements were performed under X-ray irradiation. 3D TSL measurements were performed from 10 to 300 K. Glow curve deconvolution was performed using the May – Partridge model for the glow curve description [3]. In addition to the mentioned methods, afterglow measurement technique and X-ray fluorescence (XRF) was used.

TSL glow curve deconvolution produced four glow peak maximums at 330 K, 345 K, 360 K and 415 K. TSL glow peaks show a negative correlation between their intensity and the time between irradiation and measurement. Afterglow was detected at room temperature. RL spectrum had only one peak with a maximum of 555 nm. In the 3D TSL spectra glow curves there were noticeable differences for the glow peak maximum intensities at 40 K between the sample types.

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# OBTAINING QUANTUM CONFINEMENT IN TOPOLOGICAL INSULATOR NANO-DEVICES

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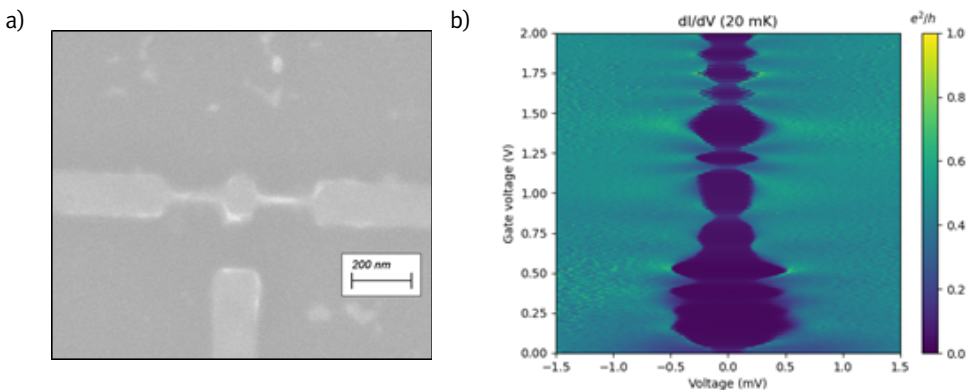
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A single electron transistor (SET) made with a topological insulator material (TI) could be the basic element of novel low-noise single electron sources, that could be used as Ampere standards in metrology. One way to do it is to create a quantum dot in a TI nanowire using constrictions as barriers. However, this is challenging since TIs are easily damaged by nano-fabrication processes.

In this work, we present a method to pattern an SET in Bi-doped  $\text{Sb}_2\text{Te}_3$  films and as-grown  $\text{Bi}_2\text{Se}_3$  nanobelts by electron-beam lithography and argon ion milling. This way, SETs could be fabricated, where constrictions down to a few 10 nm in width act as tunnel barriers (Fig. 1a). Preliminary electrical measurements at 300 mK indicated a switch from an ohmic to a tunneling behavior for constrictions of approximately 50 nm in width for both materials (Fig. 1b). Further measurements at 20 mK that confirmed the presence of Coulomb blockade in the devices, which we also discuss.



**Fig. 1.** Sample SET device in Bi-doped  $\text{Sb}_2\text{Te}_3$ , and corresponding transport measurement at 20 mK

# SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF MAGNETICALLY-DOPED $\text{Bi}_2\text{Se}_3$ NANOWIRES

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Topological insulators (TIs) are a new class of materials with an insulating bulk and topologically protected by time-reversal symmetry (TRS) conductive surface states, which has a wide range of potential applications from energy conversion devices to spintronics. Breaking the TRS of TIs makes the opportunity to emerge unique physical phenomenon as a quantum anomalous Hall effect, which may have potential applications in future electronic devices. One of the approaches to break TRS is the introducing of magnetic dopants into TIs [1].

In this work, synthesis and structural characterization of magnetically-doped bismuth selenide ( $\text{Bi}_2\text{Se}_3$ ) nanowires are presented and discussed. Different Cr- and Fe- doped  $\text{Bi}_2\text{Se}_3$  nanowires were synthesized by a simple catalyst-free physical vapour deposition method [2] on the different substrates with the dopant layers prepared in different ways on top of them.

Structural and compositional analyses of synthesized nanostructures in relation to the initial thickness and composition of the dopant layer were performed using different techniques such as atomic force microscopy (AFM), scanning (SEM) and transmission (TEM) electron microscopy, as well as energy-dispersive X-ray (EDX) spectroscopy.

TEM images and EDX spectra of synthesized nanostructures reveals the presence of magnetic impurities at a low percentage in them which opens the way for the fabrication of magnetoelectric devices with the controllable surface band gap.

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# CHARGE TRANSPORT PROPERTIES OF ENCAPSULATED TOPOLOGICAL INSULATOR NANORIBBONS

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Nanoribbons of 3D Topological Insulators are promising active elements in numerous devices of new concept relying on topological protection of surface states, for example, in topological Majorana qubits [1]. One of the key factors for realization of these exotic devices is ability to access the surface state transport in a controlled manner, using gating techniques. Nanoribbons of  $\text{Bi}_2\text{Se}_3$  are one of the best currently available TI nanomaterials, possessing high mobility and low bulk charge carrier densities. However,  $\text{Bi}_2\text{Se}_3$  is prone to formation of accumulation layers of a large carrier densities at its surfaces making it challenging to use the gate efficiently [2, 3]. More studies for selecting capping-layer material to prevent formation of the surface accumulation layer and appropriate dielectric for the gate electrode is therefore of great importance.

In this work we study  $\text{Bi}_2\text{Se}_3$  nanoribbons grown via catalyst-free physical vapor deposition. The obtained nanoribbons are free-standing and for fabrication of the all-around encapsulation layers, atomic layer deposition is used. Analysis of gate dependent magnetotransport in individual ZnO all-around encapsulated  $\text{Bi}_2\text{Se}_3$  nanoribbon Hall bar devices on  $\text{SiO}_2$  and h-BN substrates show reduced nanoribbon/substrate interface carrier density while keeping the surface mobility as high as for the as-grown nanoribbons.

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# TRITIUM RETENTION IN PLASMA FACING MATERIALS OF JET ITER-LIKE-WALL CAMPAIGNS AND FACTORS INFLUENCING IT

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ITER-Like-Wall (ILW) project has been carried out at Joint European Torus to test plasma facing materials relevant to International Thermonuclear Experimental Reactor – ITER [1]. First wall of the vacuum vessel is made of bulk beryllium tiles, whereas for the divertor bulk tungsten and tungsten coated carbon fibre (CFC) composite tiles are used.

During the shutdowns in 2012, 2014 and 2016, selected beryllium tiles were removed from the vacuum vessel. Tiles from 3 locations of the vacuum vessel were analysed, and results compared regarding both the tile position in the vacuum vessel and differences in the exploitation conditions during three ILW campaigns. Tritium results have been compared to deuterium data published by other authors [2, 3].

Tritium measurements were performed by multiple methods – thermal desorption spectroscopy, full combustion, and beryllium chemical etching. Prior to tritium measurements, scanning electron microscopy was used to study structure of the plasma-facing surfaces.

Experimental results revealed that tritium content in beryllium samples over all three campaigns is in range of  $0.75 \cdot 10^9$  to  $984 \cdot 10^9$  tritium atoms per square centimetre of the plasma-facing surface area (atoms/cm<sup>2</sup>). Highest tritium content was found in the samples from outer wall of the vacuum vessel, whereas, the lowest – in the upper part of the vacuum vessel. In contrary to results acquired for deuterium, in the outer wall tile higher tritium concentrations were found in the central part of the tiles where plasma induced erosion had occurred according to the SEM analysis data.

Results obtained within this study give possibility to assess tritium retention mechanism and make estimates of its possible inventory in larger machines such as ITER as well as gives an insight to the factors influencing tritium retention for further analysis of hydrogen isotope retention in tokamak type reactors [4].

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# SPECTROMETRIC ANALYSIS OF AGRICULTURE RESIDUAL FIBERS

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Agriculture residuals such as sheep wool fibers find place in medical and bio-engineering fields as it can be used as an alternative in producing various composite materials, in automotive, textile and construction industries [1, 2].

In order to expand field of application and create new products, thermal, radiation [3], and other modification methods can be used. For determination of induced effects, detailed characterisation of initial material is necessary.

In the present study a mechanically processed agriculture residual material – coursed sheep wool is selected. Characterization of free radicals was performed with electron spin resonance (ESR) spectrometry, chemical bonds detected with means of Fourier transform infrared (FTIR) and Raman spectrometry. Surface structure characterization – with scanning electron microscopy (SEM) and specific surface area determined with Brunauer-Emmett-Teller (BET) method. Thermal degradation processes analysed with means of thermogravimetry/ differential thermal analysis (TG/DTA) coupled with FTIR spectrometry.

ESR spectra show two relatively intensive signals: ~ 200G wide signal with an average g-factor value of 2.2; a narrow, slightly asymmetric signal ~ 9G and g-factor 2.007 in the range of organic radicals. FTIR and Raman spectra indicate presence of -CH, -CO, N-H groups and S containing compounds [4]. The BET method gives the specific surface area of 13.2 m<sup>2</sup>/g.

The obtained results will be summarized to create the data base for characteristic parameters of the mechanically treated sheep wool and will be used to estimate the thermal and radiation modification induced changes.

Research is performed within the frames of ERDF project No: 1.1.1.1./20/A/155 “Development of novel and innovative composite materials with enhanced sorption properties from renewable biological natural resources available in the Republic of Latvia for commercial air purification filtration systems”.

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# PHOTOLUMINESCENCE OF LITHIUM ORTHOSILICATE PELLETS PREPARED VIA SOLID-STATE SYNTHESIS

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Lithium orthosilicate ( $\text{Li}_4\text{SiO}_4$ ) is one of the lithium containing compounds extensively studied for various application purposes, such as lithium-ion batteries due to the ionic conductivity [1], solid sorbent for carbon dioxide capture due to its high sorption capacity and cyclic stability [2], and in future thermonuclear fusion reactors for tritium breeding due to the high lithium density, high melting point, acceptable tritium release behaviour and good radiation stability [3]. To improve mechanical properties, lithium metatitanate ( $\text{Li}_2\text{TiO}_3$ ) is added to form biphasic  $\text{Li}_4\text{SiO}_4$ - $\text{Li}_2\text{TiO}_3$  pebbles which are regarded as the most promising ceramic breeders for ITER's test blanket modules [4].

Objectives of this study include photoluminescence evaluation for possible characterization of material intrinsic and extrinsic defects) of solid-state prepared  $\text{Li}_4\text{SiO}_4$  pellets and  $\text{Li}_4\text{SiO}_4$  pellets with different amounts of  $\text{Li}_2\text{TiO}_3$ , ranging from 10 to 90 mol%.

Formation of secondary phases, such as lithium metasilicate ( $\text{Li}_2\text{SiO}_3$ ), lithium oxosilicate ( $\text{Li}_8\text{SiO}_6$ ), etc., are reduced using different synthesis approaches.

Several methods were used to determine and characterize the crystalline structure and optical properties of the prepared ceramic samples: powder X-ray diffractometry (p-XRD) and photoluminescence (PL). At room temperature,  $\text{Li}_4\text{SiO}_4$  pellets emits luminescence in the visible light range of 430–460 nm, corresponding to blue-light (~ 2.75 eV) energy. Addition of  $\text{Li}_2\text{TiO}_3$  phase during synthesis process results in another emission band in the red-light region at 720–740 nm (~ 1.7 eV).

## Acknowledgements

This research has been performed within the framework of the European Regional Development Fund (ERDF) project No. 1.1.1.2/VIAA/4/20/614.

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# MANUFACTURING AND CHARACTERISATION OF FLEXIBLE $\text{Bi}_2\text{Se}_3/\text{CNT}$ HETEROSTRUCTURE THERMOELECTRIC MATERIALS

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Some of the most promising materials for flexible thermoelectric generator production are thermoelectric nanocomposites based on conductive polymers. However, a drawback for the further application of these materials in thermoelectric generators is the low stability of n-type conductive polymers. [1]. Recently, it was shown that the application of environmentally-friendly non-conductive polyvinyl alcohol (PVA) as a matrix for  $\text{Bi}_2\text{Se}_3/\text{CNT}$  heterostructures used as filler is a successful alternative to nanocomposites based on conductive polymers [2]. However, the main drawback of the non-conductive polymers based thermoelectric composites is unwanted agglomeration of the filler, resulting in reduction of the thermoelectric figure of merit of the composites. Another way of fabrication of PVA- $\text{Bi}_2\text{Se}_3/\text{CNT}$  thermoelectric composites is coating of the as-synthesised on the solid substrate  $\text{Bi}_2\text{Se}_3/\text{CNT}$  network [3] with PVA, followed by its lift-off from the substrate to obtain flexible thermoelectric film. In such approach, fabrication of high-quality electrical contacts to the  $\text{Bi}_2\text{Se}_3/\text{CNT}$  network for the further characterisation and application of the composite films is crucial and challenging task.

This work is focused on the fabrication of PVA- $\text{Bi}_2\text{Se}_3/\text{CNT}$  flexible films with different types of electrical contacts. Thermoelectric and electric properties of the fabricated composite films were characterized with a laboratory-made experiment setup and a physical property measurement system.

The influence different contact materials on the thermoelectrical performance of the PVA- $\text{Bi}_2\text{Se}_3/\text{CNT}$  flexible films is discussed.

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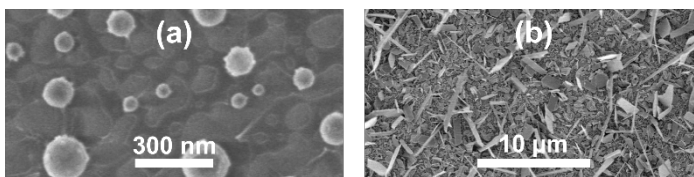
# PHYSICAL VAPOR DEPOSITION SYNTHESIS OF $\text{Bi}_2\text{Se}_3$ NANORIBBONS ON THERMALLY DEWETTED AU NANOPARTICLES

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Bismuth Selenide ( $\text{Bi}_2\text{Se}_3$ ) is a narrow bandgap semiconductor belonging to the three-dimensional topological insulator class. Due to their high surface-to-volume ratio,  $\text{Bi}_2\text{Se}_3$  nanostructures are good candidates for utilizing topological surface states [1].  $\text{Bi}_2\text{Se}_3$  nanostructures can be synthesized via solvothermal, sonochemical, chemical vapor deposition and physical vapor deposition (PVD) [2, 3] methods. To successfully use  $\text{Bi}_2\text{Se}_3$  nanostructures in device fabrication, it is necessary to develop high yield synthesis methods with controllable nanostructure morphology.

In this work  $\text{Bi}_2\text{Se}_3$  nanoribbons were synthesized via PVD on Au nanoparticles obtained by dewetting an initial Au layer. (Fig. 1) [4]. Yield and morphology of the synthesized  $\text{Bi}_2\text{Se}_3$  nanoribbons were characterized by scanning electron microscopy (SEM) and atomic force microscopy (AFM). X-ray diffraction (XRD) spectra were used to investigate nanostructure growth mechanism. Electron transport properties were characterized with a physical property measurement system. SEM and AFM measurements show that the morphology and yield of the synthesized  $\text{Bi}_2\text{Se}_3$  nanoribbons can be tuned by changing the thickness of the initial Au layer. XRD measurements suggest that different  $\text{Bi}_2\text{Se}_3$  nanoribbon growth mechanisms dominate depending on the size of the Au nanoparticles.



**Fig. 1.** SEM images: (a) Thermally dewetted Au nanoparticles; (b)  $\text{Bi}_2\text{Se}_3$  nanostructures synthesized by PVD on thermally dewetted Au nanoparticles

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# EPITAXIAL GROWTH OF TOPOLOGICAL INSULATOR THIN FILMS BY PHYSICAL VAPOUR DEPOSITION TECHNIQUE

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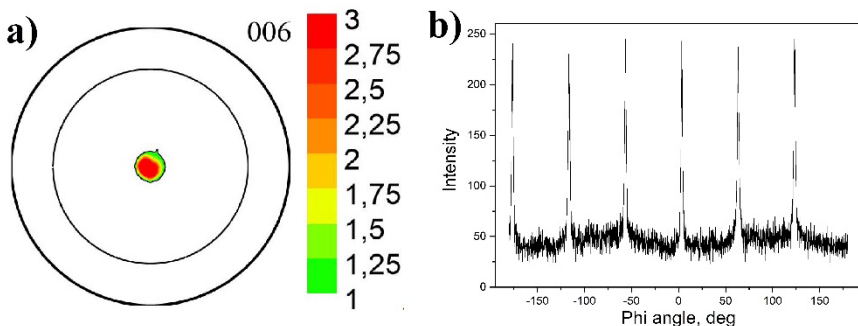
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Topological insulators (TIs) are a novel state of quantum matter with unique physical properties: linear energy–momentum dispersion and spin-momentum locking, which promise devices for electronic, spintronic, quantum computing and even domestic waste heat conversion applications. In this work, physical vapour deposition (PVD) method was used for the Volmer-Weber growth of  $\text{Bi}_2\text{Se}_3$  nanostructures, using separate Bi and Se sources materials [1].  $\text{Bi}_2\text{Se}_3$  nanostructures (nanoplates and thin films) were deposited under the same experimental conditions on different substrates (silica, mica, glass, SiC/graphene and CVD-graphene, silicon, silicon dioxide surfaces). The effect of different types of substrates (crystalline and amorphous) on nucleation and further lateral / vertical growth of the  $\text{Bi}_2\text{Se}_3$  nanostructures was analysed.

X-ray diffraction (XRD) analysis for thin films on amorphous and crystalline substrates was performed. All synthesized samples demonstrated an epitaxial growth of  $\text{Bi}_2\text{Se}_3$  thin film with crystallographic  $c$ -axis oriented perpendicularly to the substrate surface, which confirms the van der Waals growth mechanism for these films. However, the grains in the lateral plane are randomly oriented relative to each other for samples on amorphous substrates. While for crystalline substrates, highly-oriented film growth is also observed in the  $ab$  plane (Fig. 1b).



**Fig. 1.** (a) XRD pole figure of  $\text{Bi}_2\text{Se}_3$  thin film (11 nm) deposited on quartz. The presence of only one (central) maximum means that the crystals of  $\text{Bi}_2\text{Se}_3$  are coherently oriented in the  $c$ -direction; (b)  $\Phi$  XRD pattern of  $\text{Bi}_2\text{Se}_3$  thin film (11 nm) deposited on SiC/graphene. The presence of six equal maxima means that  $\text{Bi}_2\text{Se}_3$  crystals are highly oriented in the  $ab$  direction

Between the substrates investigated in this work, the crystalline ones are found to be the most promising substrates for the synthesis of high quality thin  $\text{Bi}_2\text{Se}_3$  films.

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# BOTTOM-UP SYNTHESIZED $\text{Bi}_2\text{Se}_3$ NANORIBBONS FOR APPLICATIONS IN NANOELECTROMECHANICAL SWITCHES

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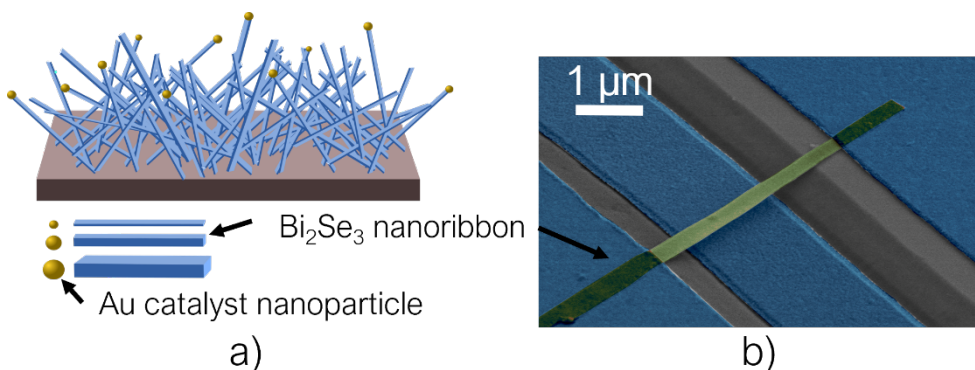
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Suspended quasi-one-dimensional nanostructures allow to implement various nanoscale devices and explore interesting phenomena that arise due to the interplay between electrical and mechanical domains. Bottom-up grown nanowires and nanoribbons are excellent candidates for application in these studies, as their composition, morphology and yield can be tuned as necessary by synthesis parameters.

Here we present a method for tuning morphology and yield of semiconductor  $\text{Bi}_2\text{Se}_3$  nanoribbons using physical vapor deposition on dewetted Au layers [1]. We employ the as-synthesized nanoribbons as active elements in nanoelectromechanical (NEM) switches [2], demonstrating for the first time switching at temperatures as low as 5 K.

We model the operation parameters and fabricate a NEM switch on a silicon chip, combining bottom-up synthesis and facile top-down photolithography. We achieve volatile and non-volatile reversible operation regimes depending on the balance between adhesion and elastic restoring forces. The fabricated devices show distinct ON and OFF states with switch-ON voltages as low as 7.8 V and switch-OFF voltages of 2.3 V and ON/OFF current ratio of  $10^3$ .

Our results expand the available material choice for cryogenic NEM systems and paves way for development of energy efficient devices working in conjunction with, e.g., quantum computers.



**Fig. 1.** (a) Schematics of nanoribbon morphology control using Au nanoparticles and (b) scanning electron microscope image of a  $\text{Bi}_2\text{Se}_3$  nanoribbon NEM switch

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# CHEMISORBED GASEOUS COMPOUNDS ON SURFACE OF LITHIUM ORTHOSILICATE CONTAINING CERAMIC MATERIALS ANALYSED BY GRAVIMETRY AND TG/DTA-FTIR SPECTROMETRY

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Lithium orthosilicate pebbles with addition of lithium metatitanate as a secondary phase are planned to be used for tritium breeding in fusion reactors [1]. This study evaluated changes of the surface chemical composition and sample mass during storage in air of the lithium orthosilicate pellets pressed from crushed pebble powder. Such study is necessary to develop an optimal pellet transportation methodology that could also be used for sample transport to high-energy heavy ion irradiation experiments at the Institute of Solid State Physics, Materials Science and Technology Centre (Kharkov, Ukraine).

The mass and surface composition changes are analysed by thermogravimetry/differential thermal analysis (TG/DTA) device coupled with Fourier transformation infrared (FTIR) spectrometer, as well as gravimetry method. The obtained data of TG/DTA-FTIR measurements indicated at least two distinct stages of weight loss during heating due to the release of gaseous compounds. Water vapour is mainly released at a temperature range of 150–320 °C, while carbon dioxide at a temperature range of 320–770 °C. On average, the mass fraction of gaseous compounds chemisorbed on the surface of the pressed pellets stored in air is  $11.4 \pm 1.2$  wt.%, determined by TG/DTA and  $10.507 \pm 0.014$  wt.% – by the gravimetric method. Therefore, ceramic samples containing lithium orthosilicate as the main phase must be stored and transported in an atmosphere free of water vapour and carbon dioxide in order to prevent chemisorption processes on the surface of the samples and the subsequent formation of lithium hydroxide and carbonate, because weight gain by 10 wt.% is too much for irradiation experiments with high-energy heavy ions.

This study has been performed within the framework of the Latvian-Ukrainian cooperation program project (agreement No. LV-UA/2021/4). The authors greatly acknowledge experimental support of Dr. Regina Knitter (Karlsruhe Institute of Technology, Institute for Applied materials).

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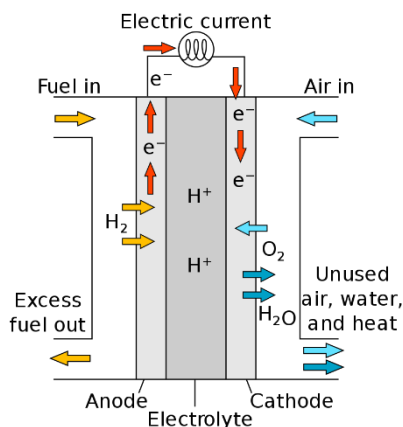
# METHOD DEVELOPMENT OF PERMEATION STUDIES FOR TRITIUM-LABELLED WATER VAPOUR FOR PROTON-EXCHANGE MEMBRANES

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Currently, the world's main energy source is fossil fuels. The continued use of fossil fuels is not advisable because fossil fuels are limited in amount, and they cause serious environmental problems. Sooner or later the energy sector will have to be restructured and a different energy carrier and source must be found. The use of hydrogen as a fuel for use in proton exchange membrane fuel cells (PEMFC) seems to be perspective [2].

To function, the membrane must conduct hydrogen ions (protons) but not electrons as this would in effect "short circuit" the fuel cell. The membrane must also not allow gas to pass to the other side of the cell, a problem known as gas diffusion [1].



**Fig.1.** Diagram of a PEM fuel cell [3]

Two different membranes (SPEEK and Nafion) were used for the determination of tritium diffusion. The tritium diffusion coefficient was determined under different conditions. The results were compared, and membrane compliance was assessed.

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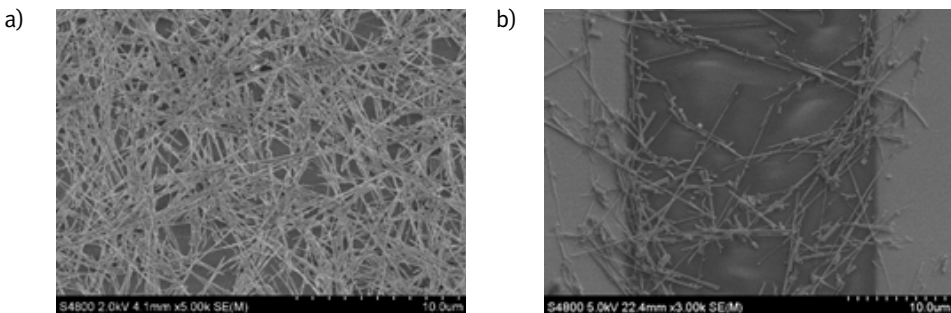
# METHODS FOR FABRICATING NETWORKS FROM COPPER OXIDE NANOWIRES SYNTHESIZED BY THERMAL OXIDATION

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Nanomaterials have been the subject of extensive research due to their potential applications in many different fields such as medicine, sensors, electronics, etc. [1] Because of copper's abundance, non-toxicity and low price, it is widely used in the synthesis of copper (II) oxide (CuO), which is considered to have great potential for various applications in superconductors, batteries, gas sensors and solar power storage devices, as well as thermoelectrics. [2]

In this work, CuO nanowires were synthesised on copper foil by thermal oxidation [3], applying different synthesis parameters (temperature, humidity, external electrical field). As-grown nanowires were ultrasonicated and aligned between the electrodes, using two different methods: dielectrophoretic alignment between two electrodes, prefabricated using lithography techniques, and drop-coating, where a nanowire suspension was added dropwise onto a glass substrate to create nanowire networks. Morphology and chemical composition of the as-grown nanowires and prepared nanowire networks was characterized using the scanning electron microscopy (SEM) equipped with energy-dispersive X-ray diffraction (EDX) system. Electrical and thermoelectric properties of the nanowire networks were measured in two-point configuration using Keithley 6430 sourceter and discussed. The choice of fabrication method was shown to affect the morphology and electrical properties of the as-fabricated CuO nanowire networks. The benefits and drawbacks of both fabrication methods are discussed.



**Fig. 1.** (a) CuO nanowire networks on a glass substrate;  
(b) Dielectrically aligned CuO nanowires

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# STUDY ON FORMATION OF RADIATION-INDUCED DEFECTS IN ADVANCED CERAMIC BREEDER PEBBLES USING DIFFERENT TYPES OF IONISING RADIATION AND ABSORBED DOSE

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Lithium orthosilicate pebbles with additions of lithium metatitanate as a secondary phase have been selected as the current EU reference material for tritium breeding in Helium Cooled Pebble Bed (HCPB) test blanket module which will be tested in ITER [1]. Considering the conditions inside a fusion reactor, the influence of different types of ionising radiation and absorbed dose on the formation and accumulation of radiation-induced defects must be investigated.

In this study, lithium orthosilicate pebbles with different nominal contents of lithium metatitanate were irradiated in air atmosphere at room temperature with X-rays using an X-ray lamp with operating parameters 45 kV and 10 mA for 30 minutes, gamma rays of a cobalt-60 source with a dose rate 2.28 kGy h<sup>-1</sup> up to absorbed doses 10, 26 and 53 kGy and Bremsstrahlung using a dose rate of 14 Gy min<sup>-1</sup> to obtain samples with 1 kGy absorbed dose. The content and thermal stability of accumulated radiation-induced defects was assessed using thermally stimulated luminescence (TSL) technique. TSL measurements were made for pebble type samples as well as for pebble samples crushed into a fine powder.

The acquired results show that the nominal content of lithium metatitanate had a significant effect on the content of TSL active radiation-induced defects in lithium orthosilicate pebbles. In addition, the observed TSL peak intensity of fine powders noticeably differed from the TSL glow curves of pebbles for the same sample.

This study has been performed within the framework of the European Regional Development Fund (ERDF) project (application No. 1.1.1.2/VIAA/4/20/614). The authors greatly acknowledge the experimental support of Julia M. Leys and Regina Knitter (Karlsruhe Institute of Technology, Institute for Applied Materials).

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# CHARACTERISTICS OF IONIC TRANSPORT IN HIGHLY ORDERED NANOPOROUS ALUMINUM OXIDE MEMBRANES

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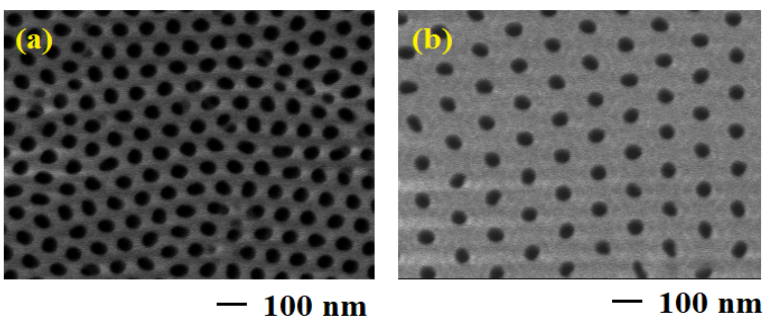
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Highly ordered nanoporous aluminum oxide (PAAO) is one of the most popular and cost-effective platform for various applications: from templates and molecular separation to drug delivery and energy generation [1]. Its' unique optical and electrochemical properties have been intensively explored as a platform for developing biosensing and energy harvesting nanodevices [2]. Ionic transport properties of membrane materials have the key importance for application of the membrane in the system of generation and storage of energy

Highly ordered nanoporous aluminum oxide (PAAO) membranes with different morphological characteristics have been obtained via the standard two-stage scheme anodization of aluminum with subsequent removal of the barrier layer. As the result, membranes with pore diameter from 15 to 40 nm and thickness of PAAO layer ( $h_{\text{PAAO}}$ ) from 9 to 112  $\mu\text{m}$  have been fabricated by anodizing aluminum in 0.3 M oxalic and sulfuric acids. The diameters of the porous channels in sulfuric-PAAO membranes were much smaller (13–20 nm) compared to oxalic-PAAO (33–40 nm) samples.

The morphology of the PAAO membranes was characterized by the means of scanning electron microscopy (SEM) (Fig.1.). The average values of pore diameters and inter-pore distances for membranes of different thicknesses depend on the type of electrolyte used.



**Fig. 1.** Top-view SEM images of oxalic-PAAO (a) at the top of sample, (b) at the bottom of sample,  $h_{\text{PAAO}} = 20,5 \mu\text{m}$ , average  $d_{\text{pore}} = 35 \text{ nm}$

For electrochemical measurements PAAO membranes were impregnated with sodium sulfate solution with different concentrations. Resistance of the PAAO membranes filled with electrolyte solution was measured with the use of electrochemical impedance spectroscopy. To this end special electrochemical cell was constructed.

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# TUNGSTEN NANOLAYER GRADUAL OXIDATION AND OXIDE ANALYSIS BY INFRARED SPECTROMETRY

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Tungsten (W) nanolayers can be used for the fabrication of field emitters in microelectronics due to the low work function and high thermal stability of these materials [1]. Magnetron sputtering is among the methods for producing the nanolayers. In order to monitor quality of the synthesized nanolayers, it is necessary to develop an analytical procedure suitable for the analysis of such nanolayers.

Infrared spectrometry is a sensitive method for the analysis of chemical bonds. However, W nanolayers contain weakly polar and non-polar bonds, that cannot be detected by FTIR, therefore a modifying step is introduced. Oxidation of W is selected as treatment procedure.

Oxidation of W nanolayers (150 nm thick W deposited by magnetron sputtering on Si/SiO<sub>2</sub> substrate) was performed in a thermal analysis device SEIKO EXSTAR 6300. The samples were heated up to 200, 400, 600, 800 and 1000 °C with a heating rate of 10 °C/min and then were allowed to cool down to room temperature. Infrared spectra were recorder prior treatment and after each applied temperature. Infrared spectra were recorded using Bruker Fourier transform infrared (FTIR) spectrometer Vertex 70v in the range of 400–4000 cm<sup>-1</sup>, with resolution of ± 2 cm<sup>-1</sup>, and in vacuum 2.95 hPa. FTIR spectra of the as-deposited layer showed weak peaks at 900 and 960 cm<sup>-1</sup> related to Si-O bonds, while a number of new peaks were observed in the spectra of the oxidized layers. The main peaks in the oxidized layers are at 636, 862 cm<sup>-1</sup> related to W-O bonds, and 797 cm<sup>-1</sup> related to W=O bonds [2, 3]. Additional peaks observed at 448, 1122 cm<sup>-1</sup> correspond to Si-O bonds, and the peaks at 549, 1039, 797 cm<sup>-1</sup> correspond to Si-O-Si bonds [4].

Applying thermal treatment, modification of the W bonds to W=O and W-O bonds is achieved and synthesized bonds are detected in FTIR spectra. It was observed, that in parallel to oxidation of the nanolayers, oxidation of the substrate is achieved, that could be from both – oxidation of Si and recombination of already existing oxide bonds in SiO<sub>2</sub>. These factors should to be taken into account for the analysis of W nanofilms.

The research was supported by the ERDF project No. 1.1.1.1/20/A/109 “Planar field emission microtriode structure”.

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