



Euroopa Liit
Europa
Regionaalarengu Fond



Eesti tuleviku heaks

Nanostruktuursete materjalide valmistamise ja karakteriseerimise võimalustest TTÜ-s

Dr. Marit Kauk-Kuusik
TTÜ Materjali-ja keskkonnatehnoloogia instituut



"Nanomaterjalide tehnoloogiate ja uuringute keskus (NAMUR+) (2014-2020.4.01.16-0123)

Nanomaterjalide valmistamise ja karakteriseerimise kompleks (MAT-SEM) TTÜ-s

- Asukoht: TTÜ Materjaliteaduste instituut
- Teadustöö algus: 2014
- Igapäevane teadustöö TTÜs ja eraklientide teenendamine
- Kontakt: marit.kauk-kuusik@ttu.ee



Kõrglahutusega Skaneeriv Elektronmikroskoop (HR-SEM)
Zeiss MERLIN



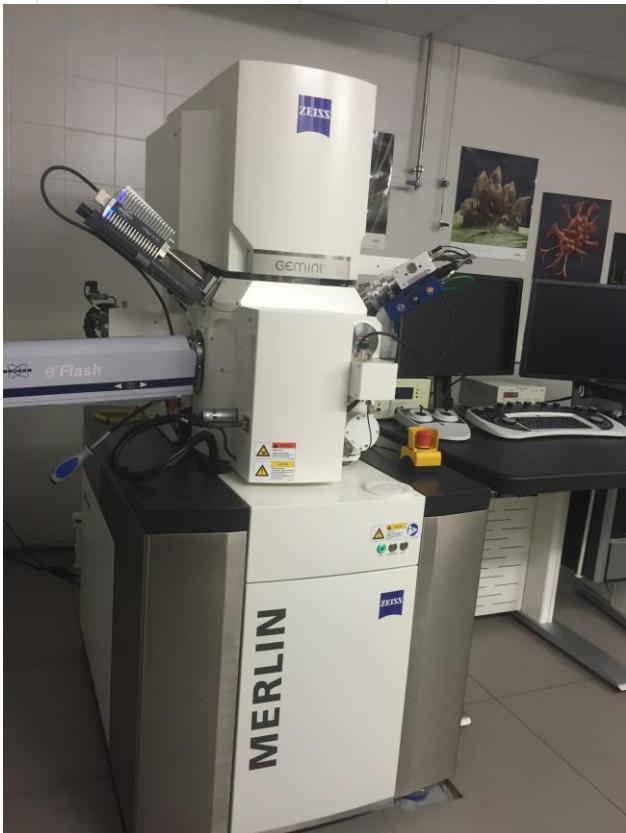
Kvartskristallide mikrokaalude
automaatsüsteem SamX



Pulss-laser sadestussüsteem
Neocera Pioneer 120

High Resolution Scanning Electron Microscope (HR-SEM)

Kompleksi põhiosaks on ülikõrgresolutsiooniga elektronmikroskoop (Zeiss MERLIN), mis võimaldab uurida nii **juhtivatest, magnetiseeruvatest ja kui ka mittejuhtivatest** materjalidest valmistatud objekte nii kõrgel lahutusvõimel kui ka suure analüütilise tundlikkusega.



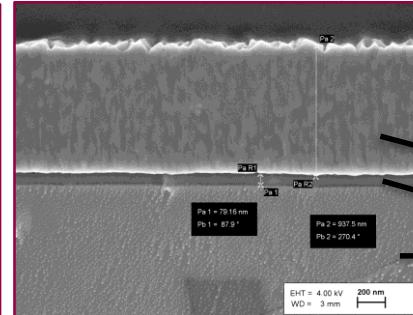
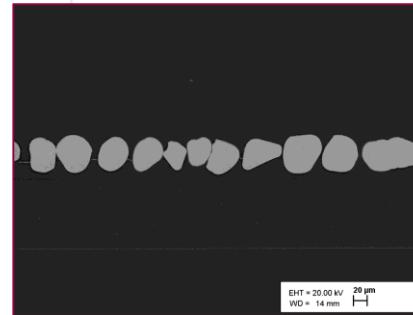
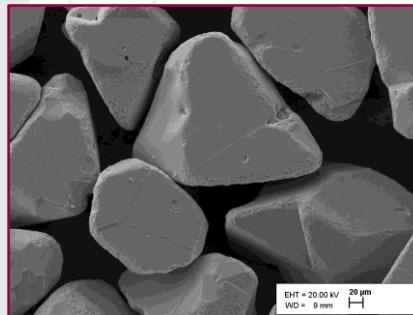
SEM kompleksi kuuluvad erinevad andurid : visualiseerimiseks EsB ja analüüsiks EDS (Energy-dispersive X-ray spectroscopy), EBSD (Electron backscatter diffraction), EBIC (Electron beam induced current).

- Ultra high resolution imaging at low kV**
 - 0.8 nm @ 15 kV; 1.4 nm @ 1 kV; ≤ 4 nm @ 0.1 kV

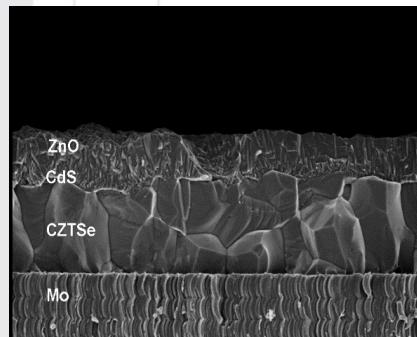
Resolution: Up to 3072 x 2304 pixel
- Ideal for precise boundary, feature, and particle measurements**
- Detectors**
 - EsB detector with filtering grid (grid voltage 0 - 1500V) BSE
 - High efficiency In-lens SE detector SE
 - Everhart-Thornley Secondary Electron Detector SE, BSE
 - EDS Detector EDAX Phoenix
- Large five axes motorised eucentric stage**
 - X= 130 mm; Y= 130 mm; Z= 50 mm
 - T= -3 - 70°
 - R= 360° (continuous)
- Chamber**
 - 330 mm(Ø) x 270 mm (h)
 - 2 EDS ports 35° TOA
 - CCD-camera with IR illumination
- Charge compensator with N₂ gas / sample cleaning with O₂ gas**
- Built in plasma cleaner**

HR-SEM rakendamine päikesenenergeetika materjalide uuringuteks TTÜ-s

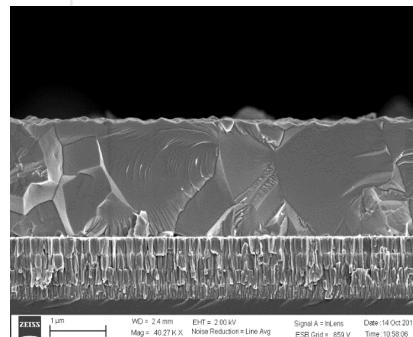
Monograin powder technology for photovoltaic applications



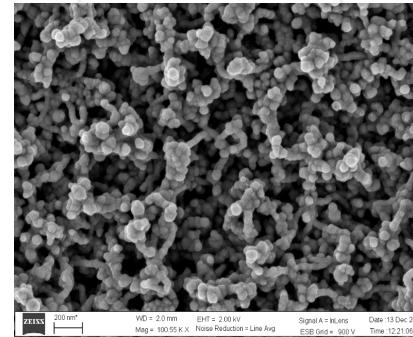
ZnO
CdS
CZTS



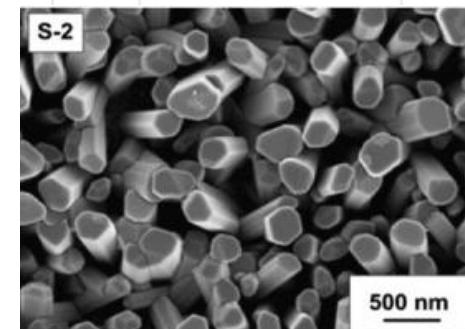
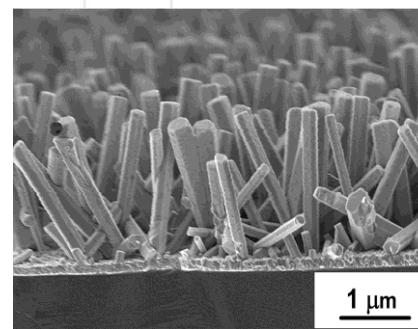
Cross-section of glass/Mo/CZTSe/
CdS/ZnO structures



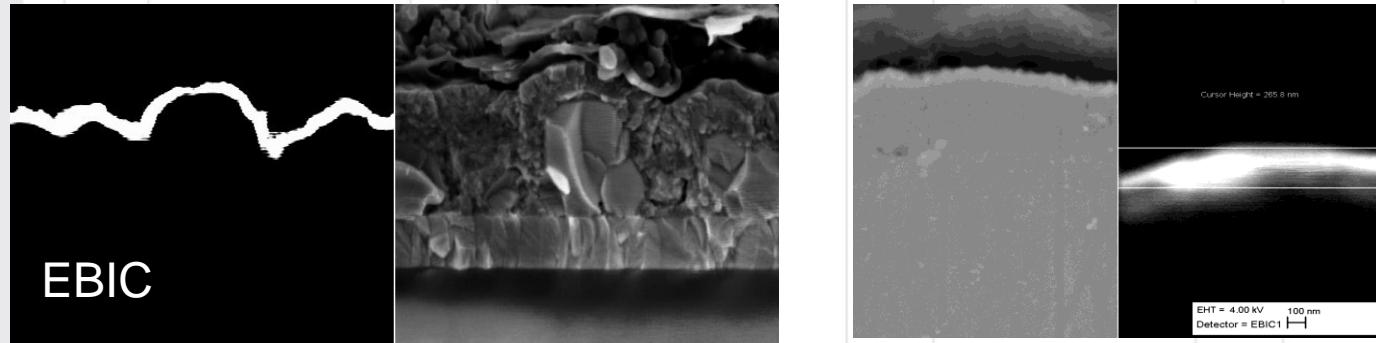
Cross-section of Mo/SnS thin films



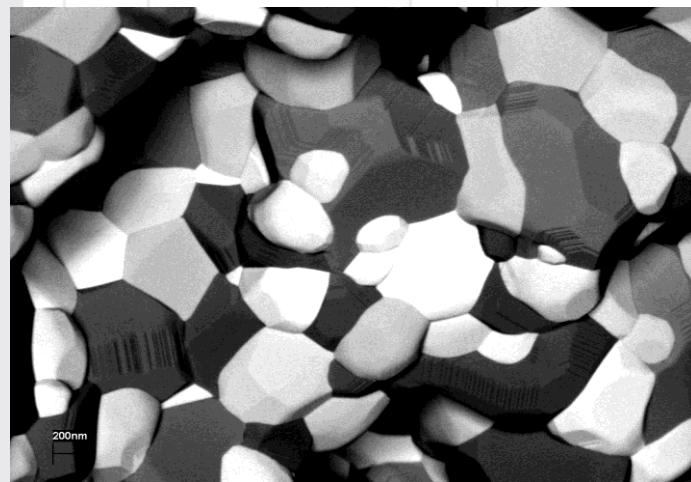
Surface of PPY/CdSe/ITO/glass structure



ZnO nanorods by spray pyrolysis method

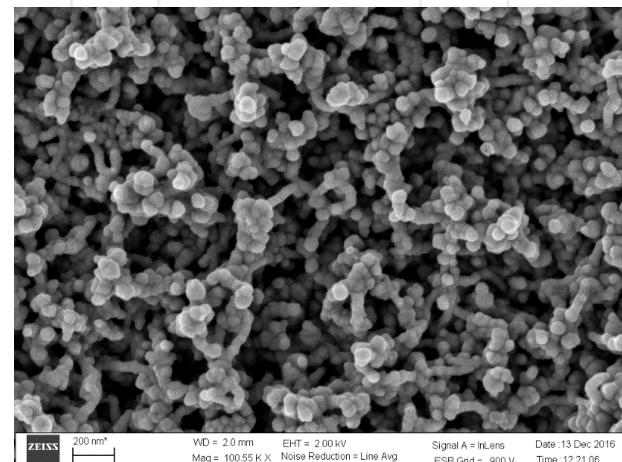
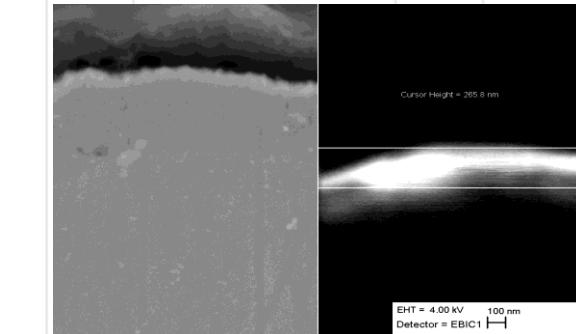


Electron Beam Induced Current (EBIC) for conductivity mappings



AsB Detector

angle selective back-scattered detector for crystallographic and compositional contrast imaging

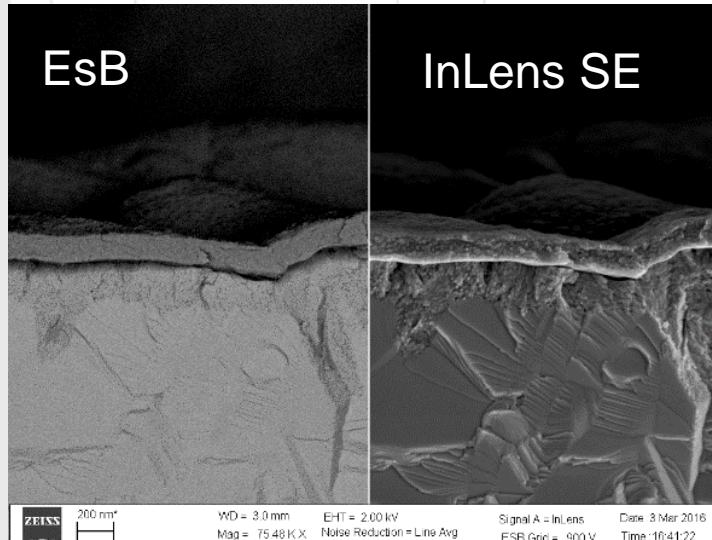


Surface image of PPY/CdSe/ITO/glass structure

ZEISS 200 nm² W/D = 2.0 mm EHT = 200 kV Signal A = InLens Date : 13 Dec 2016
Mag = 100.55 K X Noise Reduction = Line Avg ESB Grid = 900 V Time : 12.21.06



backscattered
electron image

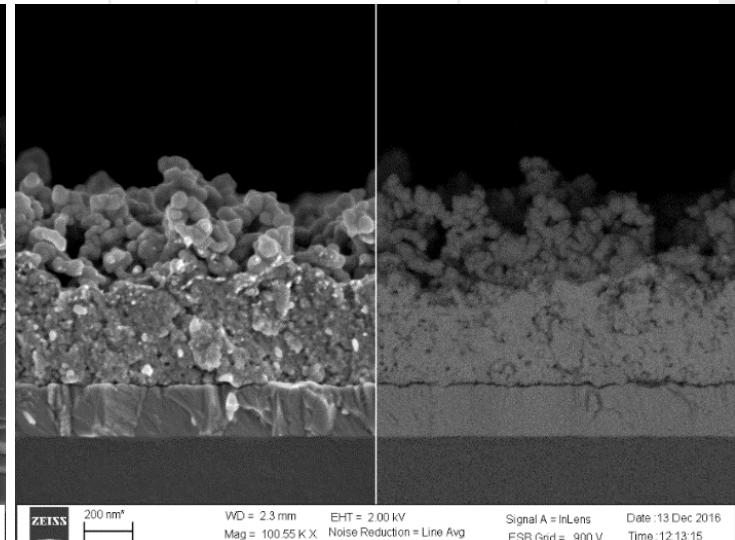


Cross-sectional view of glass/ITO/CdS/
CdTe/Polymer structure

secondary electron image

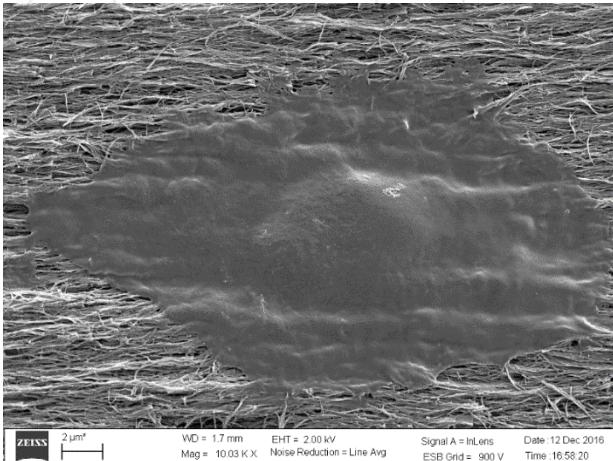


backscattered
electron image

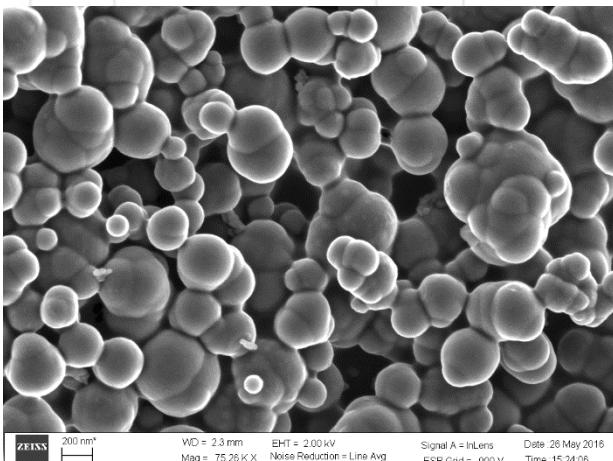


Cross sectional image of PPY/CdSe/ITO/
glass structure

Teaduskoostöö teiste uurimisgruppidega projektide raames

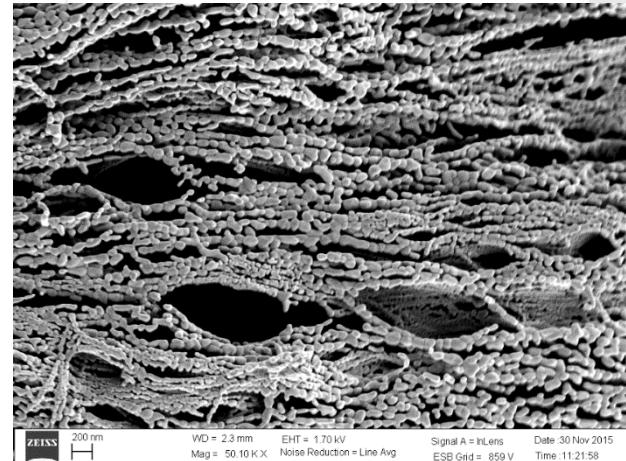


MDA-MB Cancer cell on graphene
(Project ULTRINIA - MTÜ PROTORTON)
ULTRINIA loob nanotehnoloogial põhineva meetodi haiguste või dopingujuhtude kiiremaks ja odavamaks tuvastamiseks.



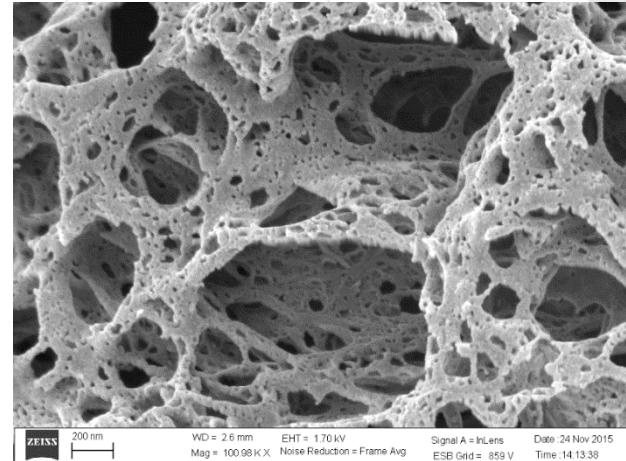
Sample from Skeleton
(Project LEP16077)

"Nanomaterjalide tehnoloogiate ja uuringute keskus (NAMUR+) (2014-2020.4.01.16-0123)



Top view image of ceramic nanofibers
(Project PUT 1063)

Keraamika kiudude nanovõrgustik sihitute funktsioonidega

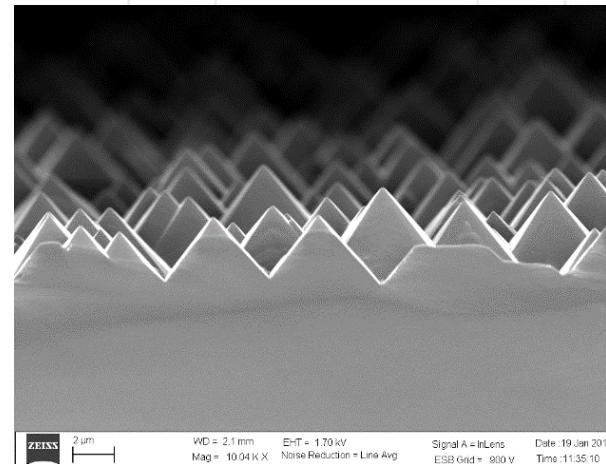
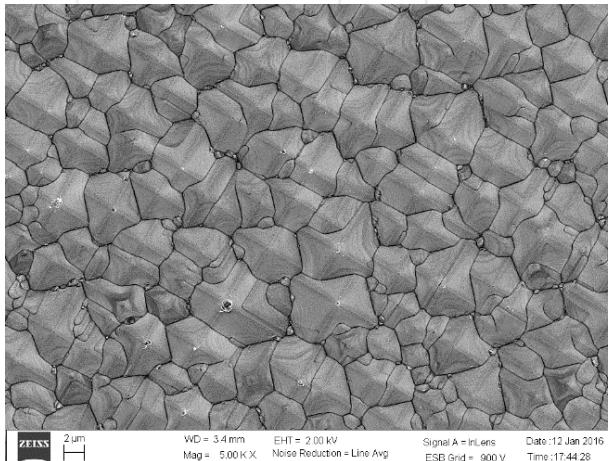


Top view of nanonet of ceramic fibers
(Project PUT 1063)

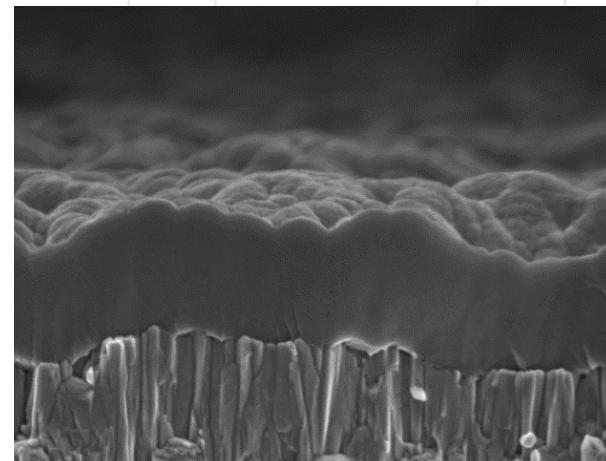
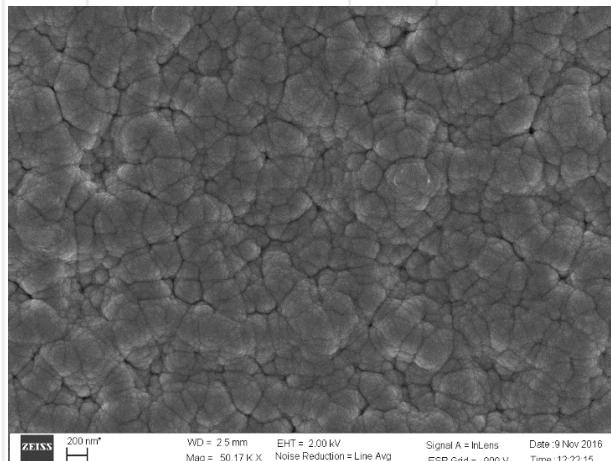
Teaduskoostöö teiste uurimisgruppidega projektide raames



ERA.Net RUS PLUS Project Flexapp (**ETAG15028**)

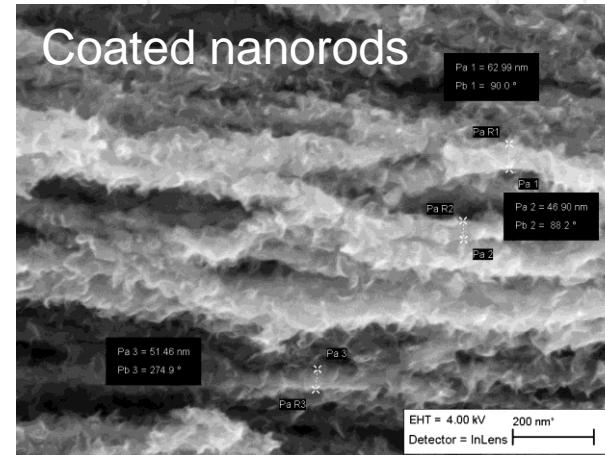


HR-SEM surface and cross-sectional view of c-Si

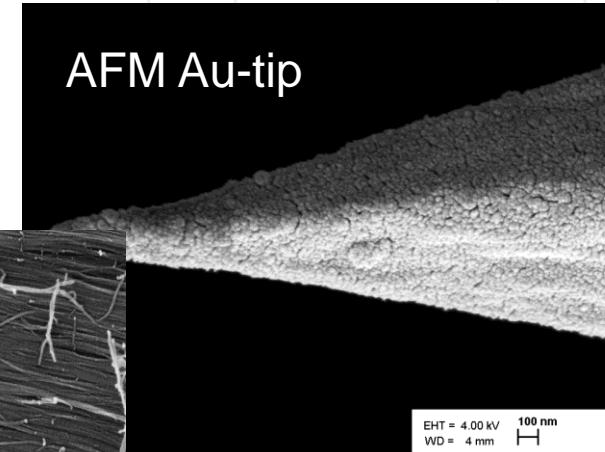
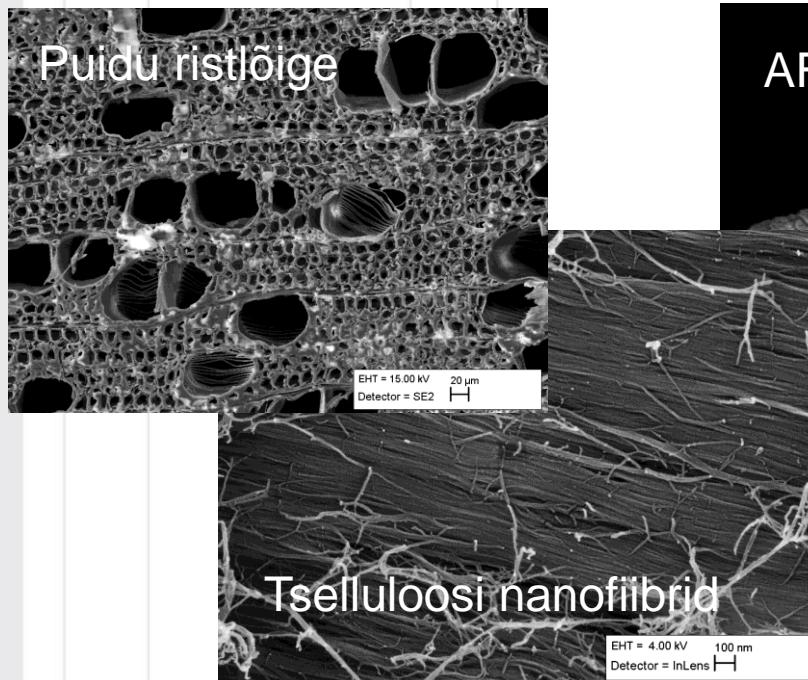


HR-SEM surface and cross-sectional view of glass/AZO/a-Si structure

Teaduskoostöö teiste uurimisgruppidega tellimustööde raames



Advanced EDS Detection





Koostöö erinevate ettevõtetega lepingute raames

ENICS Eesti AS, elektroonikakomponentide tootja

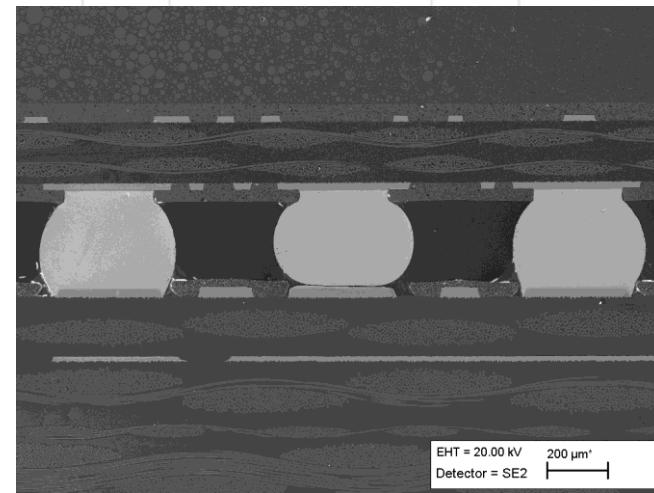
Nende peamiseks probleemiks on uute tarnijate elektronika-komponentide mittesobivus standardses tootmis-protsessis- kvaliteediprobleemid.

Teiseks probleemiks on valmistoote kvaliteedikontroll ning valmistoote mittevastavus eeldatavatele tulemus-tele.

Meie ülesandeks on põhjuste väljaselgitamisele kaasa-aitamine ning soovitused toote parandamiseks.

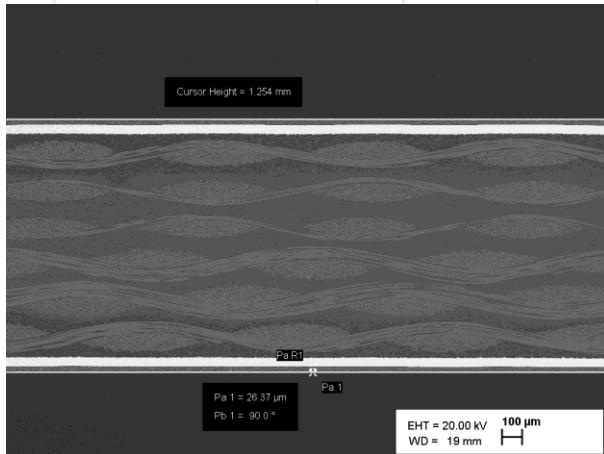


Tinutuskvaliteet- nanopoorid kihtide vahel

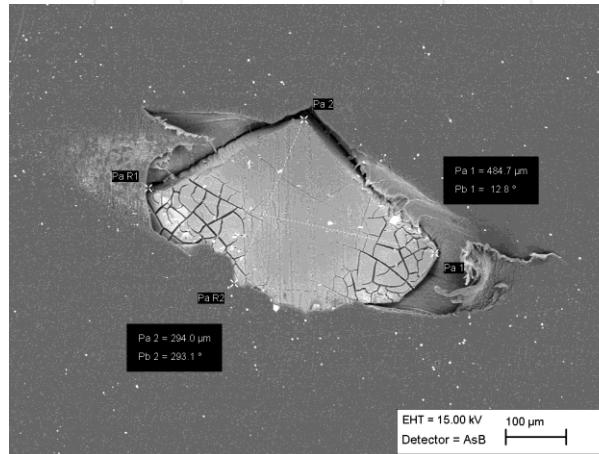


Mikrokiibi jalg defektiga

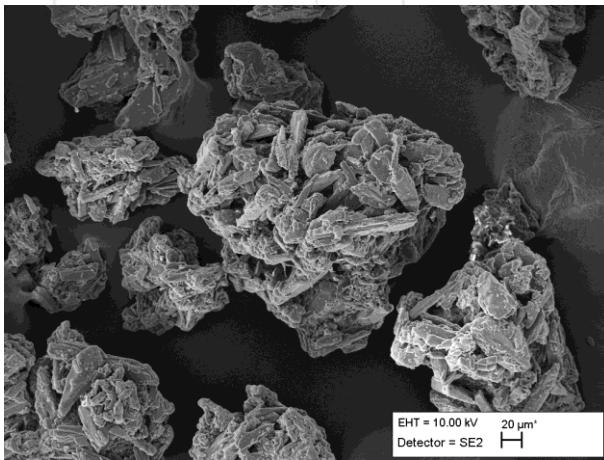
Koostöö erinevate ettevõtetega tellimustööde raames



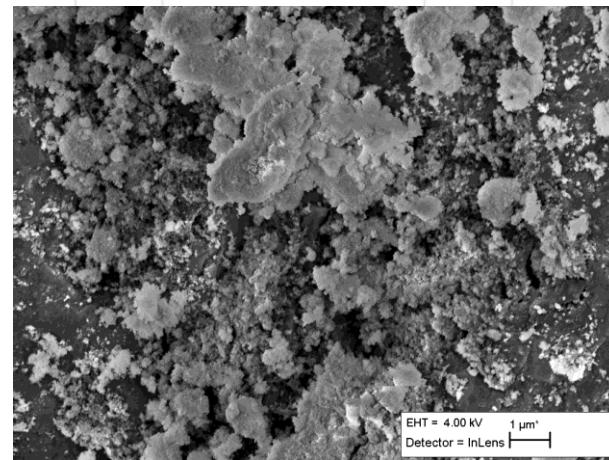
Trükkplaat, Stoneridge Electronics AS



Defekt plastik pudelis, Greiner Packaging AS



Ravimitööstus- paracetamol seotud maatriksis



Al-Ce powder, Silmet AS

Surface Acoustic Waves (SAW) for molecular interactions studies

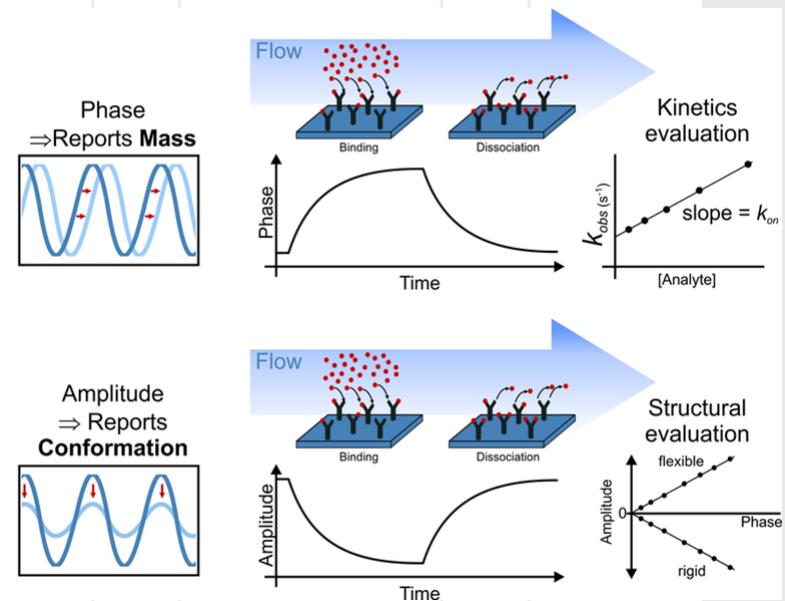


SAW chips with 8 sensing elements



Kvartskristallide mikrokaalude automaatsüsteem SamX, SAW Instruments GmbH (now part of NanoTemper Technologies GmbH.)

The Surface Acoustic Wave technology (**SAW**) allows for an in-depth analysis of molecular interactions in real time. Binding kinetics can be precisely determined by detecting mass and binding-induced conformational changes.



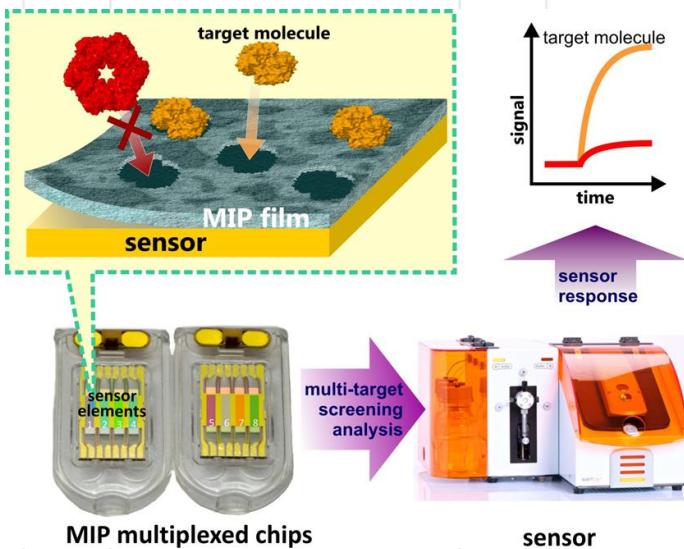
Development of chemo- and biosensors based on synthetic receptors



Kasutust leiab peamiselt teadustöös, mis on suunatud molekulaarse jälgendamise tehnoloogia abil biotundlike funktsionaalsete materjalide väljatöötamisele, mis on võimelised selektiivselt kinni püüdma ja määrama nii väikeseid (aminohapped, erinevad ravimijäandid jt.) kui ka biomakromolekule (eeskätt valgumolekule nagu antikehi ja neurotroopseid ühendeid).

Molekulaarselt jälgendatud polümeerid (MJP) integreeritakse erinevate sensorplatvormidega, mis võimaldavad määrata analüüdi sihtmolekule märgistamiseta ja kõrge tundlikkusega. Eeskätt on erinevatel sensorplatvormidel valmistatud MJP materjalide eelisteks töökindlus, omaduste stabiilsus ja odavam ning lihtsam süntees.

MJP on perspektiivsed materjalid biosensorite valmistamiseks, olles alternatiiviks bioloogilistele retseptoritele, kemosensorite valmistamiseks kliinilise diagnostika ja keskkonna analüütika tarbeks ja spetsiifiliste ravimikandjate loomiseks.



Surface Acoustic Waves (SAW) for study molecular interactions

Koostöö teiste TA asutuste ja ettevõteteega (sh välisriikidest)

- **Icosagen AS** (Eesti): MJP sünteetiliste retseptorite väljatöötamine ja uurimine spetsifiliste antikehade ja neurotroopsete faktorite selektiivseks tuvastamiseks.
- **University of Helsinki**, Prof. M. Saarma's group (Soome): MJP sünteetiliste retseptorite väljatöötamine neurotroopsete faktorite selektiivseks tuvastamiseks.
- **University of Helsinki**, Prof. Risto Kostiainen's group (Soome): MJPide integreerimine mikrofluidik kiipidele proovide valmistamiseks ja puastamiseks (Integration of MIPs with microfluidic chips for sample preparation and purification).
- **AirDetect Oy** (Soome): Moodul-sensori väljatöötamine ruumide siseõhu kvaliteedi jälgimiseks. (Development of a modular sensor system for indoor air quality monitoring).
- **Institute of Physical Chemistry**, Prof. W. Kutner's and Prof. K. Noworyta's group (Poola): Eesti Teaduste Akadeemia ja Poola Teaduste Akadeemia teaduskoostööprojekt "Imprinted polymers, integrated with acoustic sensors, for cancer biomarker determination".

Olulisemad publikatsioonid:

- Ayankojo, A. G., Tretjakov, A., Reut, J., Boroznjak, R., Öpik, A., Rappich, J., Furchner, A., Hinrichs, K. and Syritski, V. **Molecularly Imprinted Polymer Integrated with a Surface Acoustic Wave Technique for Detection of Sulfamethizole**, *Analytical Chemistry* (2016), 88 (2), 1476-1484.
- Tretjakov, A., Syritski, V., Reut, J., Boroznjak, R. & Öpik, A. **Molecularly imprinted polymer film interfaced with Surface Acoustic Wave technology as a sensing platform for label-free protein detection**, (2016) *Analytica Chimica Acta*, 902, 182-188.

Dr. Vitali Syritski vitali.syritski@ttu.ee

Pulsed laser deposition (PLD) Neocera Pioneer 120



Key Features

- Max. size of substrate: one 2 inches or multiple small substrates.
- Max. substrate temperature: 900 °C.
- Operating pressure range: 5×10^{-7} Torr to 500 mTorr.
- Target Size: six 1 or three 2-inch diameter targets.
- Laser: 248 nm UV excimer laser, pulse energy till 400 mJ, max. repetition rate – 20 Hz, average power 6 W, pulse duration – 20 ns.

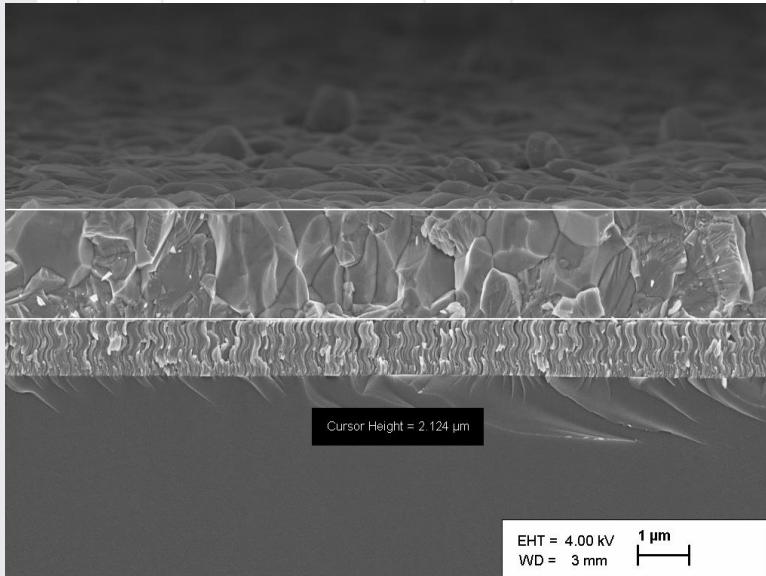
PLD method gives possibilities to deposit high quality layers of sublimable and non-sublimable materials (ceramics etc.) onto rotated and heated substrates in high vacuum and in controllable process gas atmosphere (N_2 or O_2).

Method is not effective for evaporation of metals (Pt, Mo, etc.) due to reflection of laser beam.

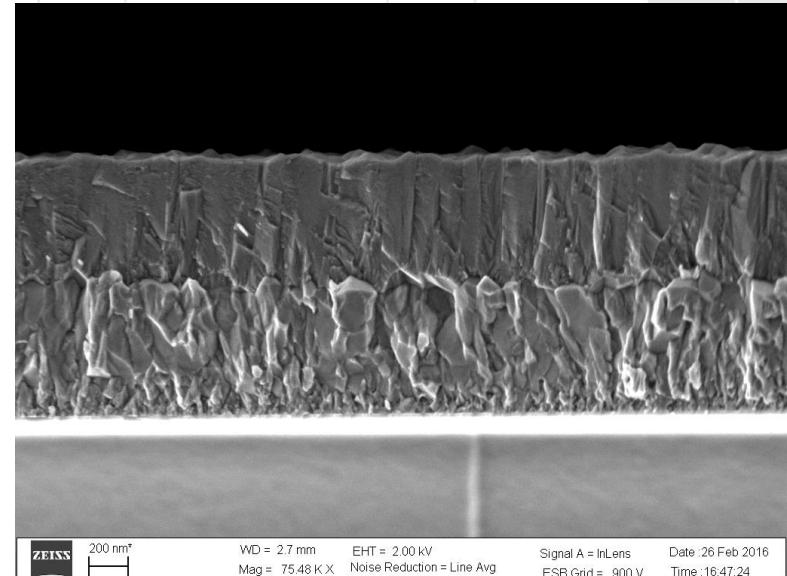
Dr. Sergei Bereznev sergei.bereznev@tu.ee



Structures prepared by PLD – examples.



HR-SEM cross-sectional view of
glass/Mo/CZTS structure prepared by PLD
at 400 °C.

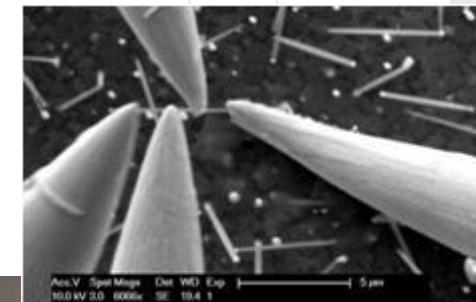


HR-SEM cross-sectional view of
glass/FTO/Zn(O,S) structure prepared by
PLD at 300 °C.

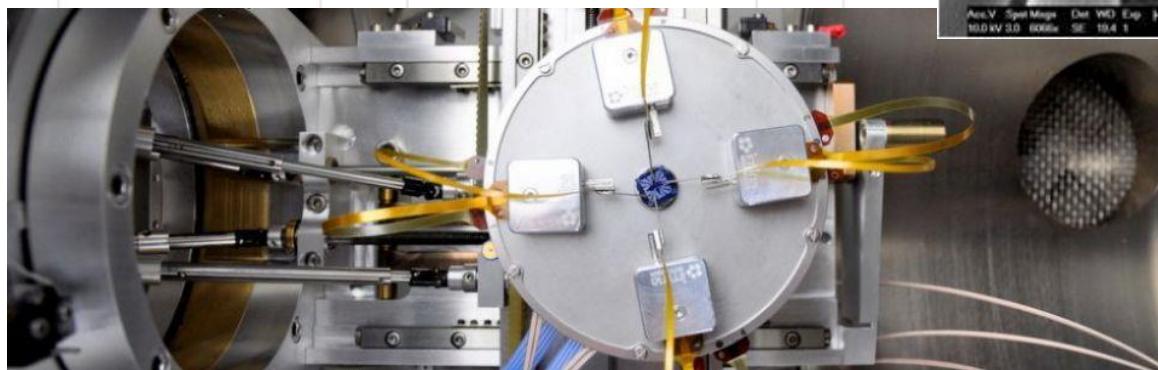
NAMUR+ raames planeeritavad seadmed



ALD seadme põhiliseks rakenduseks TTÜs on erinevate päikesepatarei struktuuri vahekihtide sadestamine (puhverkikhid, passiveerivad kihid, kaitsekihid). Põhiliselt erinevad metalli sulfiid ja metalli oksisulfiid kiled kontrollitud paksusega nanomeeter-skaalas.



Mikro/nanomanipulaatorid HR-SEM kompleksile





Tänan tähelepanu eest!

