Plasma and Surface Technologies for Energy Applications & Sustainability

Stefan Saager

Manufacturing World 2024, Tokyo

Fraunhofer Society – Bridging basic research and industrial development

Applied Research in Germany

world's leading applied research organization, founded in 1949

76 institutes and research institutions

approx. 32 000 employees

headquarter in Munich

each institute has its own core competences and act as individual profit center on the market

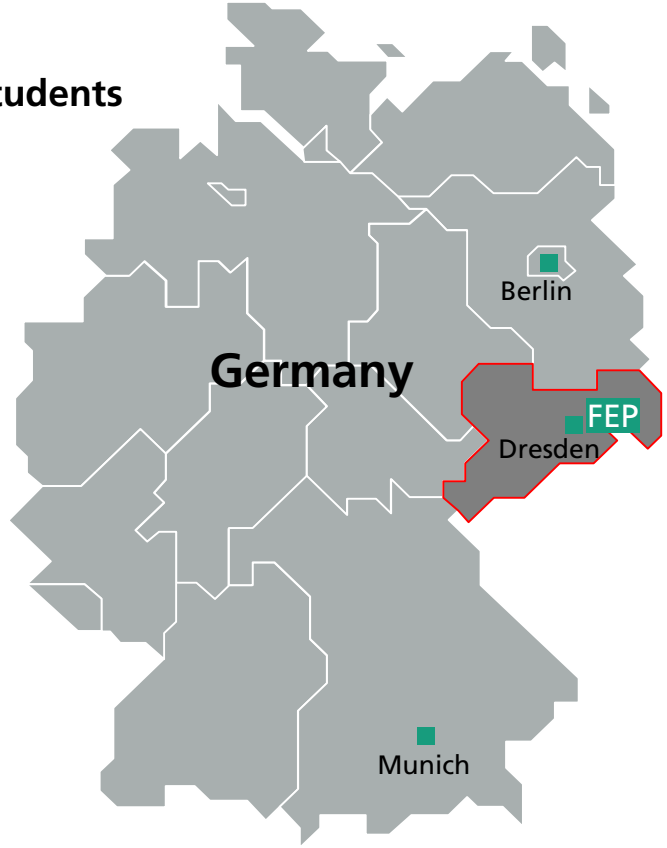
Fraunhofer FEP

Fraunhofer Institute for Electron Beam and Plasma Technology FEP

The Institute in Figures

Employees	**196 incl. students**
Total budget	**30.7 M€**
Industry returns	**11.6 M€**
Public funding	**6.2 M€**
Investments	**2.1 M€**

(April 2024 | Figures from 2023)



Director

Prof. Dr. Elizabeth von Hauff

Core competences

Electron Beam Technologies

Plasma Surface Technologies

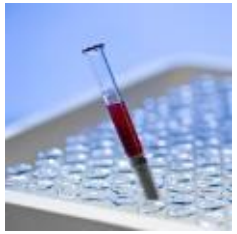
Sputter Technologies

Technological Key Components

Fraunhofer FEP

Industry Solutions

Energy and Sustainability



Life Sciences

Environmental Technologies

Smart Building

Mobility

Optic, Sensorics and Electronics

Smart Farming

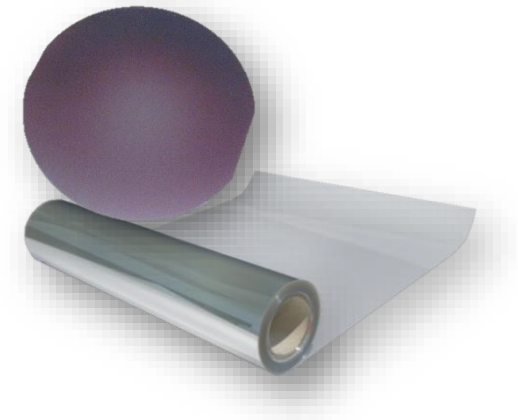
Packaging

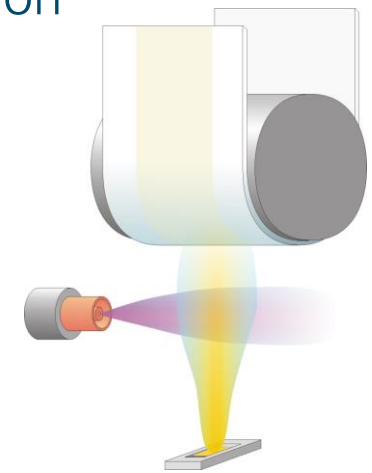
Mechanical Engineering

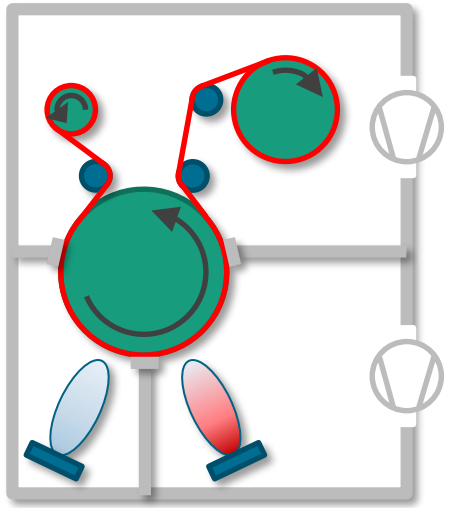
Cultural Heritage and Preservation

Fraunhofer FEP support for product development

From feasibility studies to pilot production



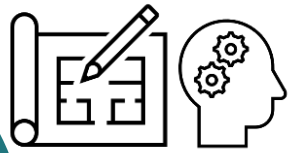




testing substrate materials

developing coating technologies

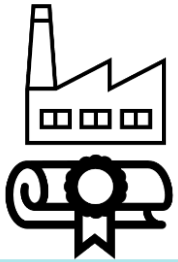
developing key components



prototyping

pilot production

technology transfer & licensing



application

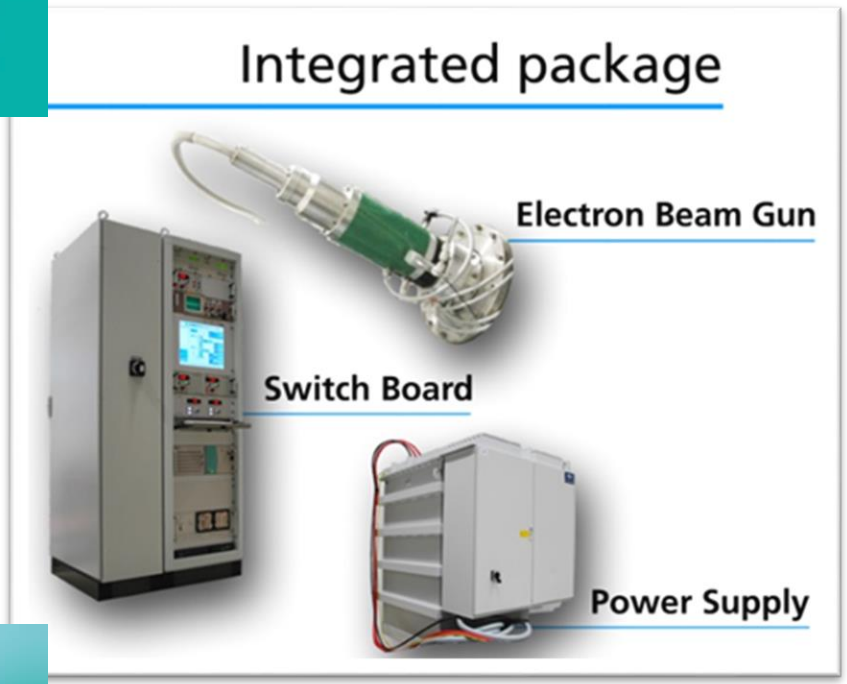
Fraunhofer FEP

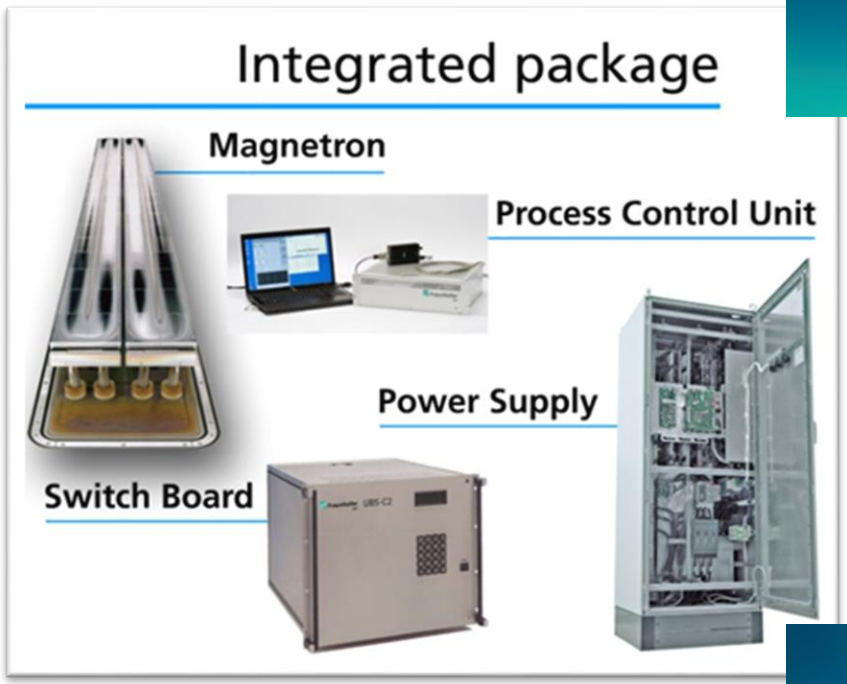
Key components

Integrated Packages

Mechanical Components

Technology





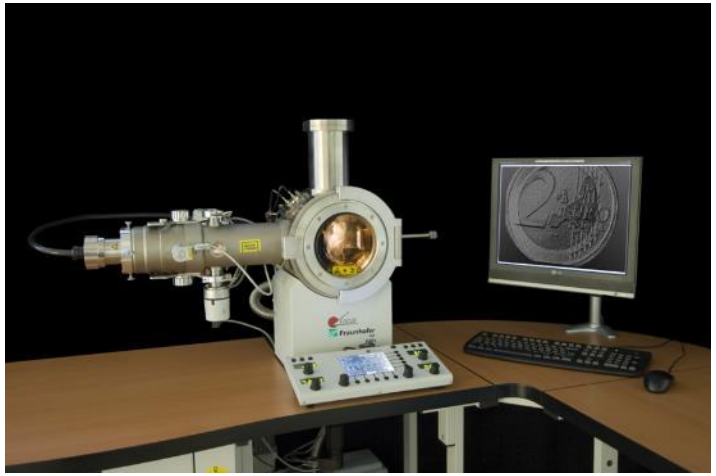
Electronical Components

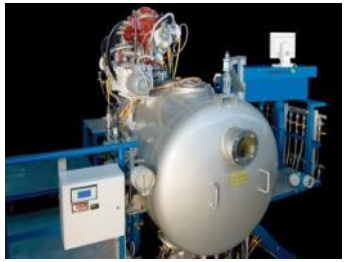
Control / Visualization

Fraunhofer FEP

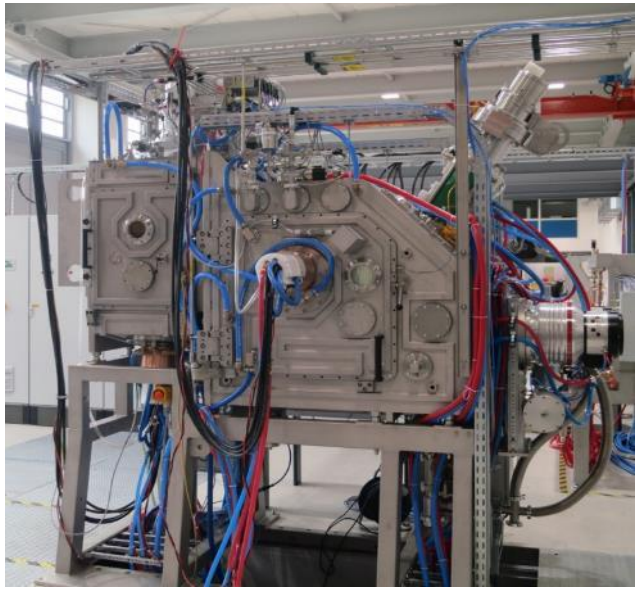
Platforms for Developing Coating Technologies

Lab-Scale Infrastructure





Process Development & Feasibility Studies



Upscaling & Pilot Production

Fraunhofer FEP

Fraunhofer FEP process technologies

Large area, in-line configuration

Surface treatment
(plasma, ion, bias, electron beam, flash lamp annealing)

High-rate evaporation
(boat, inductive heating, electron beam, plasma assisted)

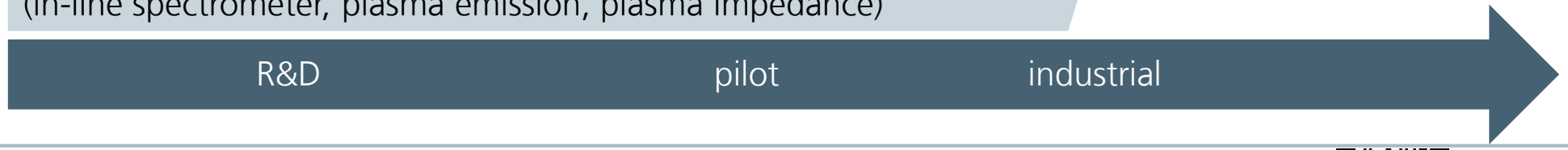
Magnetron sputtering
(DC, pulsed, hybrid, dual magnetron, reactive & non-reactive)

High-rate PECVD
(magPECVD, arcPECVD)

Non-vacuum processes
(slot die, electron beam)

Gas Phase Condensation
for nanoparticle generation

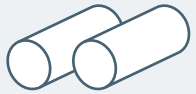
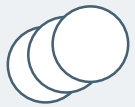
Process control
(in-line spectrometer, plasma emission, plasma impedance)



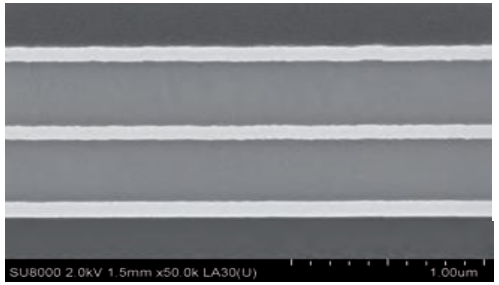
Fraunhofer FEP

Substrate Form Factors at Fraunhofer FEP

Process development on different substrate geometries and surfaces

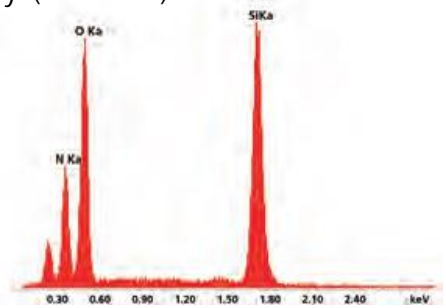
		Method	max. substrate size	Material						Thickness
				Metals	Polymers	Flexible glass				
Roll-to-roll		Sputtering Electron beam evaporation	300 – 600 mm width							7 µm … 200 µm
Sheet-to-sheet		Inline sputtering (planar, tubular) magPECVD	600 x 1200 mm² 650 x 750 mm²				Ceramics	Glass		50 µm … 6 mm 120 mm
Sheets and strips		Electron beam evaporation	500 x 500 mm² 300 mm width							15µm …1.5 mm
3D objects		Sputter deposition PECVD	500 x 500 x 500 mm³							-
Wafer		Sputter deposition (high rate)	Up to 300 mm diameter						Si	max. 20 mm

Materials Characterization



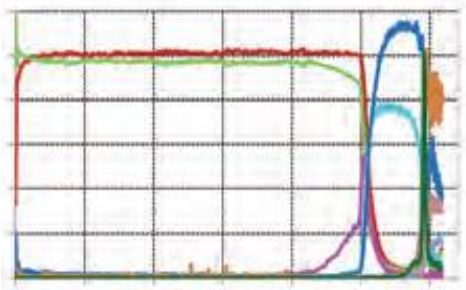
Microstructure

- Ultra-high resolution scanning electron microscopy (FE-SEM)
- Ion preparation of cross sections
- Metallography



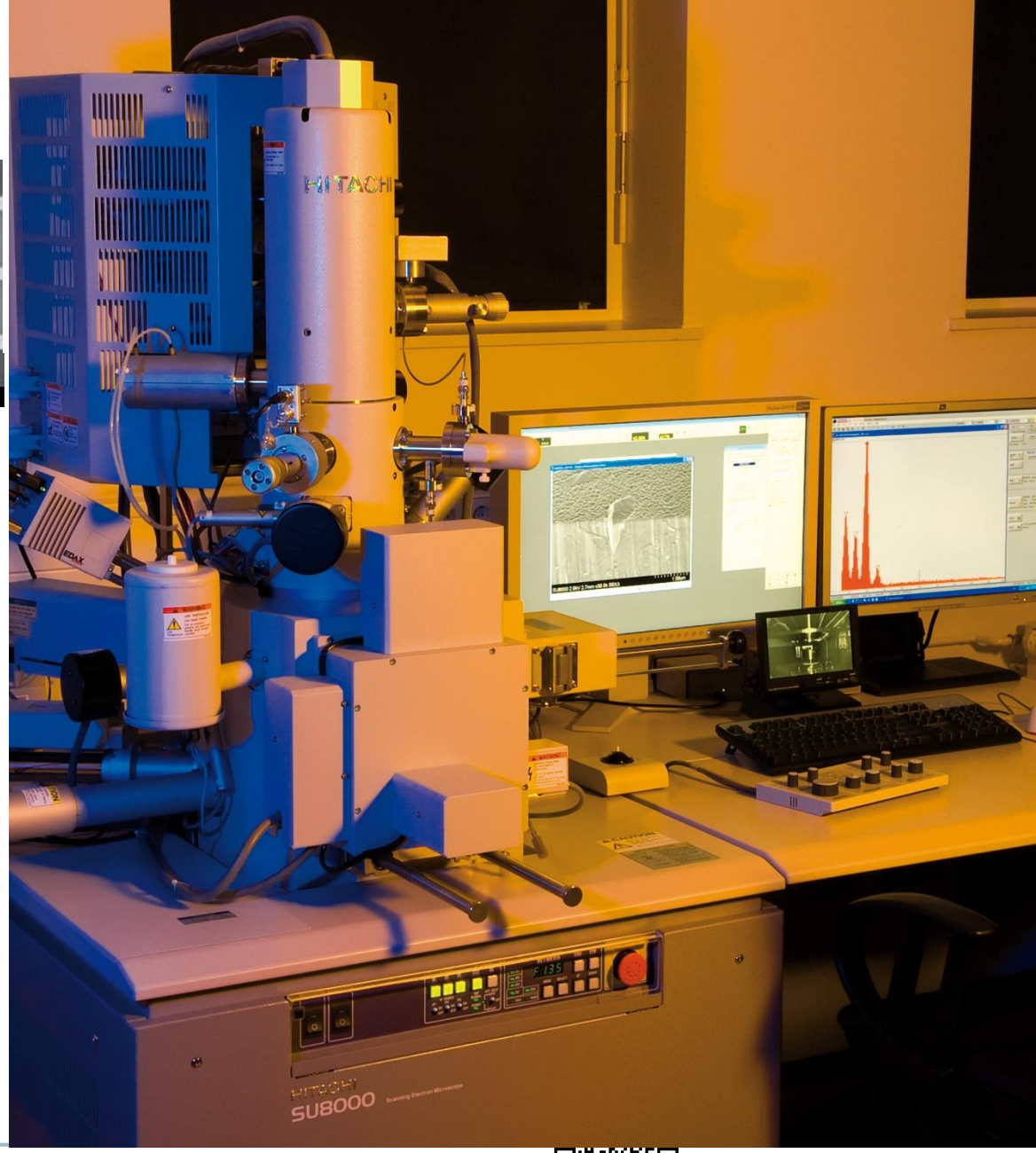
Chemical composition and topography

- Glow discharge optical emission spectrometry (GD-OES)
- Energy dispersive spectrometry (EDS)
- Atomic force microscopy (AFM)



Layer properties

- Permeation measurement (WVTR, OTR)
- Measurement of hardness (nanoindentation technique)
- Optical properties (spectroscopic ellipsometry)



https://www.fep.fraunhofer.de/en/Kernkompetenzen/analytik.html

Fraunhofer FEP

Selected project and application examples

- Coatings for emerging & sustainable technologies
- Technologies for life science applications

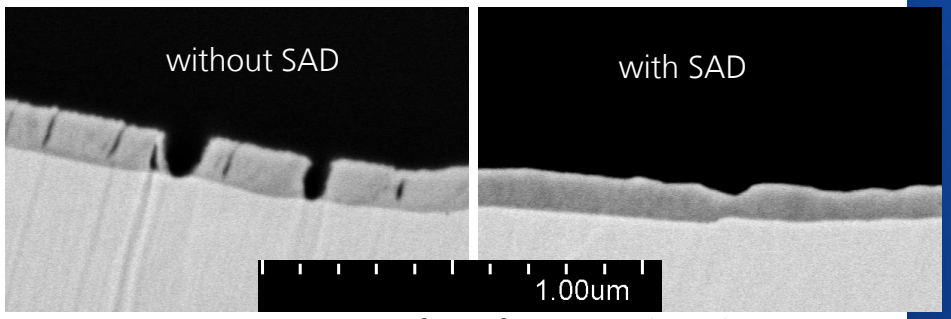
Fraunhofer FEP

USE CASE: Deformable protective coatings for PEM BPP by R2R

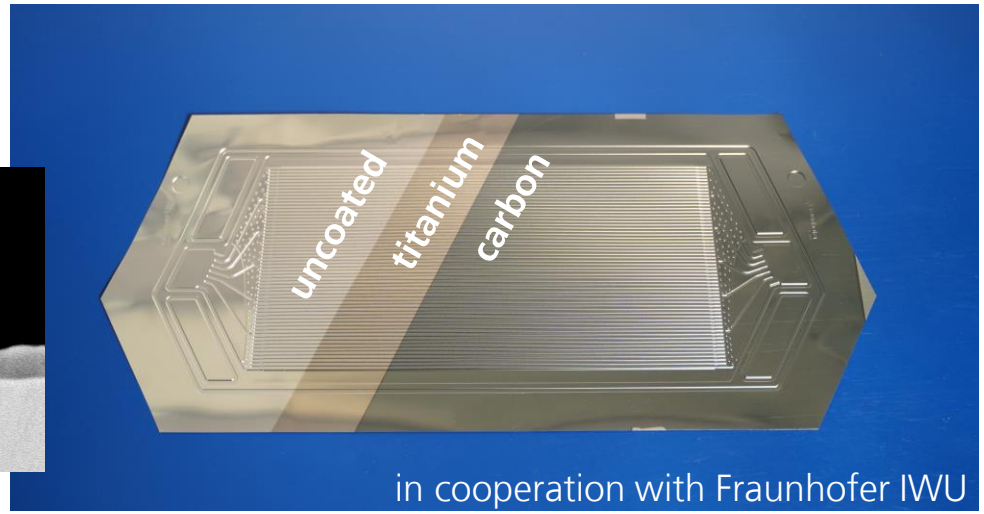
By high-rate plasma-activated EB evaporation

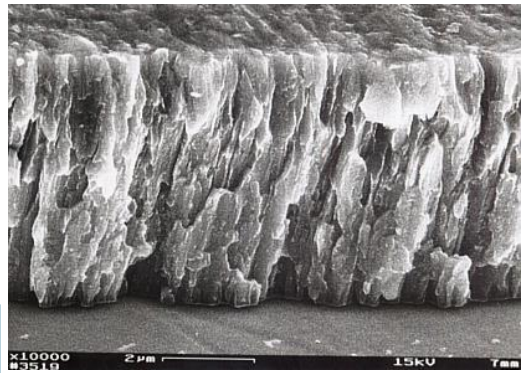
Demands	■ Low contact resistance ■ High resistance to corrosion in a wide oxidizing potential range ■ Good formability without layer cracking ■ Low cost processing
Technology	■ High-power plasma processing + High-rate EB evaporation ■ Roll-to-Roll vacuum processing with thin metal strips (pre-coating)

- EBPVD: high throughput technology
- Increased ductility and formability by plasma-activated evaporation (SAD)

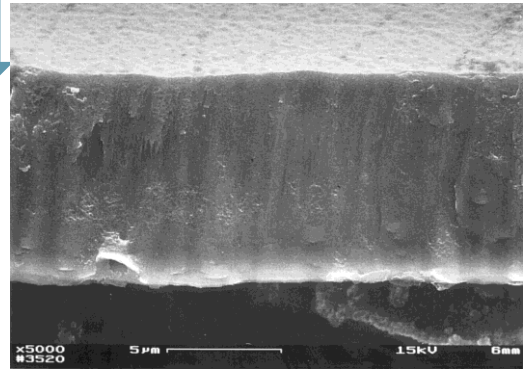


cross-sections of Ti after 180° bending test





Plasma effect on the microstructure of thin titanium layers



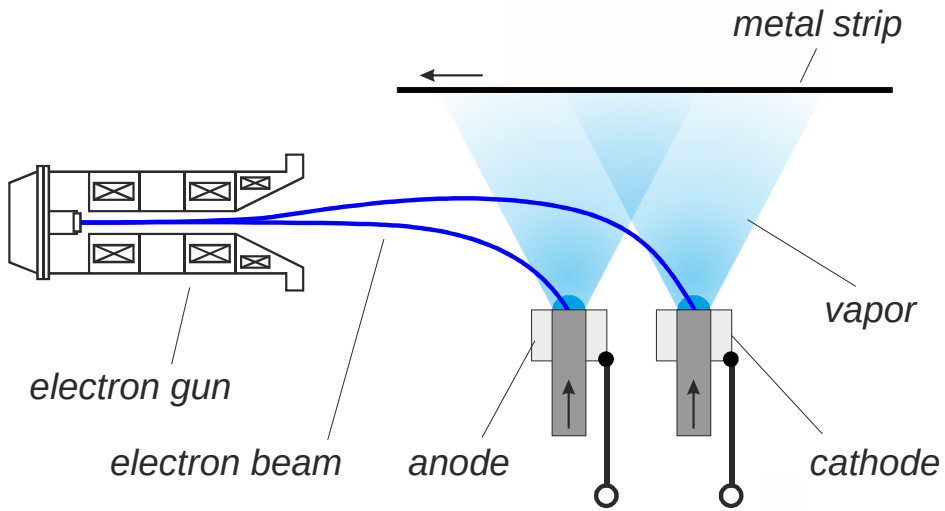
Scheffel et al., Surf. Coat. Technol. 287, 138 - 144 (2016)

PEM – Proton Exchange Membrane
BPP – Bipolar Plate
R2R – Roll-to-Roll
SAD – Spotless-arc Activated Deposition

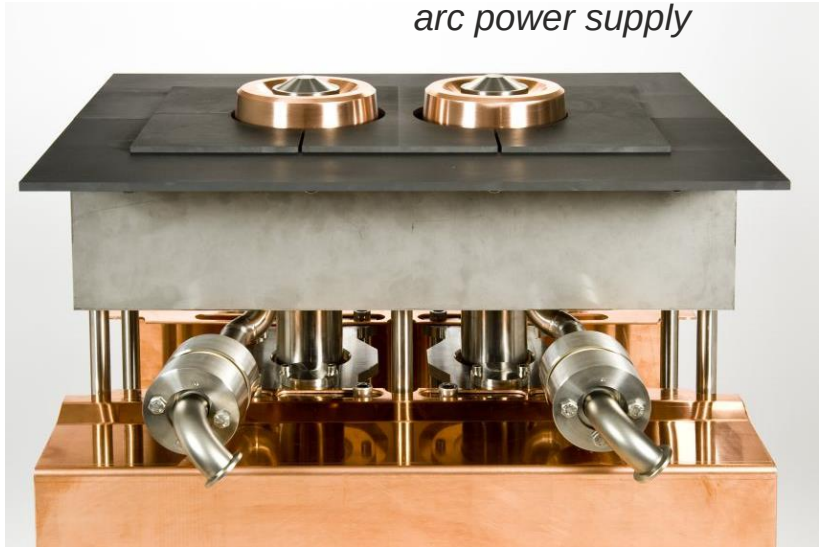
Fraunhofer FEP

EQUIPMENT: Device for Spotless arc Activated Deposition (SAD)

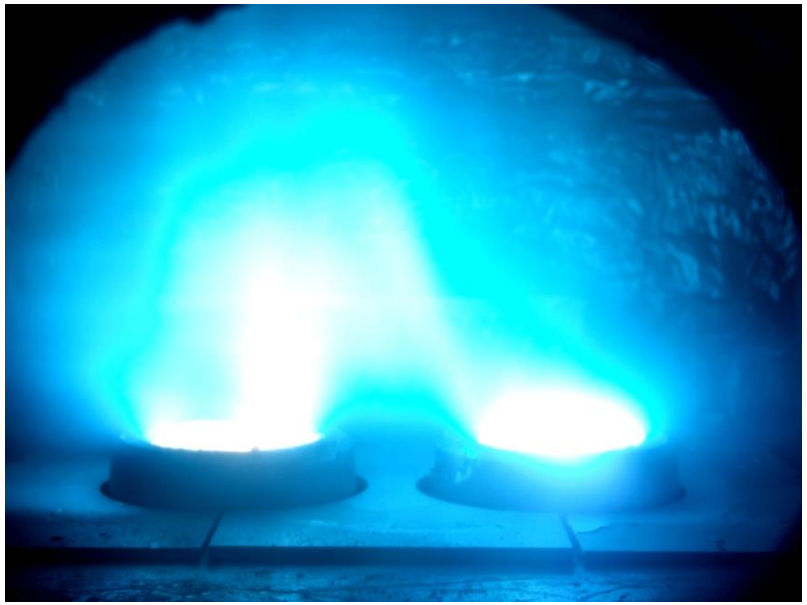
Using dual crucible



- Spotless arc discharge on two evaporating electrodes
- Cathode: high-melting metal, anode: same or other metal
- Plasma generation at cathode and anode
- Reactive processing possible
- Electrodes keep free by evaporation
- No layer deposition at any solid anode
- Optional DC / AC



Water-cooled dual crucible



spotless arc using dual crucible (2 x Ti)

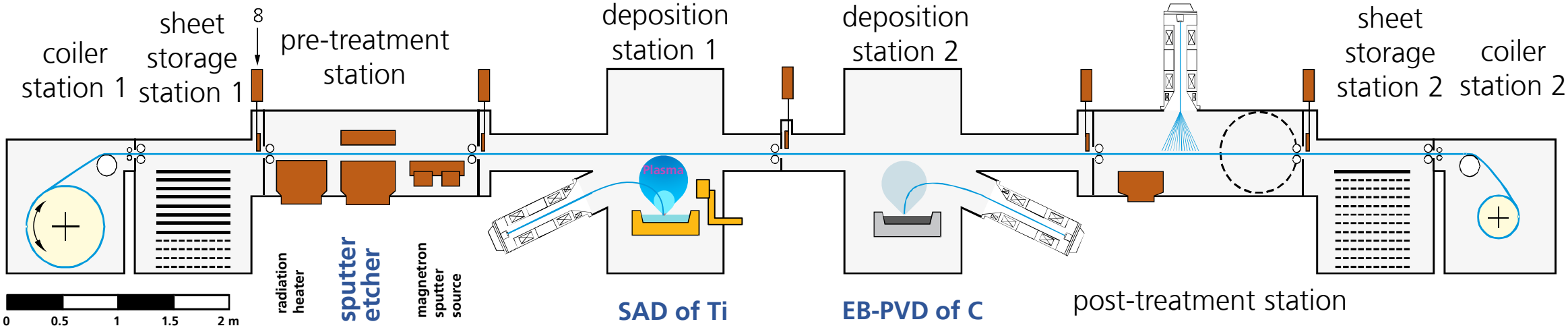
EB evaporation of Ti from 2 crucibles by jumping beam

PLATFORM: In-line electron beam and sputter Coater MAXI

In-line vacuum coating equipment for metal strips and sheets

Features

- Single and double side coating of metal, plastic, glass or ceramic sheets with a maximum dimension of 500 mm × 500 mm
- Coating of metal strips with a maximum width of 300 mm and a maximum thickness of 1.5 mm
- Testing of key components under near-industrial conditions

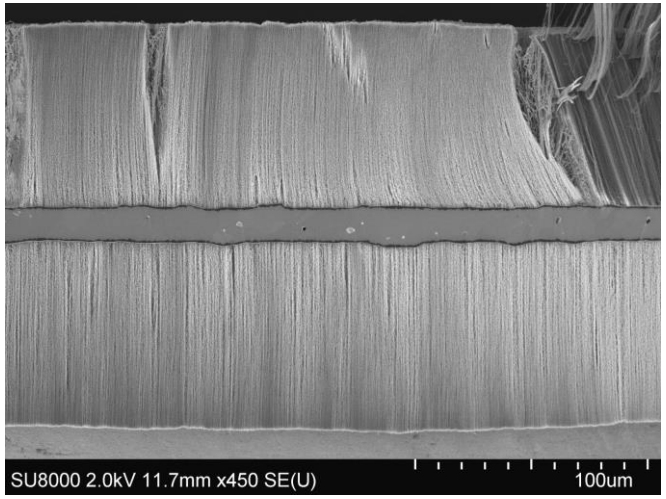


https://www.fep.fraunhofer.de/en/Kernkompetenzen/Anlagentechnik/maxi.html

USE CASE: Deposition of Carbon Nano Tubes (CNTs)

By chemical vapor deposition (CVD)

Process	■ Double side coating of a metal strip with catalyst ■ Heating by thermal radiation ■ Addition of precursor gas ➔ CVD of CNTs
Reached results	■ Double side deposition of CNTs in an inline mode ■ Various substrate materials usable



Cross section of CNTSs deposited (partially damaged by preparation)

Top side CNTs ~80 µm

Metal foil 13 µm

Bottom side CNTs ~80 µm

It's the blackest material I have ever seen!

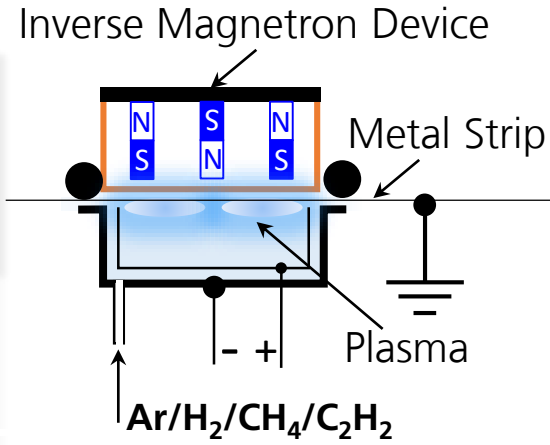
Fraunhofer FEP Annual Report 2022/23 (Page 17)

Fraunhofer FEP

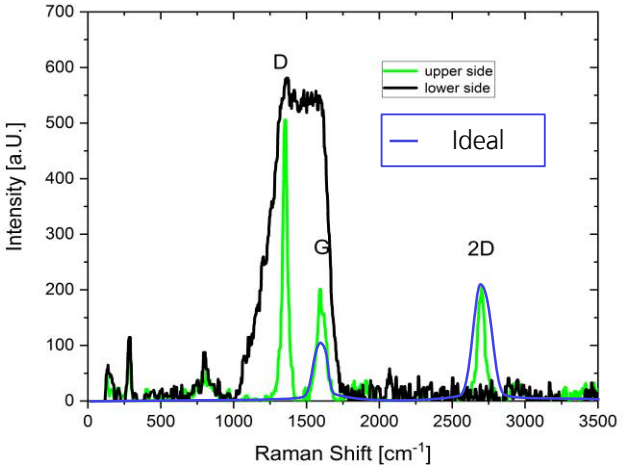
USE CASE: Deposition of Graphene Layers

By plasma-enhanced chemical vapor deposition in a roll-to-roll process

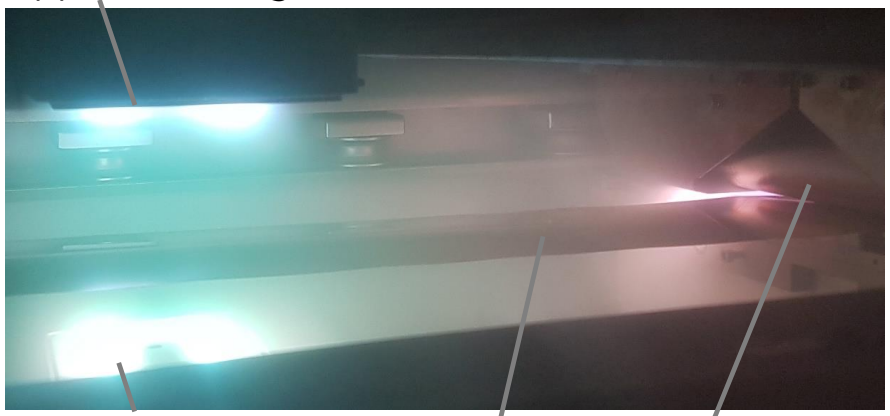
Process	■ Double side coating of a steel strip with catalyst ■ Rapid heating up to 1000°C by plasma device ■ Addition of precursor gas ➔ deposition of graphene by PE-CVD
Reached results	■ Formation of graphene has been proven by Raman ■ Lower temperatures (650 °C) & higher throughputs (17m²/h) feasible ■ Quality of the layers has a high need for optimization



processed pieces of steel strip



Raman spectrum



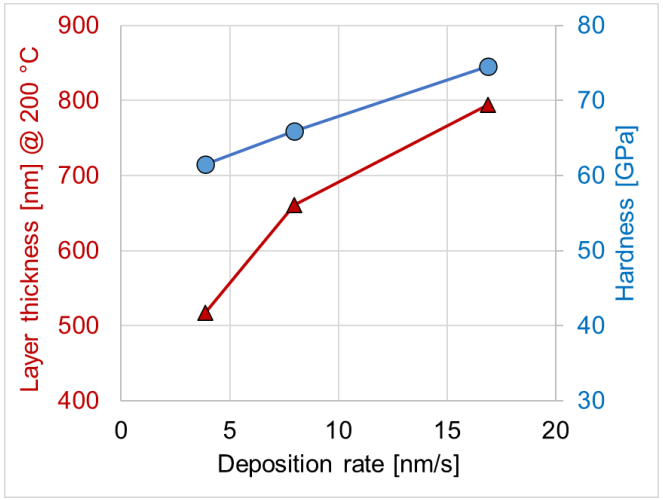
"Plasma-Enhanced Chemical Vapor Deposition of Graphene Layers by a Roll-to-Roll Process", 2022 Technical Conference Proceedings of SVC https://doi.org/10.14332/svc22.proc.0026

Fraunhofer FEP

USE CASE: Alternative protective coatings for PEM/electrolyser BPP

Performance-adjusted carbon coatings by anodic arc evaporation

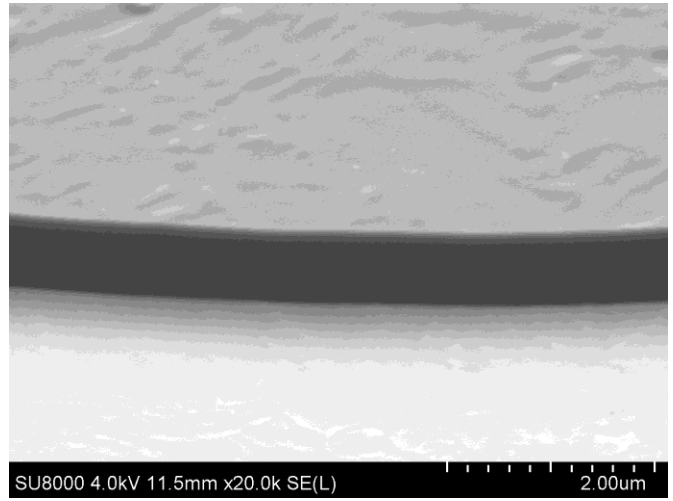
Challenge	■ High cell voltage and high anode potential → corrosion sensitivity ■ Demands for high corrosion resistance ■ low contact resistance (target ≤ 0.01 Ω cm²)
Approach	■ Thin doped ta-C layers (extremely corrosion-resistant and electrically conducting) ■ New plasma PVD method by anodic arc evaporation



Further use cases: Leveraging the high hardness for tribological applications

ta-C coatings (tetrahedral amorphous carbon)

- Hydrogen-free carbon coatings
- Dominating sp^3 content
- Doping for enhanced electrical conductivity
- Hollow cathode arc source + Anodic arc evaporation
- Sublimation of graphite
- High ionization degree of vapor
- Motor driven material feed
- Relative high deposition rate **(≈ 17 nm/s = 61 µm/h)**
- Low droplet emission mode



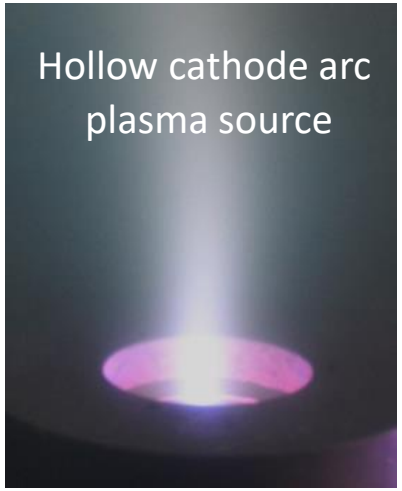
"Diamond-like films of tetrahedral amorphous carbon deposited by anodic arc evaporation of graphite", Surf. Coat. Tech., 477 (2024) 130305
https://doi.org/10.1016/j.surfcoat.2023.130305

Fraunhofer FEP

EQUIPMENT: Anodic Arc Evaporator

Features

- Plasma activated deposition
- High ionization degree (up to 100%)
- Relative low particle energies (compared to sputtering)
- High deposition rate
- Low droplet emission

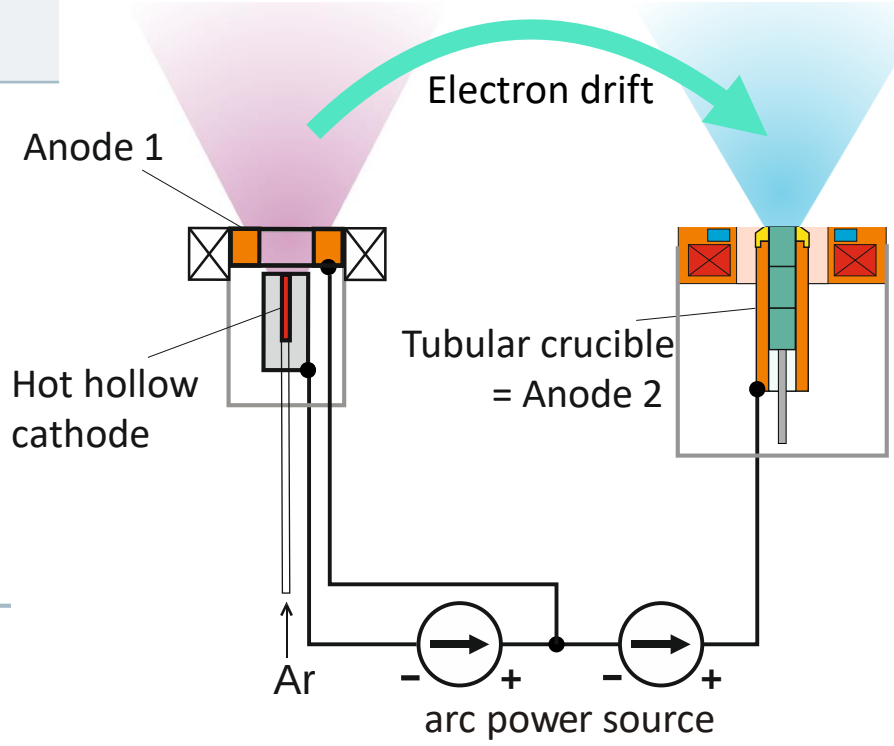


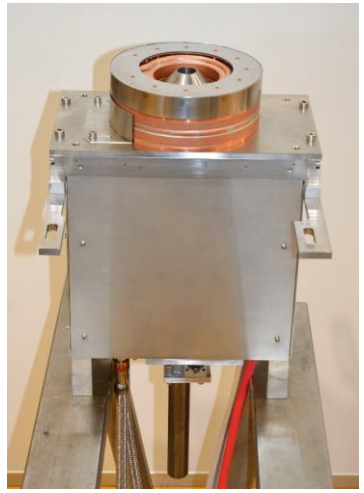
Evaporation of Indium-Tin-Oxide (ITO)

Transparent conductive oxides (TCO)

- Successfully with ITO, IZO, AZO
- Crystalline ITO layers at low temperature
- Excellent electrical and optical properties
- Extremely low roughness
  Ra = 0,3 nm at ITO 300 nm thick
- Dep. rate 300 - 500 nm m/min

Hollow cathode arc plasma source LAVOPLAS
Large volume plasma source, 50 - 200 A





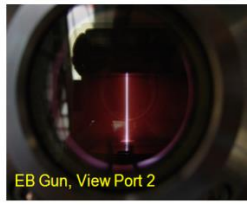
Anodic arc evaporator
0 - 200 A,
max. 20 kW
Motor-driven evaporant feeding

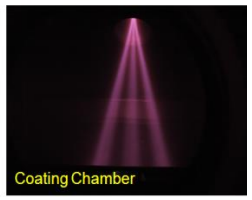
Fraunhofer FEP

USE CASE: Electron Beam and Plasma Technology for EB-PVD

Equipment for advanced thermal barrier coating technologies for turbine components

Objectives	Application of enhanced thermal barrier coatings on turbine components for increased performance: higher operation temperature, engine efficiency, reliability and lifetime. -> Enabling technology for expanded process parameter windows
Technology	■ High-performance Electron Beam System ERIC-LVO ■ Plasma-activated evaporation to adjust microstructure at exotic process conditions

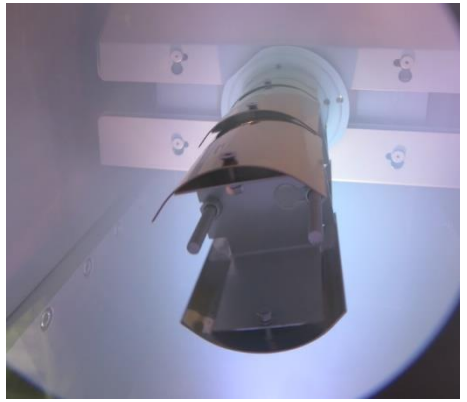




Fraunhofer FEP

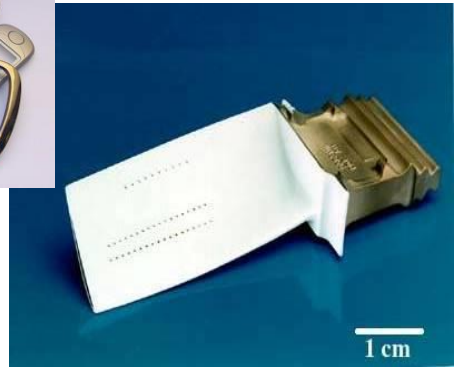
PLATFORM: Electron Beam Coating System NOVELLA

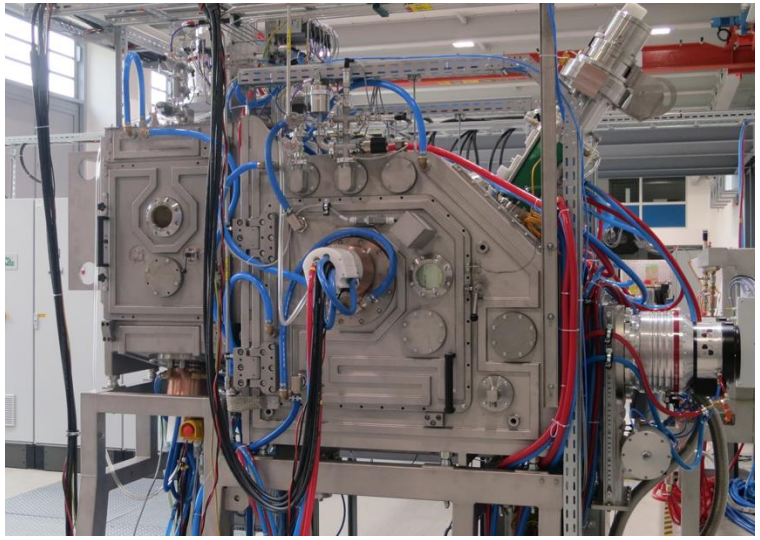
For processing of 3D parts

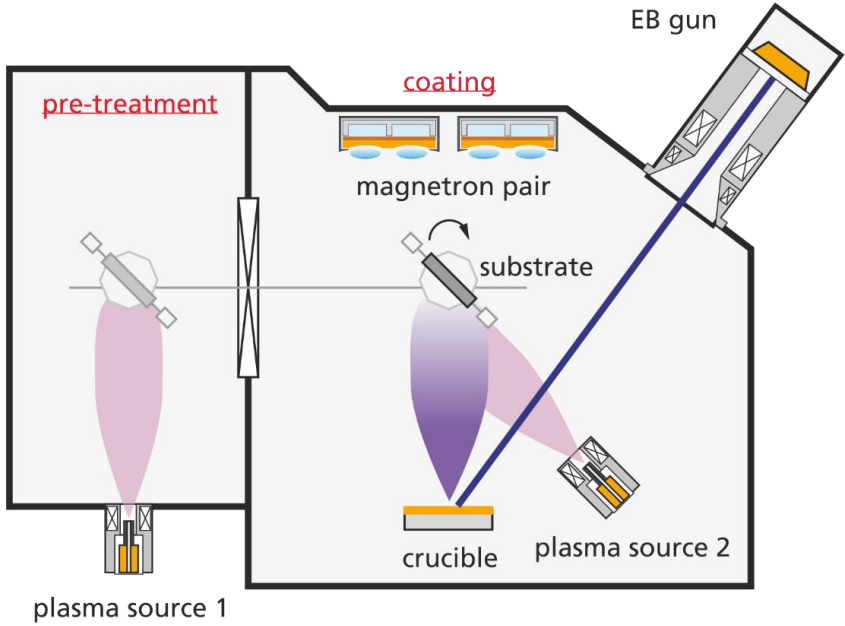


Features

- short-cycle plant with load-lock chamber
- plasma-based electron gun (40 kV, 120 kW)
- hollow cathode plasma sources (pre-treatment, plasma activation)
- magnetron sputter sources (optional)
- programmable substrate movement (translation + rotation)







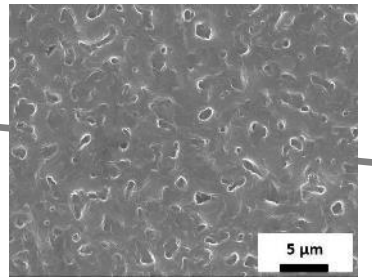
https://www.fep.fraunhofer.de/content/dam/fep/en/documents/Anlagenflyer/H14_NOVELLA_EN_net.pdf

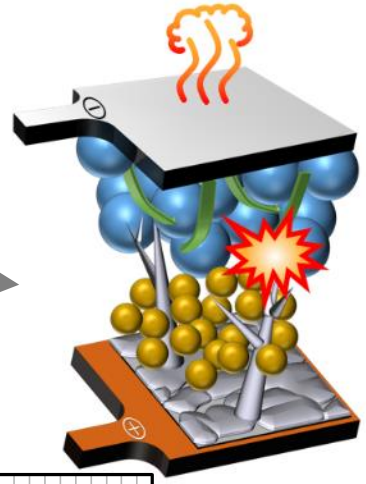
USE CASE: Silicon-based Next Generation Anodes for Lithium-Ion Batteries

low-cost, high-performance batteries for electromobility and the energy transition

Lithium metal anodes & prelithiation

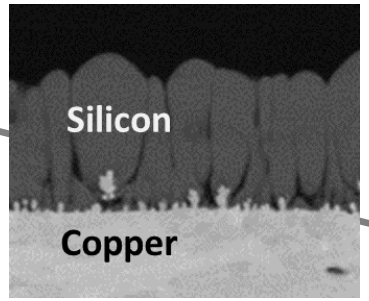
- by deposition of porous lithium layers
- by coating of electrodes

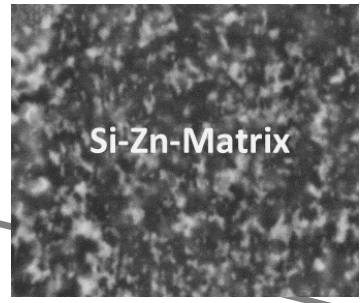


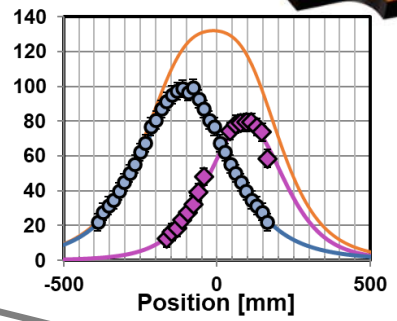


Porous silicon anodes

- by deposition of nodular silicon structures
- by vapor phase dealloying
- by deposition of pre-**lithiated silicon layers**

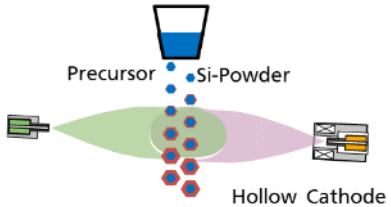






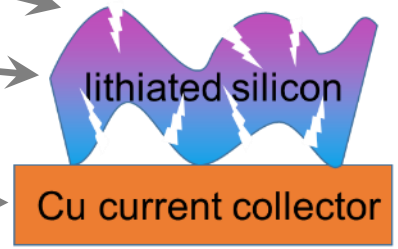
Silicon-carbon composite anodes

- by **functionalization of silicon particles**



Lightweight current collector

- by metallization of polymer films



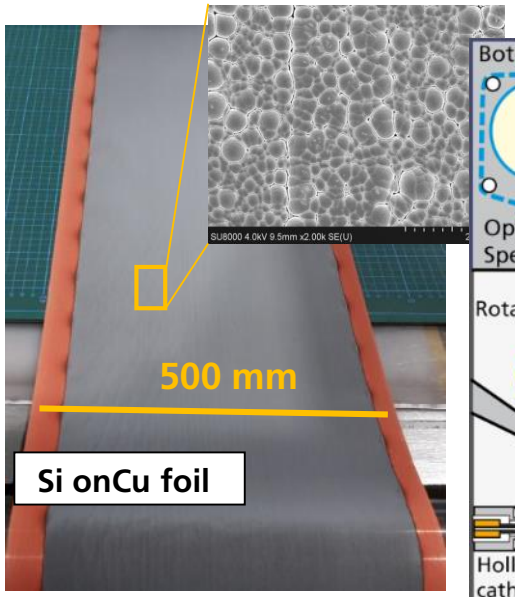
Piwko et al., J. Power Sources, 351 (**2017**) 183-191
Saager et al., Surf. Coat. Technol., 358 (**2019**) 586-593
Saager et al., Batteries, 9(2):75 (**2023**)

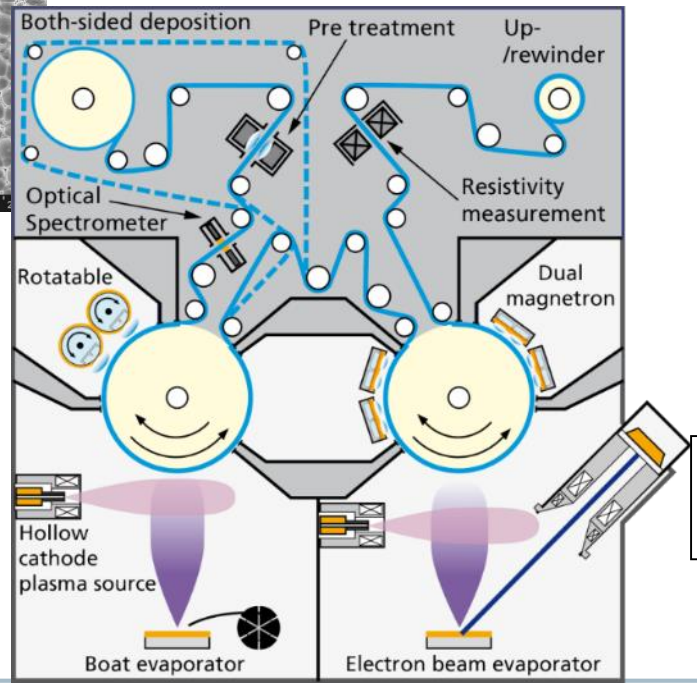
PLATFORM:

Pilot R2R Coater *novoFlex® 600* Pilot

Features

- Polymer films, metal foils up to 600 mm width
- Plasma assisted EBPVD, Sputtering, PECVD
- Inline pre-treatment, optical/electrical metrology

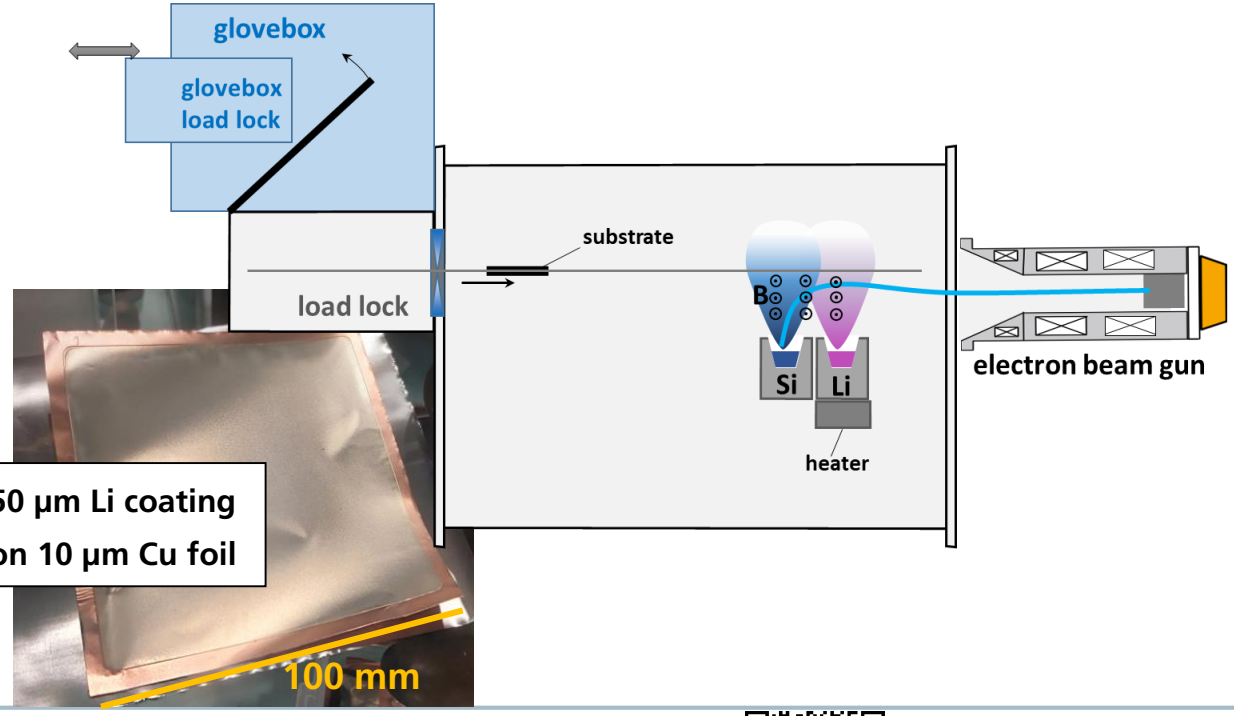




S2S Coater VERSA

Features

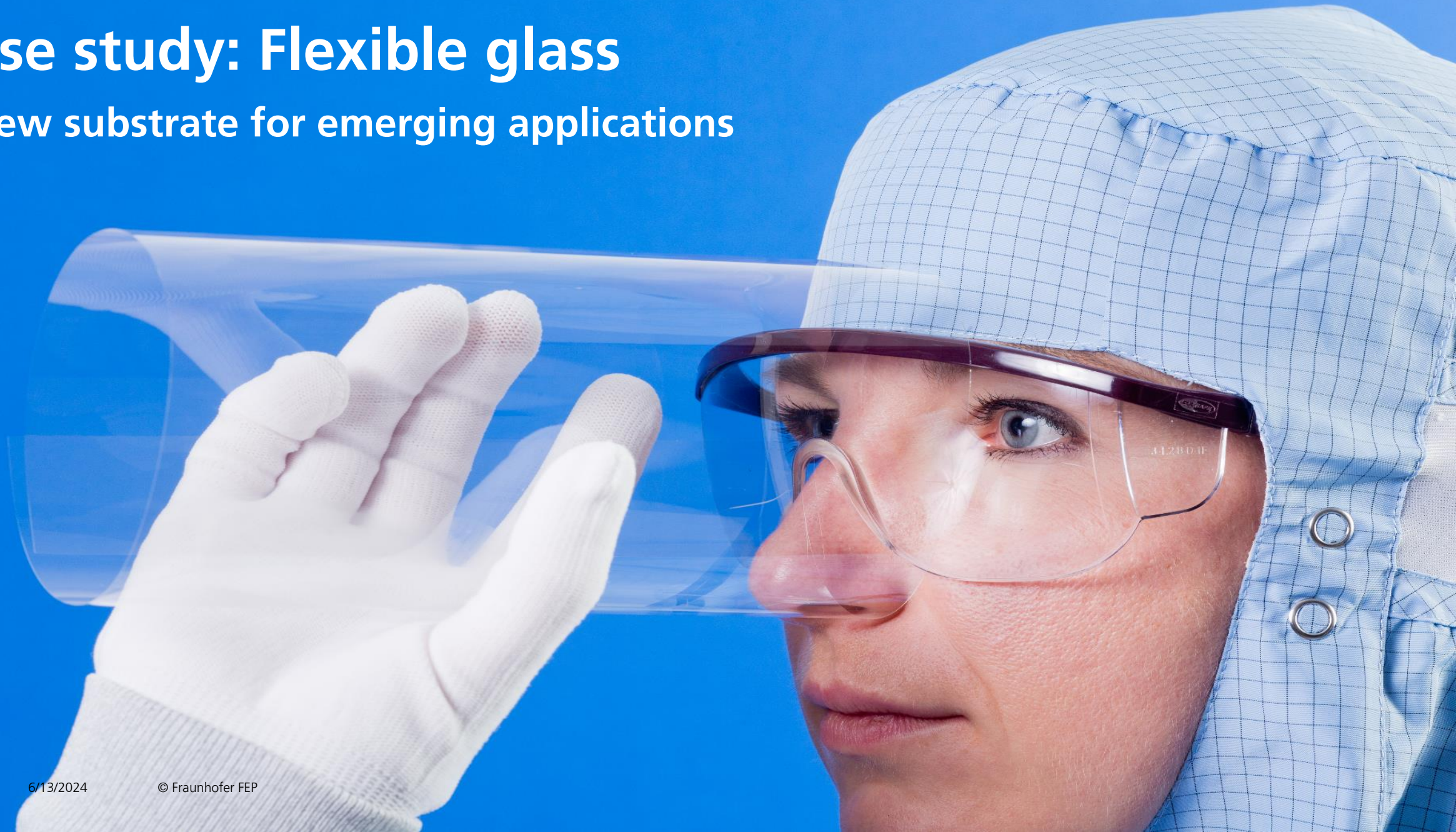
- Sheets of metal, plastic, glass or ceramics up to 12×20 cm²
- Plasma assisted EBPVD, Sputtering, PECVD, thermal PVD
- Inert handling of air sensitive materials



Fraunhofer FEP

Case study: Flexible glass

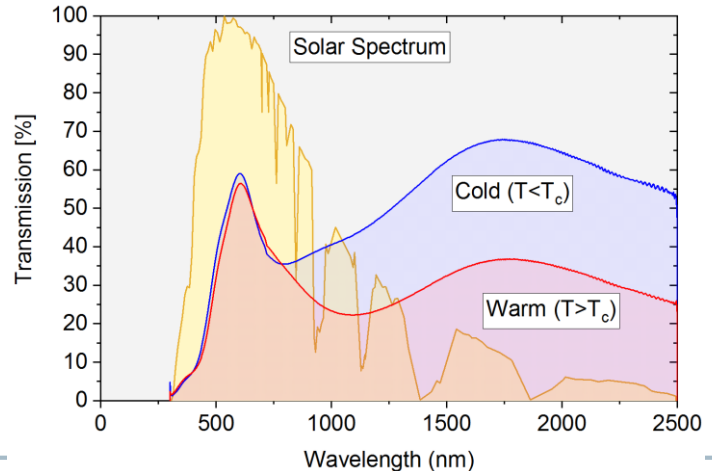
A new substrate for emerging applications

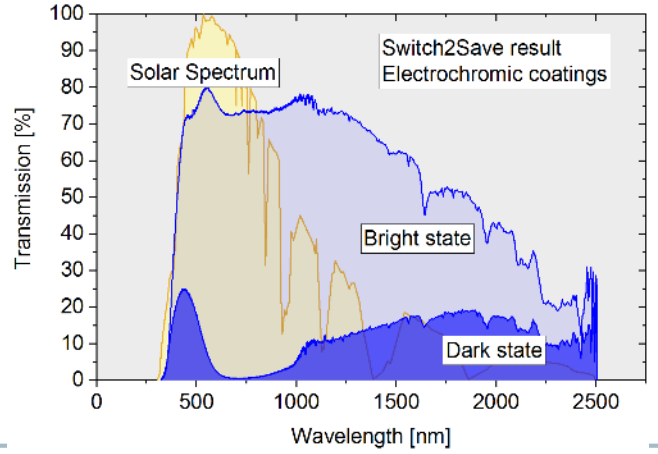


USE CASE: Thin film technologies for energy saving in architecture

Thermochromic coatings and electrochromic devices

Objectives	■ Smart window technology for retrofitting existing buildings ■ R2R sputtering of thermochromic coatings on ultrathin flexible glass NEG G-Leaf, R2R sputtering of permeation barrier layers, transparent electrodes, optical layers
Technology	■ Reactive HiPIMS at elevated substrate temperatures ■ Precise inline property monitoring
Results	■ Roll-to-roll sputtering of thermochromic films on ultra-thin glass ■ Implementation of electrochromic windows in Vasakronan building in Sweden



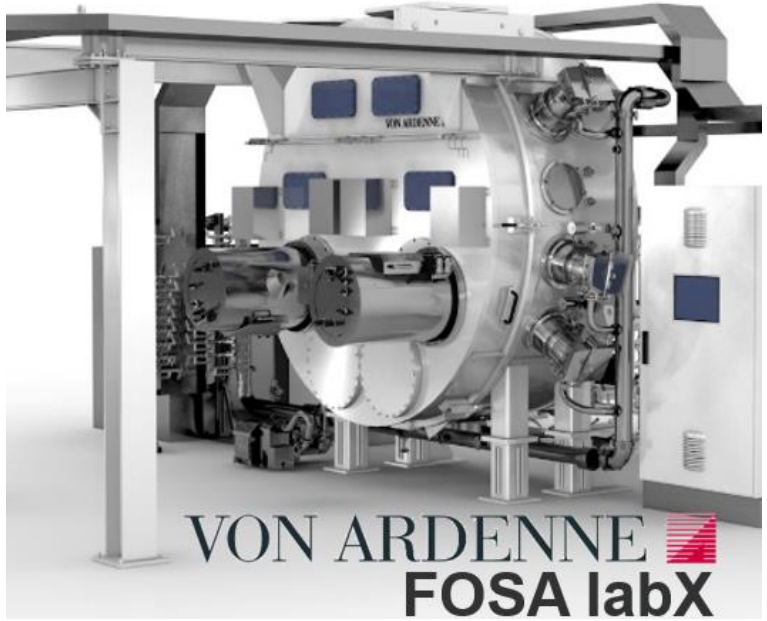


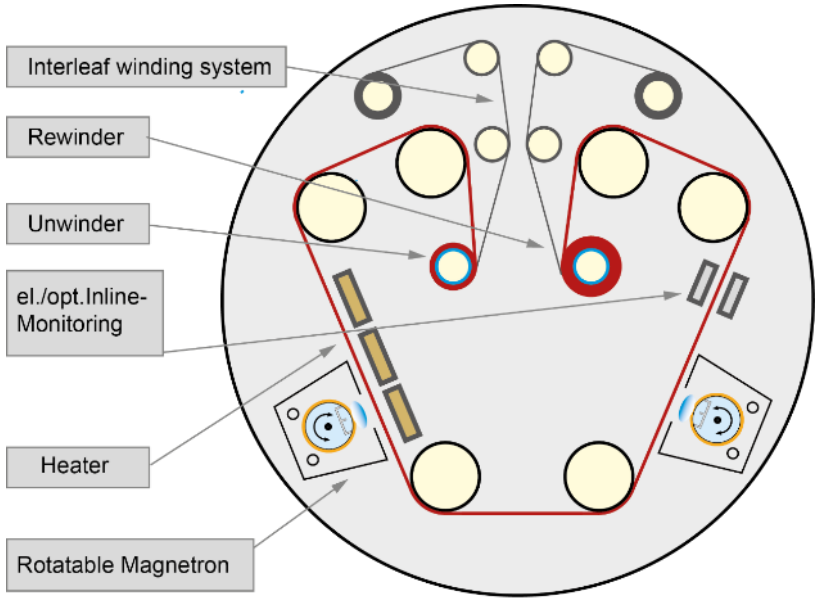
FKZ: 03EN1048
2022 – 2026

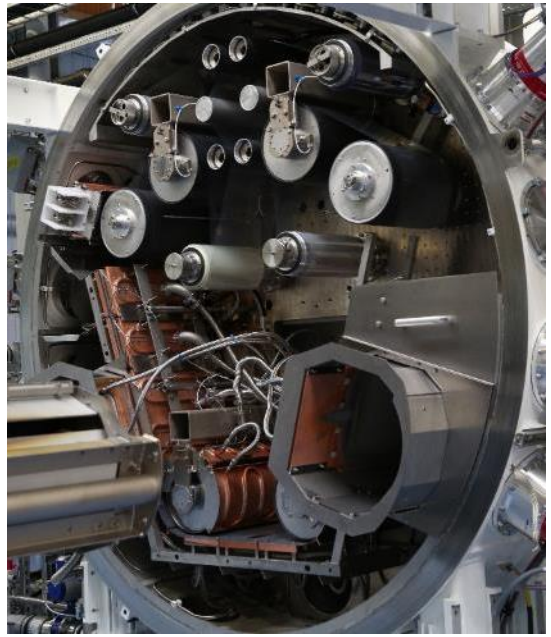
Grant No: 869929
2019 – 2023

PLATFORM: Laboratory Roll Coater FOSA labX

Objectives	■ Sputtering & PECVD on flexible glass ■ Large area upscaling of roll-to-roll processes, pilot deposition
Features	■ 300 mm deposition width, substrate temperature up to 300 °C ■ Pulsed sputtering, HIPIMS, optical and electric inline monitoring



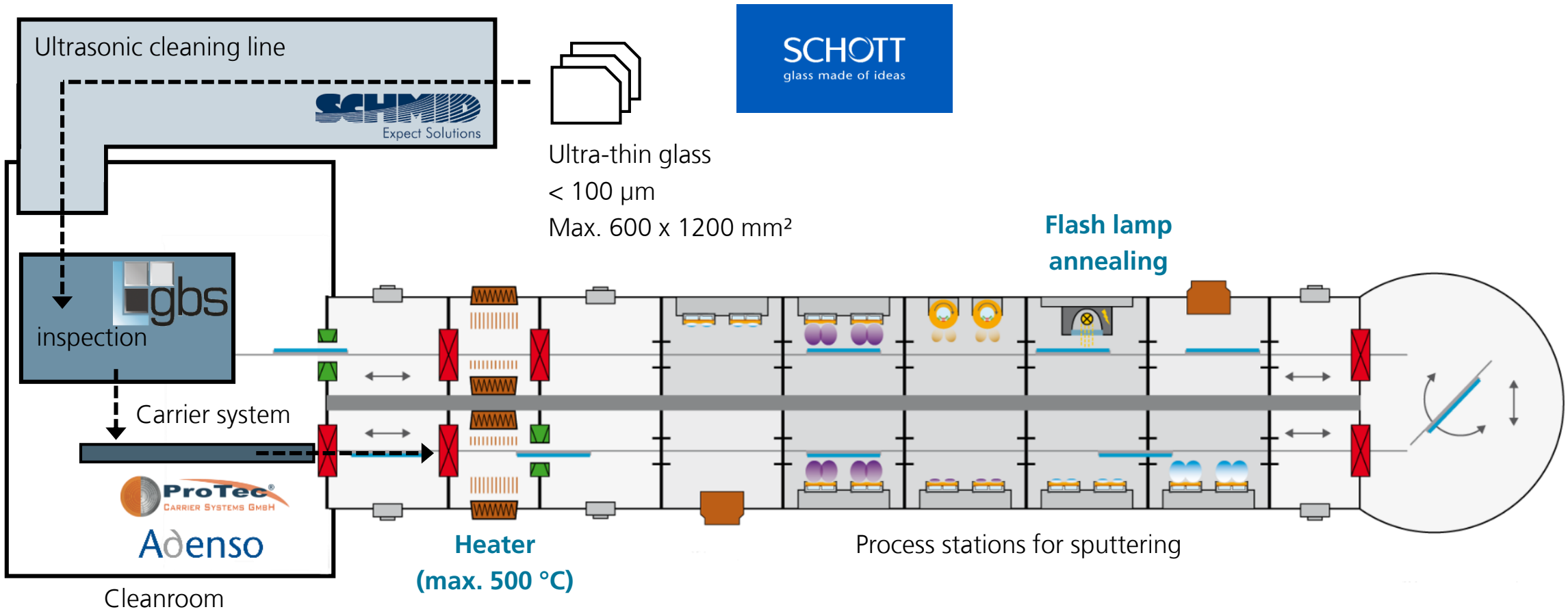




VON ARDENNE
Fraunhofer FEP

PLATFORM: S2S Process line for flexible glass

New process technologies pave the way for flexible glass applications



Glass4Flex
Grant no.: 13N14615
2019 – 2023

Federal Ministry of Education and Research

Fraunhofer FEP

Selected project and application examples

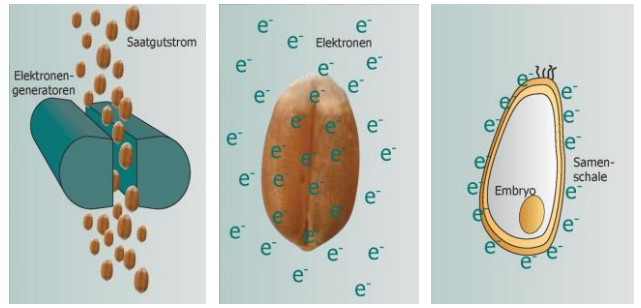
- Coatings for emerging & sustainable technologies
- Technologies for life science applications

Fraunhofer FEP

Phytosanitary Treatment of Seeds with Electrons

Control of pathogens to stop crop diseases and promote germination

Demands	■ Ecological (non-chemical) seed treatment on production scale
Technology	■ Phytosanitary effect of electron seed treatment proven in long term studies together with German Federal Authorities
	■ > 20 years practical field experience by farmers in Germany
	■ No chemical agents, recommended for ecological farming
	■ Stops pollution of air, soil, water and protects users' health
	■ Mobile treatment plants with productivity up to 25 tons/hour
	■ New: Combination with biostimulants and nutrients dressings



Delivery of electrons by two linear sources

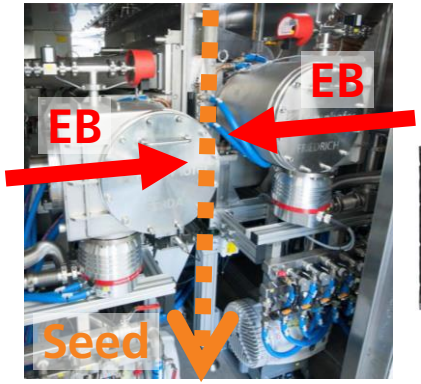
Singling of grains, free fall during treatment

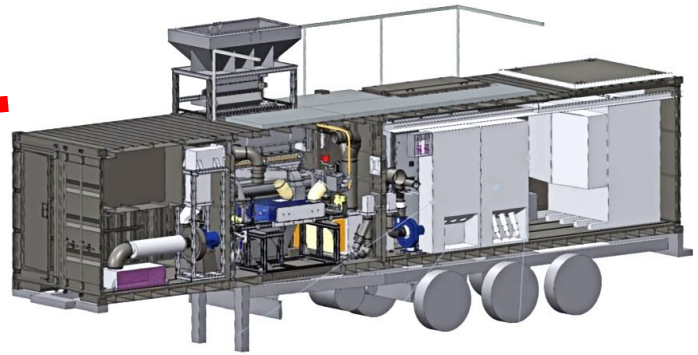
All-round exposure by low-energy electrons

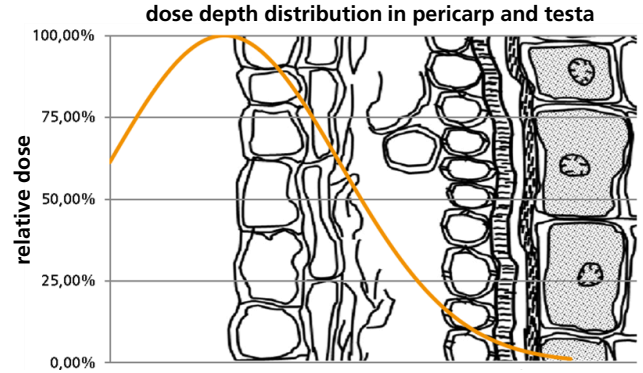
Physical disinfection of complete surface

Penetration of episperm: precise depth control

Embryo remains unaffected by electrons





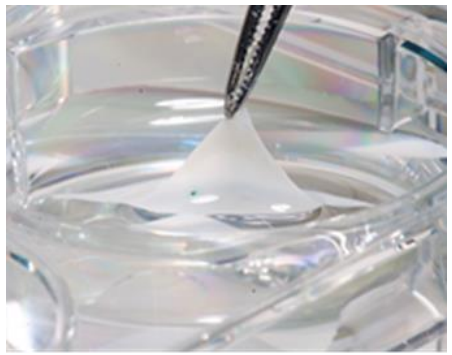
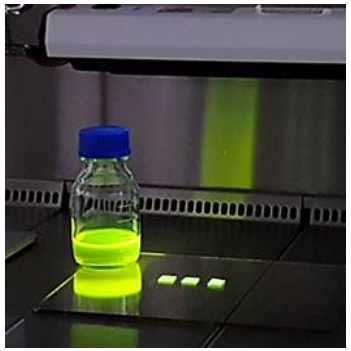


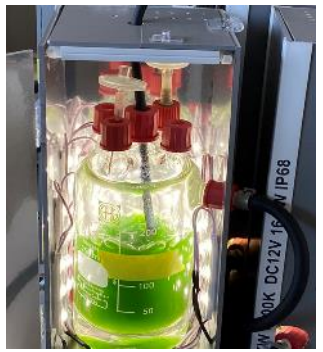
[1] Useful effects of nutrients dressings (source: https://seedforward.com/de/maisguard/) .

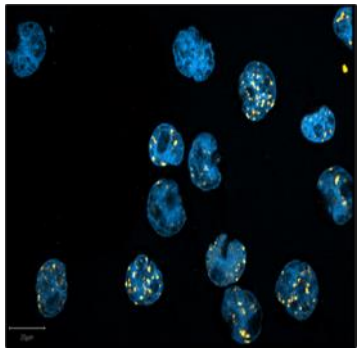
Surface and Material Engineering

Assisted by non-thermal, low-energy electron beam technology

Demands	■ sustainable biobased material engineering ■ Glutaraldehyde-free preparation of biological tissue for medical devices like pericardial tissue applied to biological heart valve prostheses (improve functional life) ■ Biogenic material development via cells and microorganisms ■ Resource-saving, climate-neutral material innovations
Technologies	■ Multi-step biological tissue preparation (patented SULEEI-Processing) ■ Development of living building materials by scalable production of biogenic components via phototrophic microorganisms (substitution processes, assisted by EB technology) ■ Development of biohybrid or synthetic cell-based tissues (assisted by EB-technology)

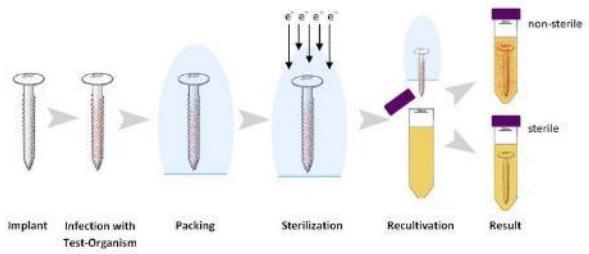


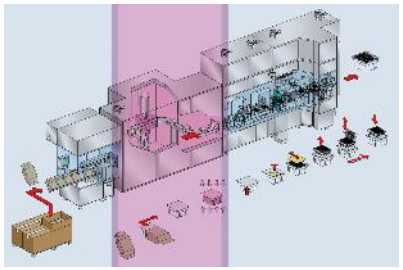


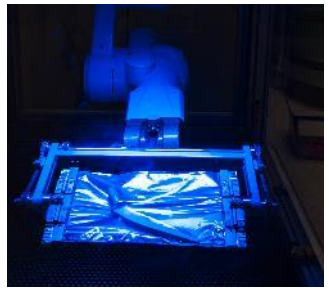
FEP Technologies for Hygienization

Applications from public sector up to medical technology

Demands	■ Gentle sterilization of medical technical and pharmaceutical products ■ In-line suitable technologies ■ Sustainable alternatives to traditional disinfection and sterilization techniques ■ Hygienization of surfaces in public environment, pharma industry as well as in the medical sector
Solutions	■ Disinfection and sterilization by low-energy electron-beam technology ■ Direct and indirect UV-based technologies (UV disinfection, UV induced photoactivity ■ Hygienic cleaning up to disinfection by means of plasma activated water (PAW) ■ Microbiological evaluation and individualized test setups in FEP lab unit





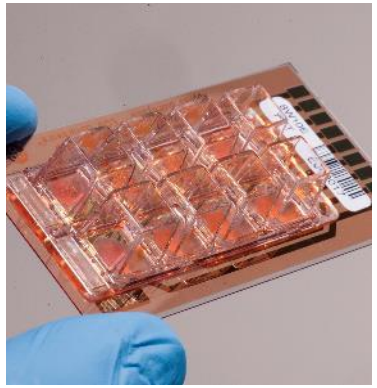


Fraunhofer FEP

Biomedical Laboratory Unit

R&D Lab and service platform for customer specific requirements

Demands	■ State of the art, smart lab unit for services and R&D in life sciences ■ Equipment for microbiology, cell biology, biotechnology, chemistry, material and bio-analytics
Solutions	■ Facility for the development of customer- and project-specific test regimes in accordance with DIN standards ■ Expansion of the range of methods e.g. for testing the effectiveness of antimicrobial surfaces ■ Extension **to biological protection level 2** for testing in a realistic environment


Summary

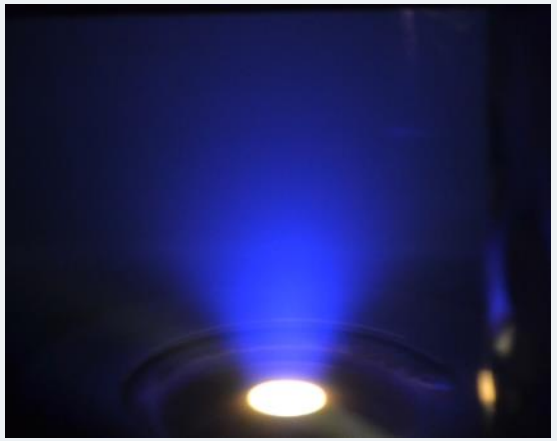
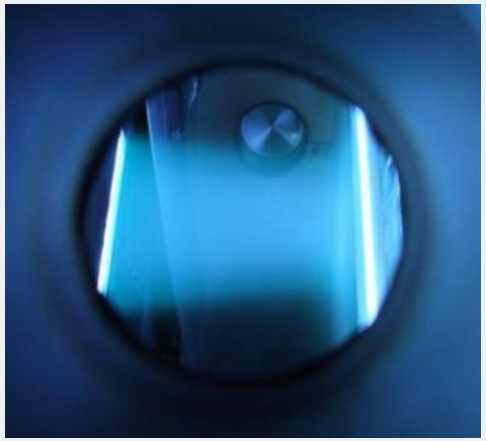
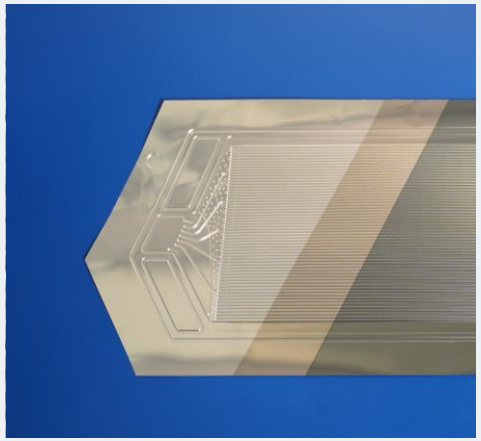
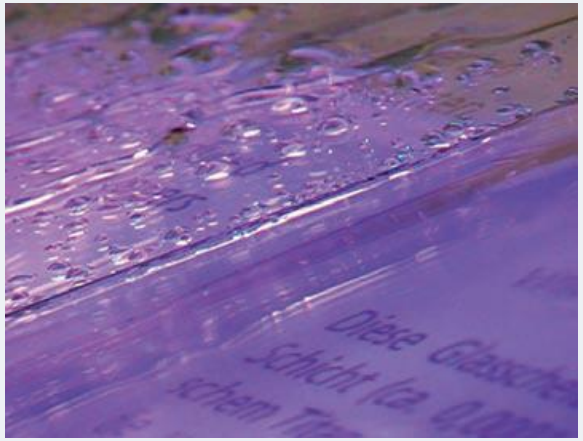
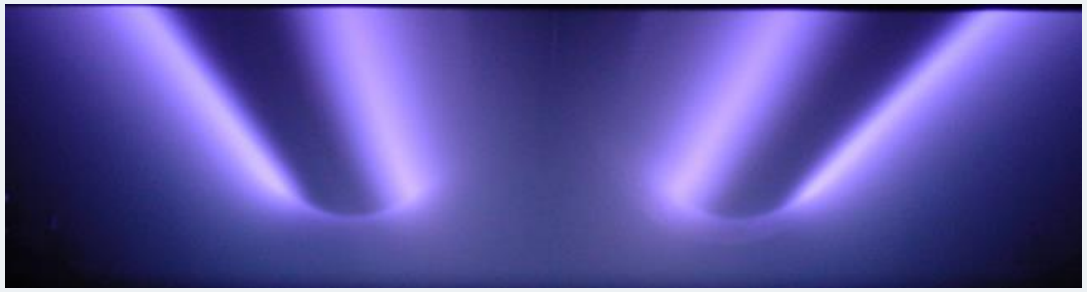
Fraunhofer FEP

Our offer:
Competences and R&D Service

- Thin-film layer and process development mainly by plasma assisted PVD & EBPVD, magnetron sputtering, CVD and PECVD
- Refinement of thin-films by flash lamp annealing
- Flexible glass handling and processing
- Layer stack modeling, design and transfer
- Process setup, adjustment and optimization
- Coatings with high reproducibility
- Up-scaling, sampling, technology transfer
- Key component development

→ Contract research, bilateral, exclusive

→ R&D, Feasibility studies, Sampling, prototyping

We look forward to working with you from the concept phase right through to industrial implementation.

Fraunhofer FEP

Fraunhofer Institute for Electron Beam and Plasma Technology FEP

Contact

Dr. Stefan Saager
phone +49 351 2586-316
stefan.saager@fep.fraunhofer.de

Fraunhofer FEP
Winterbergstraße 28
01277 Dresden, Germany