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Hybrid semiconducting materials for photoelectrochemical energy conversion

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F-Praktikum Review
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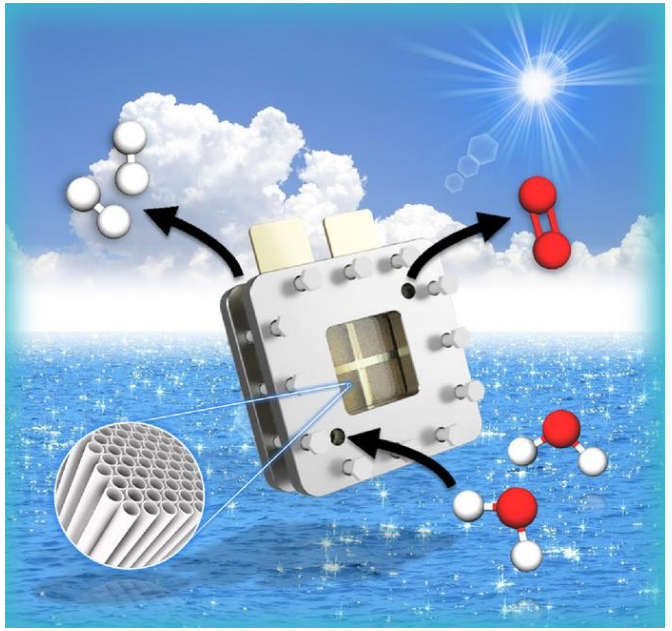
Semiconductor thin-films for PEC water splitting

- **PEC water splitting:** storing solar energy into chemical bonds – fossils to renewables energy transition.
- **Link to bachelor program?** Solid State Physics (Physik IV) – crystal structure, X-ray diffraction, semiconductor physics and devices
- **Emphasis of the experiments?** Characterization of emerging photoactive materials in terms of crystalline phase, morphology, PEC properties.
- **Equipment**: hands-on with state-of-the-art PXRD, SEM and PEC setup (PEC cell, potentiostat, solar simulator, LED light sources)



ChemSusChem, 2019, *12*(9), 1925-1930

<u>Aim of the course</u>: Studying the PEC properties of emerging materials in PEC water splitting.

- ❑ Deposition of thin-films (e.g. metal-oxides) on a conductive substrate.
- ❑ Characterization using XRD, SEM
- ❑ Evaluation of PEC properties of the films in a PEC cell
- ❑ Data analysis – comparison with available literature

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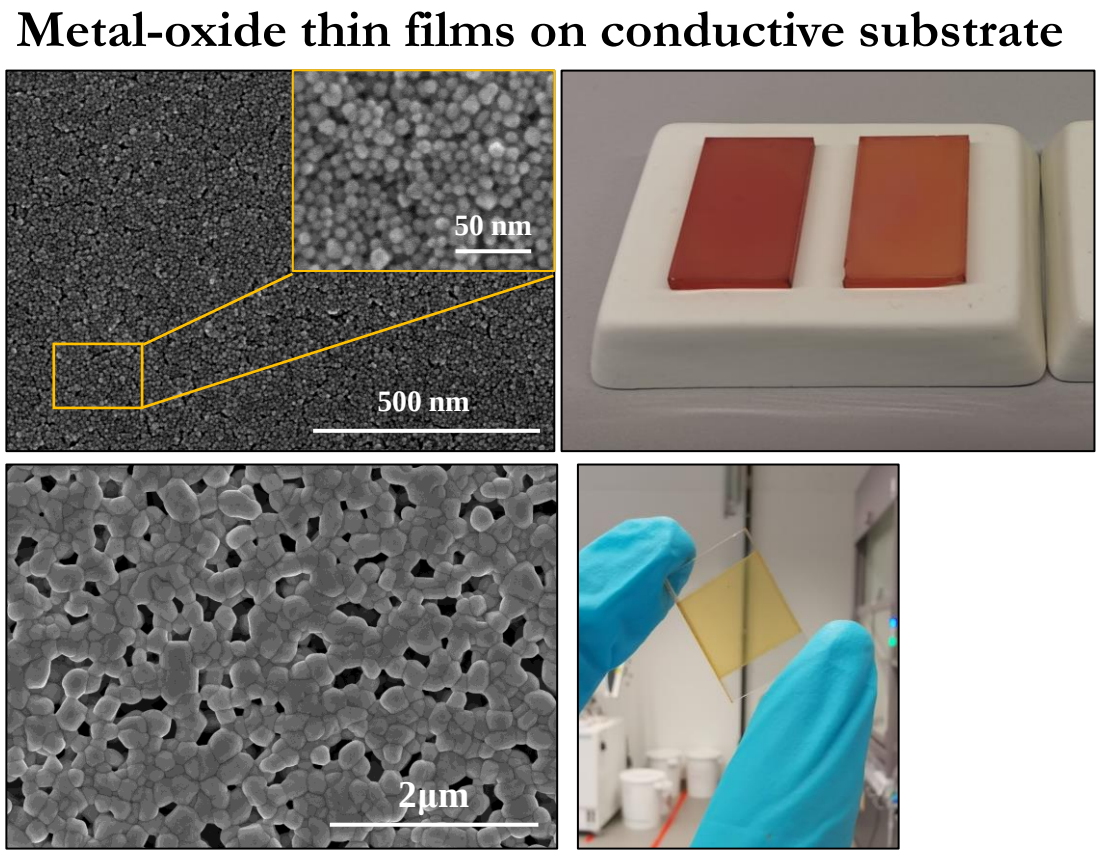
Day 1 – Deposition of thin-films on a conductive substrate

- Planning of the synthetic strategy – preparation of precursor solution – deposition via spin-coating
- Learn how to obtain uniform film depositions
- Preparation of films with different thicknesses and morphology



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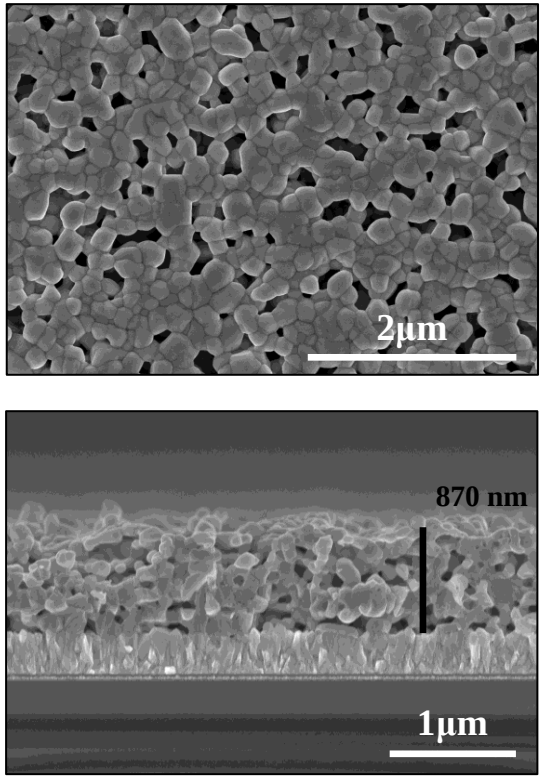
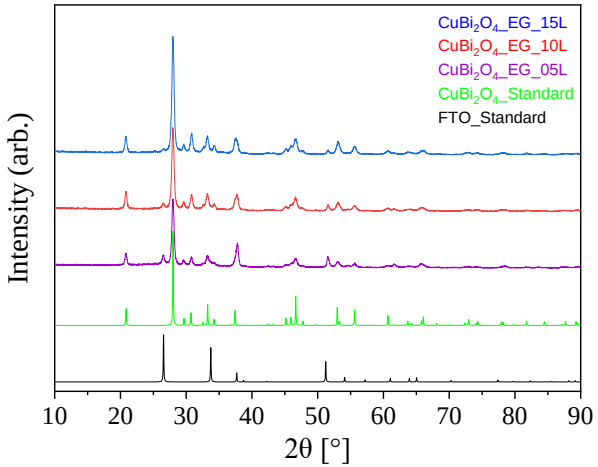
Day 2: Characterization of the films

- Crystalline and morphological characterization of the films
- Learn how to obtain the PXRD pattern of the films
- Comparison with references – evaluation of purity of crystalline phase
- Learn how to obtain SEM images (top view + cross section)

SEM



XRD



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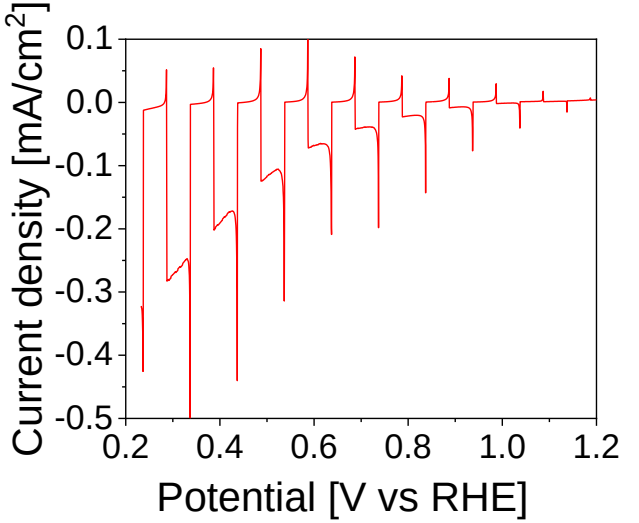
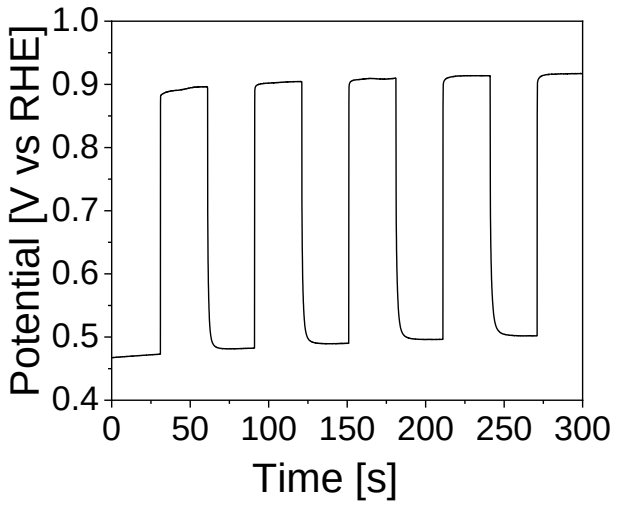
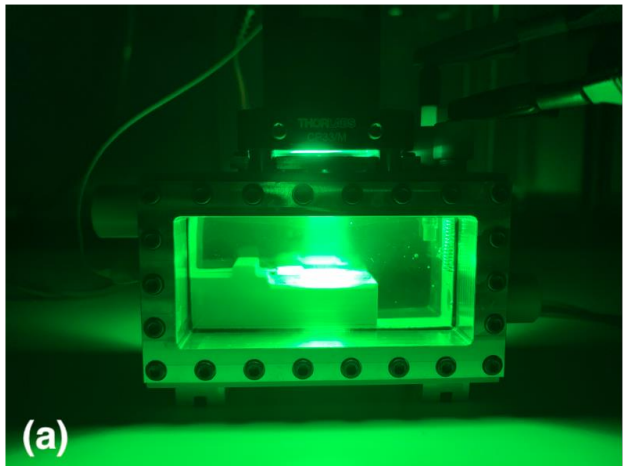
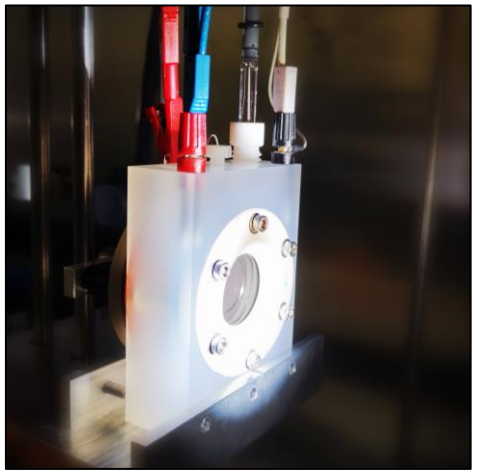
Day 3 – Evaluation of the PEC properties

- Learn how to use state-of-the-art PEC setup – PEC cell, potentiostat, solar simulator, LED light source.
- Calibration of the different light sources (power output and spectrum)
- Assembly of PEC cell components – connection of WE, RE, CE and understanding of their function
- Understanding the role of the 3-electrode setup and reference electrode – the Ag/AgCl vs. RHE scale – comparability of experimental data with literature.
- Carry out basic PEC diagnostic measurements: OCP, LSV, Chronoamperometry, Cyclic voltammetry (ECSA).
- Carry out measurements changing light source – solar simulator vs. LED light with different wavelength – how do these affect the photocurrents? And why?
- Carry out long-term stability test (chronoamperometry) – discussion about stability of the prepared films – possible source of instability? Photocorrosion?









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After the lab – data analysis

Data analysis and figure of merits:

- ❑ Crystallinity – phase purity via PXRD – comparison with standards from the literature
- ❑ Evaluation of morphology – thickness of the film via SEM
- ❑ Determine n-type / p-type semiconductive behaviour via chopped-light OCP
- ❑ Determine on-set potential and photocurrents achieved (LSV) – measurement of multiple sample for statistical data treatment
- ❑ Determination of the electrochemically active surface area (ECSA, cyclic voltammetry)
- ❑ Evaluation of the long-term stability of the film (chronoamperometry)

Theory / preparation	Setup / experimental	Data taking	Analysis	Protokol
1-2	3	3	3	1-2