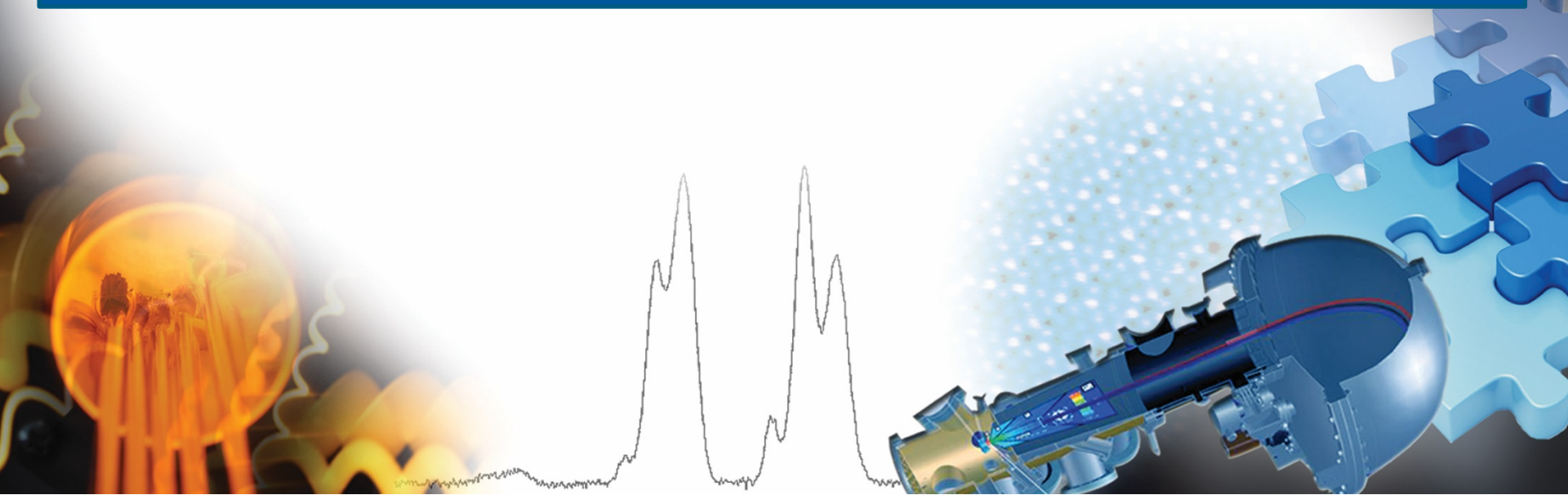


# Helmholtz Energy Materials Characterization Platform

Harald Bolt

7<sup>th</sup> April 2016, HELSMAC, Cambridge

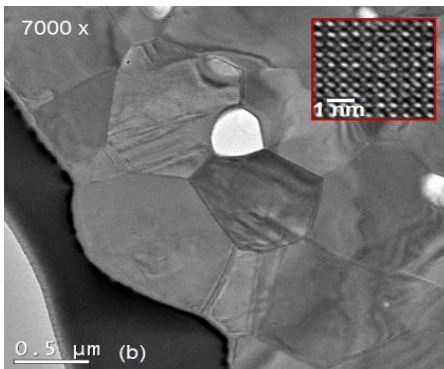
# Essential Elements for Transformation of the Energy System



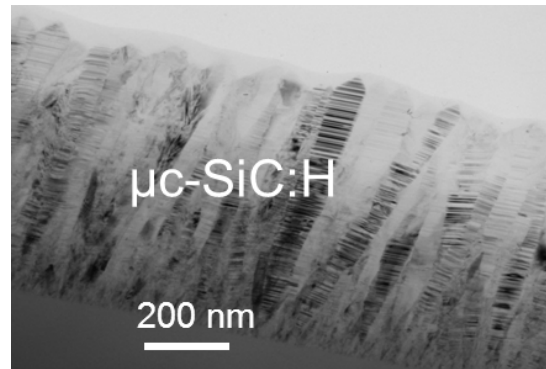
- **Storage**
- **Renewables**
- **Grids**
- **Efficiency**
- **Flexible Power Plants**

# Motivation

- New materials are essential for the realization of new energy conversion and storage technologies.
  - central importance for future energy systems
- Timescale for development: 10 -30 years;  
Operation time: 20 – 50 years (long-term stability)
- Material design and the determination of long-term properties require detailed knowledge of the structure and functional properties; from atomic scale to component

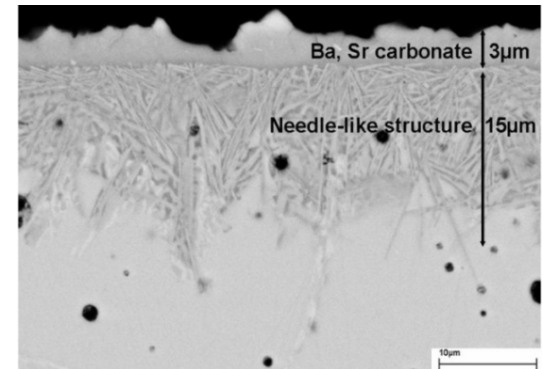


Defects and microstructure in perovskites



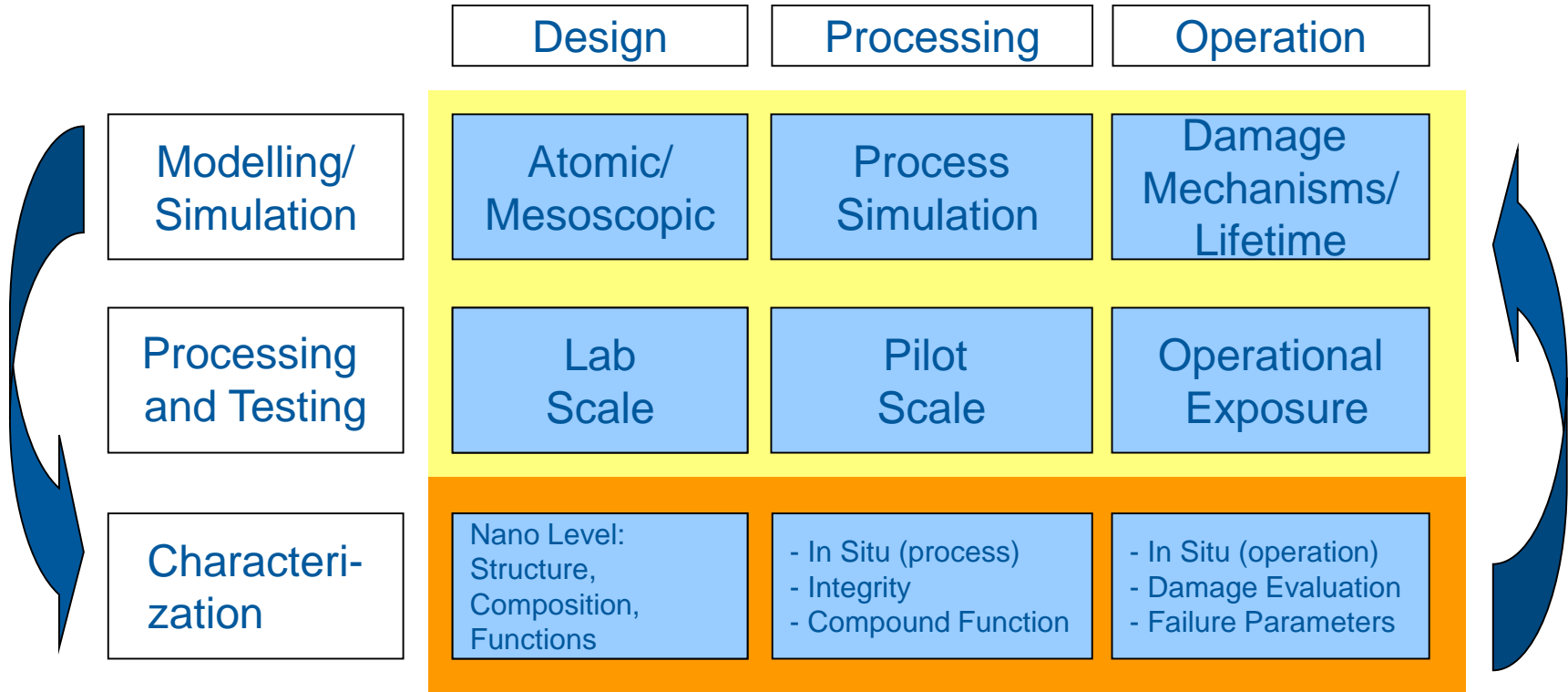
Microstructure of SiC:H

72 vol% N<sub>2</sub>, 18 vol% O<sub>2</sub>, 10 vol% CO<sub>2</sub>



Stability in CO<sub>2</sub> containing the atmosphere

# Energy-material research in Helmholtz



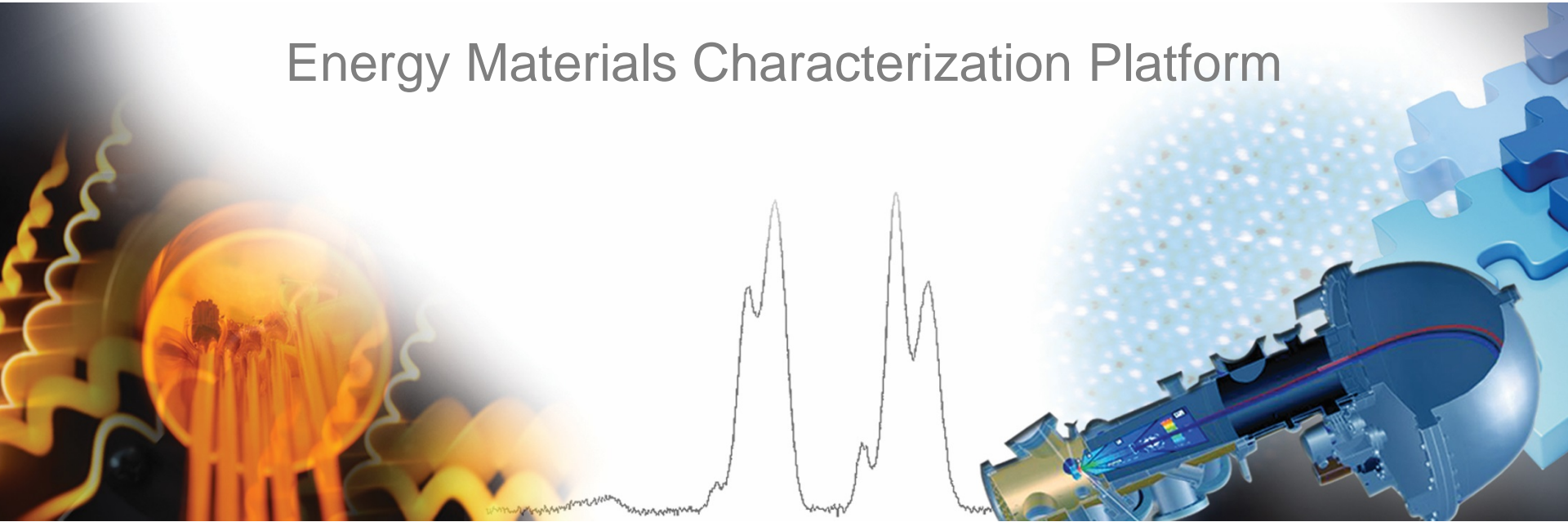
yellow

POF- and Portfolio topic „Materials Research for Future Energy Supply“

orange

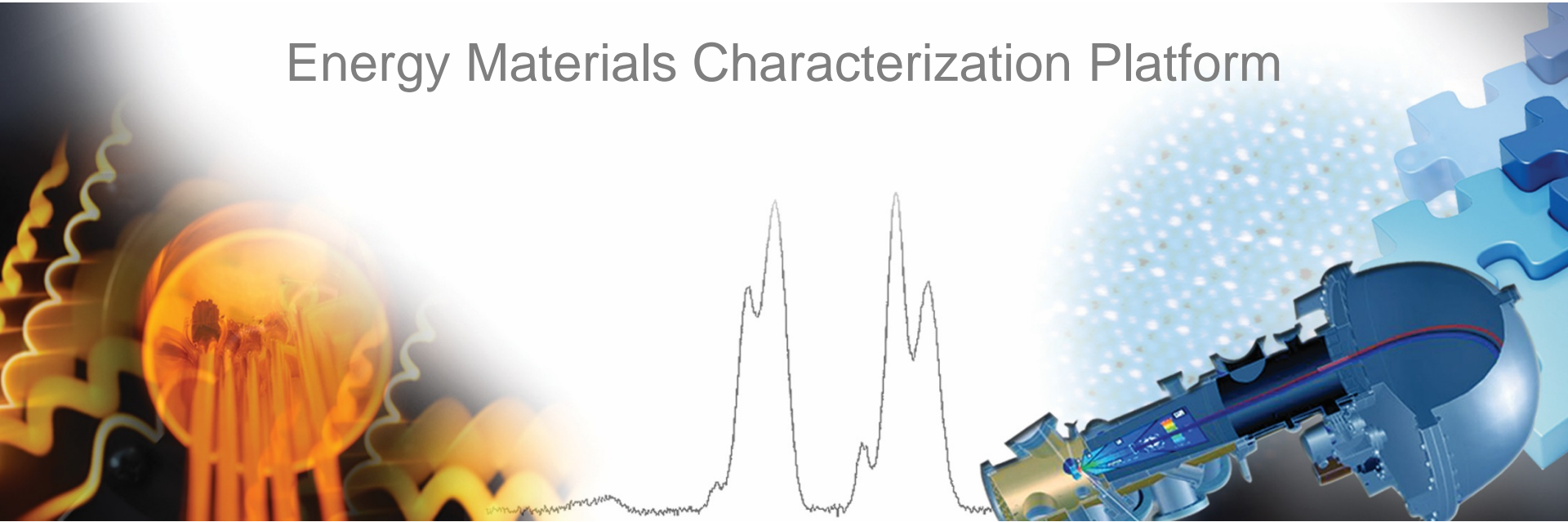
HEMCP subjects in the energy materials research context

## Energy Materials Characterization Platform



- Infrastructure supported by the Helmholtz Association
- Unique collection of devices and analytical methods
- Seven research institutions under one virtual roof
- Wide range of options for research issues on materials for energy technology

## Energy Materials Characterization Platform



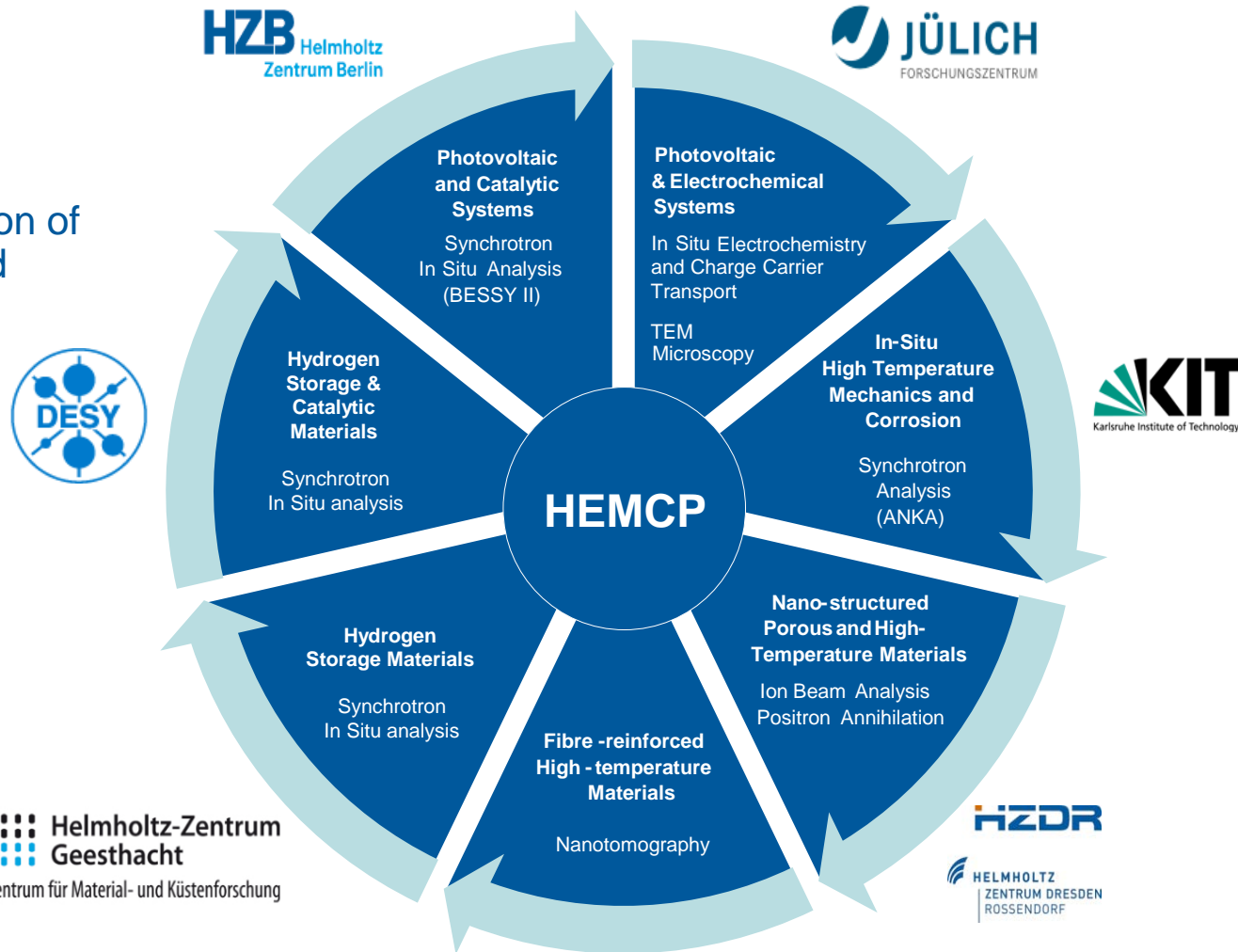
- Development of efficient and sustainable energy conversion and storage technologies
- Time and spatial resolution (3D) information on structural, electrical and chemical properties under preparation and operating conditions (in-situ methods)

# HEMCP: Participating Helmholtz Centers, Subjects

Complementary consolidation of large scale equipment and high-end-analytics

In-situ characterization platform

Platform for collaboration between centres, with universities and industry



# HEMCP: Funding

## Investment (in €)

Center	Funding
FZJ	15.500
KIT	6.800
HZDR	6.300
HZB	6.600
DLR	1.730
HZG	1.700
<b>Total</b>	<b>38.630</b>

- Particular relevance of HEMCP on research policy (“Energiewende”)
- Substantial funds of the centers;
- DESY: in-kind contribution

Gefördert durch:



Bundesministerium  
für Wirtschaft  
und Energie

aufgrund eines Beschlusses  
des Deutschen Bundestages

GEFÖRDERT VOM

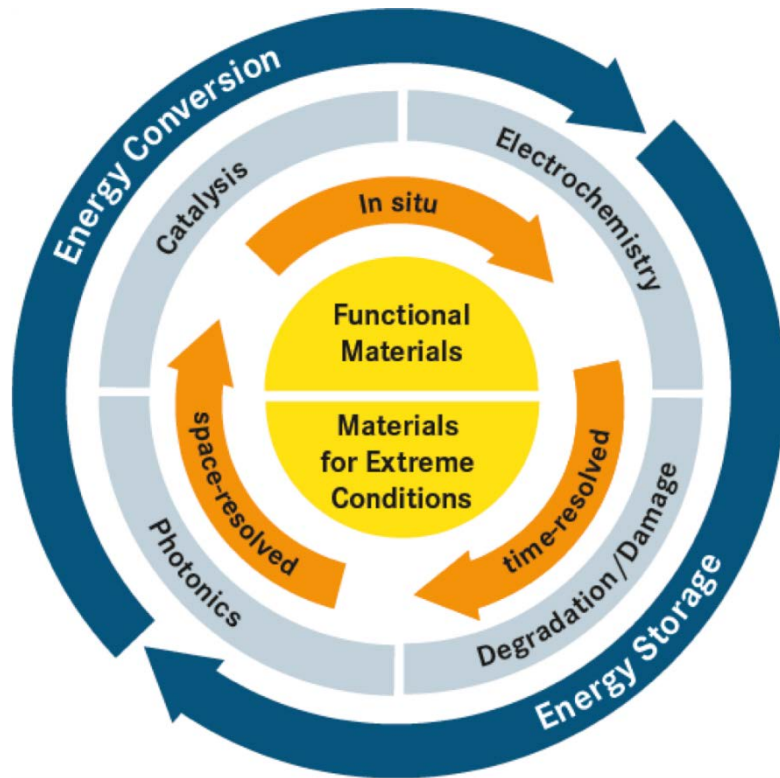


Bundesministerium  
für Bildung  
und Forschung





# HEMCP: Research fields



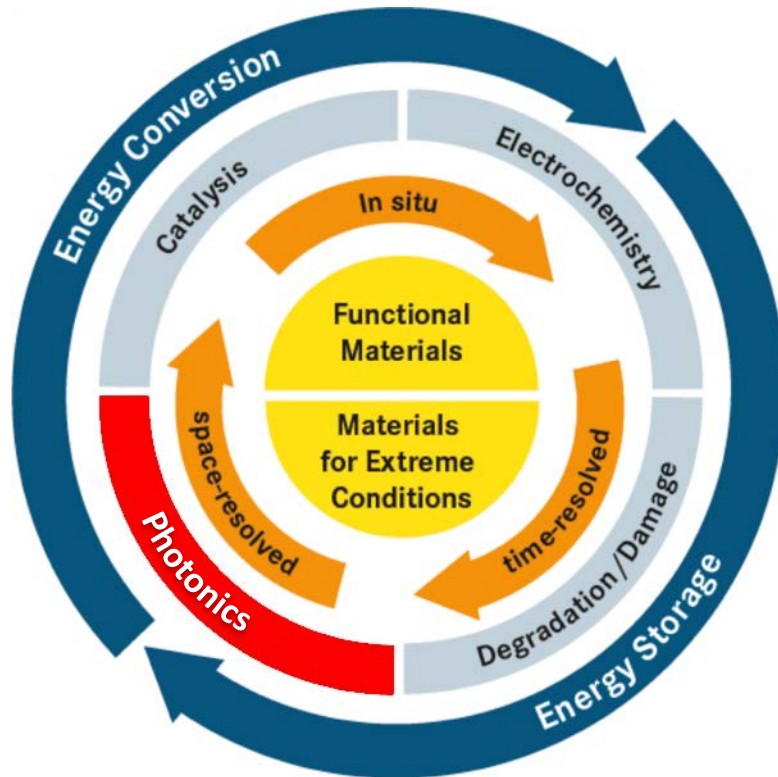
## Energy Conversion and Storage

HEMCP concentrates on technology areas where improvements are especially dependent on progress in material science:

- Photovoltaic systems
- Chemical conversion and storage
- Electrochemical energy conversion and storage
- Thermal energy conversion

# HEMCP: Research fields

## Photonics (esp. PV)

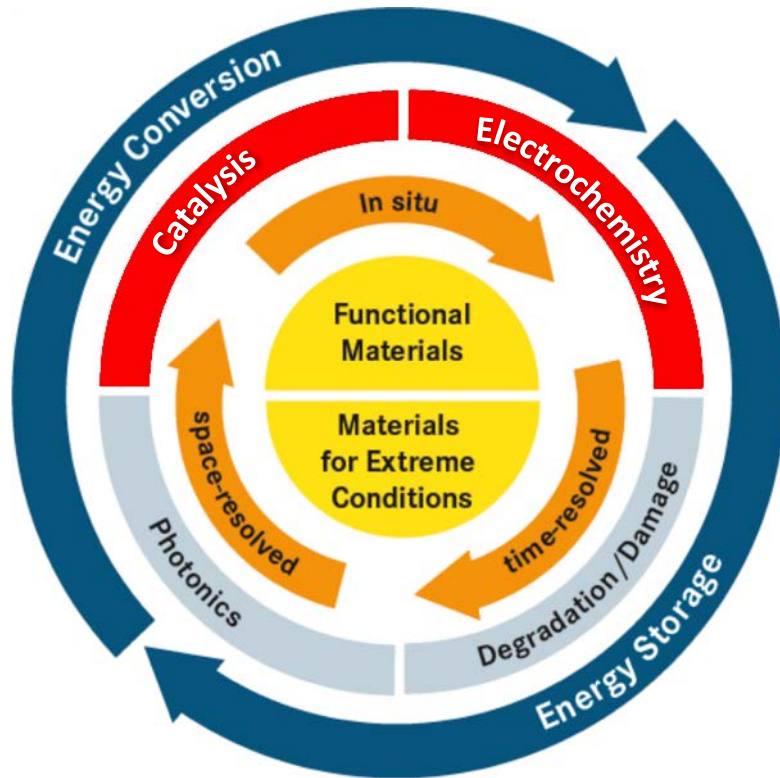


- Improvement of functional layers and interfaces relies on precise analysis of electronic and chemical properties
- Characterization methods and simulation capabilities to analyze optoelectronic properties
- All relevant length scales from atomistic resolution of nanostructures and elementary processes up to the yield analysis of modules or photovoltaic systems.

### Analytical tasks:

- Absorption and reflection from rough structures
- Optoelectronic properties of surfaces and nanoparticles
- Work function „design“ and characterization
- Electronic properties and atomic structure of interfaces

# HEMCP: Research fields



## Electrochemistry / Catalysis

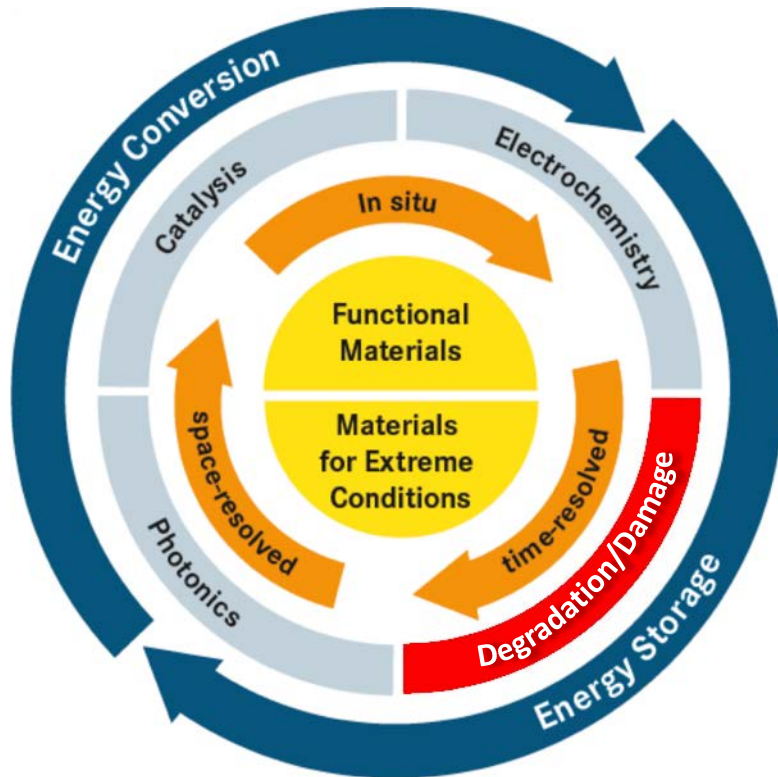
- Understanding of electrochemical and catalytic reactions and ionic transport properties
- Understanding of kinetics and mechanisms of the e.g. electrode processes for the development of novel electrolyte and electrode materials.
- Physico-chemical analysis of materials, components and functional interfaces with respect to the defect chemistry

### Analytical tasks:

- Surface reactions in liquids and gases
- In-situ chemical and structural analysis
- In-situ analysis of film and interface formation, electronic properties
- Electrode/electrolyte interaction
- Hydrogen loading/deloading kinetics

# HEMCP: Research fields

## Degradation/Damage



- Analysis of the microstructure of ceramic-based composites and new metallic alloys under highest thermo-mechanical and corrosive loading
- Investigation of degradation and life time predictions and on the other hand to develop improved materials and components.

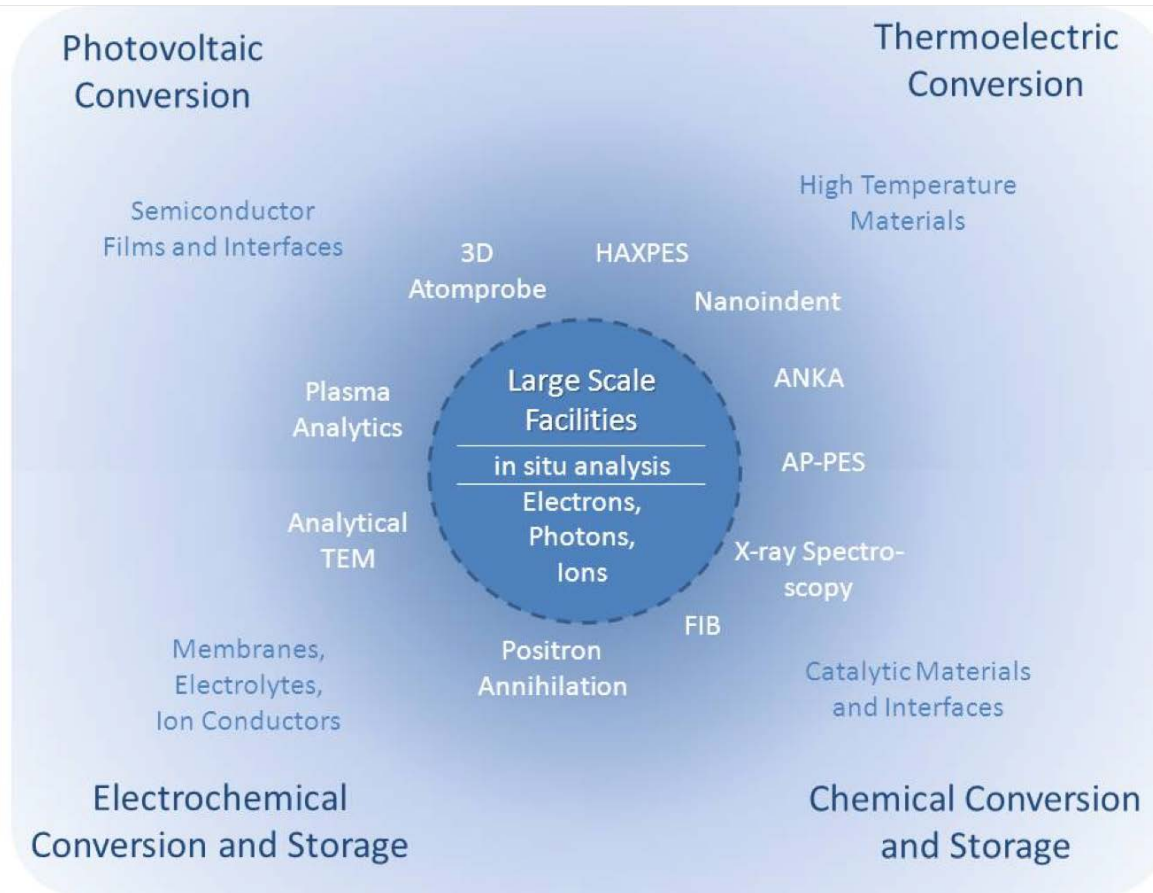
### Analytical tasks:

- Structural evolution under operating conditions
- Surface reactions in liquids and gases
- Element analysis on atomic scale
- Pore structure of electrodes, electrode/electrolyte interaction
- Fatigue and corrosion reactions of structural materials and functional protective

# HEMCP: Instrumentation

## Characterization methods

- In-situ methods close to preparation or operation conditions
- Structural characterization of functional nanostructured materials for energy applications
- Enabling of large-scale facilities for in-situ analysis and characterization methods with high resolution



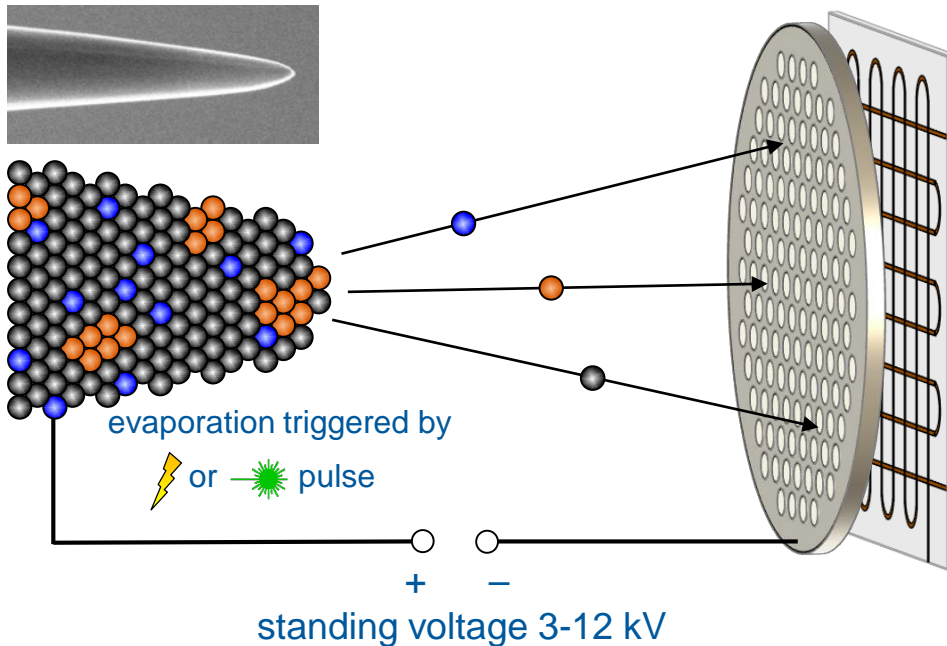
# HEMCP: Instrumentation

<u>Instrument</u>	<u>Center</u>
Cryogenic Impact Testing	KIT
Cryo-Heatcapacity Physical Properties Measurement System (PPMS)	KIT
Cryo X-ray Diffraction (Cryo-XRD)	KIT
Field Emission Scanning Electron Microscope (FEG-SEM)	KIT
Field Emission Scanning Electron Microscope combined with Focussed Ion Beam (FEG-SEM/FIB)	KIT
Raman Microscope	KIT
<b>Focussed Ion Beam (FIB)</b>	DLR
Hard X-Ray Photoelectron Emission Microscopy (HAX-PEEM)	FZJ
<b>3-D Atomprobe Tomography (APT)</b>	FZJ
Transmission Electron Microscopy (TEM) – Titan G2 80-200 Crewely	FZJ
Ambient Pressure Photoelectron Spectroscopy (AP-XPS)	FZJ
<b>Juelich Online Silicon Growth Experiment for Photovoltaics (JOSEPH)</b>	FZJ
<b>Solar Energy Material In-situ Spectroscopy at the Synchrotron (SISSY)</b>	HZB
100 liter milling drum set up (CM100B)	HZG
Hydrogen Tank Test Facility II (HTTF II)	HZG
In-situ Synchrotron Radiation Powder X-ray Diffraction (in-situ SR-PXD)	HZG
Helium Ion Microscopy (HIM)	HZDR
Apparatus for in-situ Defect Analysis (AIDA)	HZDR
Secondary Ion Mass Spectrometry coupled to Tandem Accelerator (Super-SIMS)	HZDR
<b>Transmission Electron Microscopy (TEM) – TalosF200X</b>	HZDR

# Instrumentation: Atom probe tomography

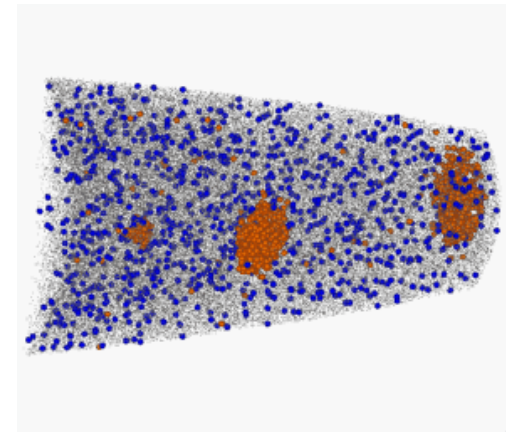
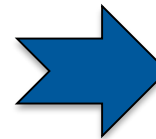
Tip-shaped specimen  
( $R \sim 50$  nm)

Position-sensitive  
detector



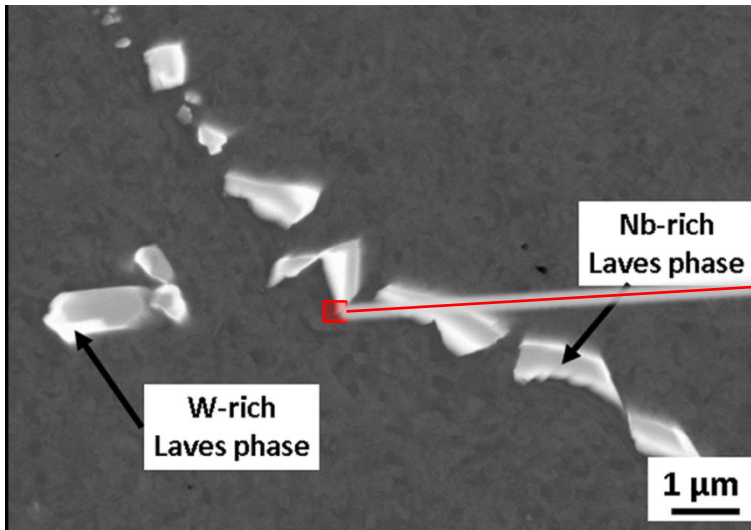
- High spatial resolution ( $< 1$  nm)
- High mass resolution ( $m/\Delta m > 1000$ )
- High detection sensitivity (down to 10 ppm)
- Equal sensitivity to all elements  
...at the expense of analysis volume

Hit position at the detector  $\rightarrow$  X-Y coordinates  
Ion evaporation sequence  $\rightarrow$  Z coordinate  
Time of flight  $\rightarrow$  chemical identity

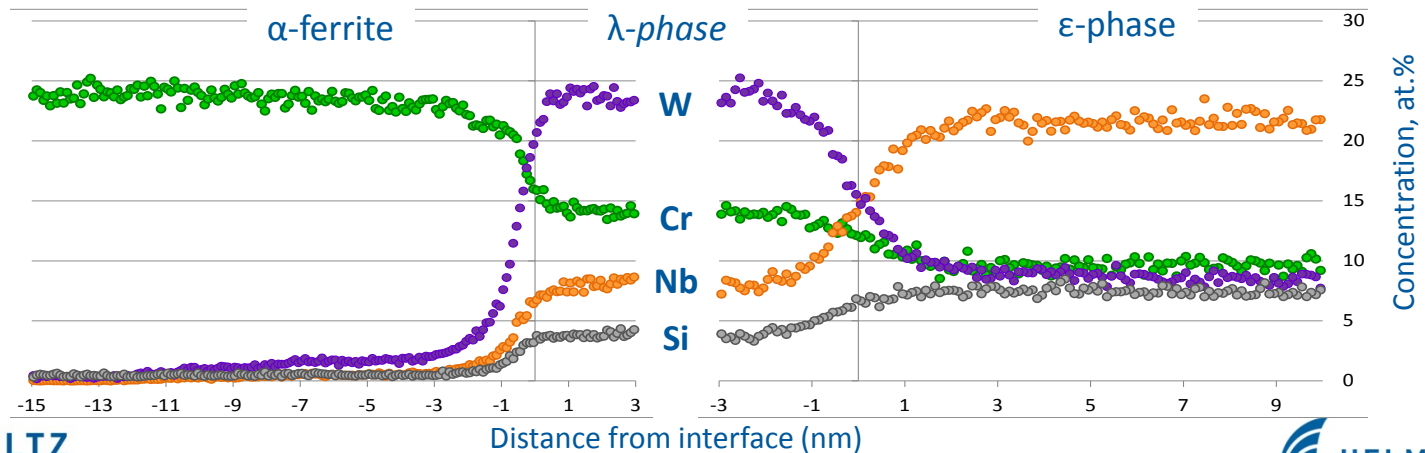
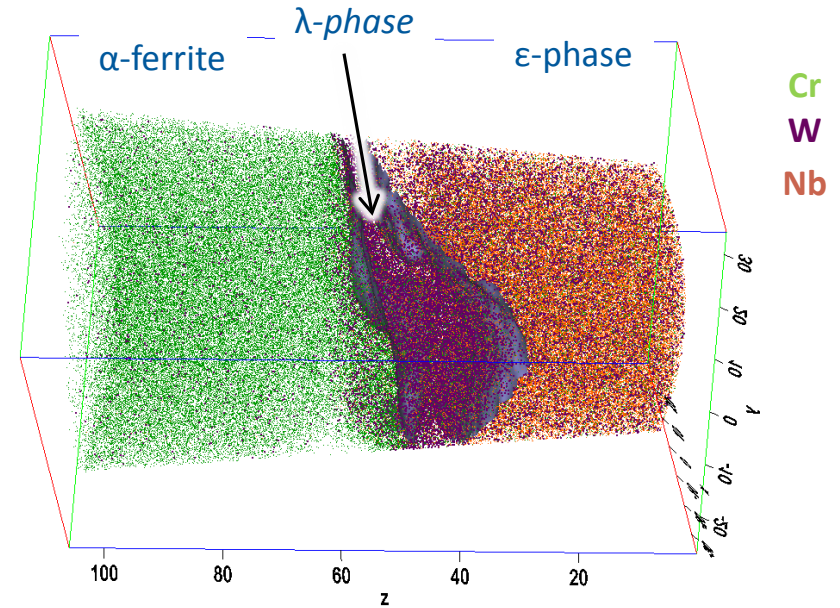


# Instrumentation: Atom probe tomography

Crofer 22H - ferritic steel for solid oxide fuel cells interconnects. Laves phase chemistry?



L. Niewolak et al., *J. Ph. Equil. Diff.* 36 (2015) 471





# Instrumentation: TEM

## Transmission Electron Microscopy FEI Talos F200X



200 kV FEG-TEM equipped with

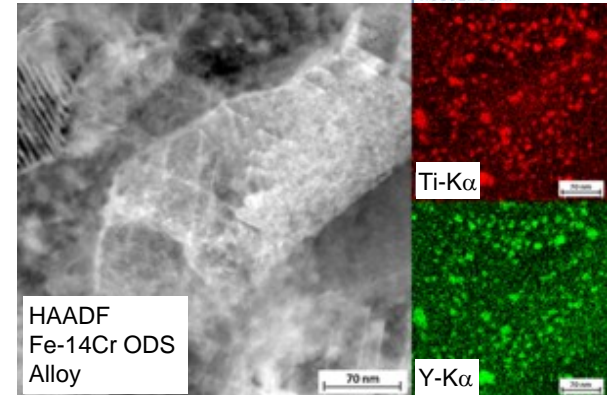
- High-performance EDXS system for efficient mapping in STEM
- STEM, BF/LAADF/HAADF
- In-situ straining holder

### Applications

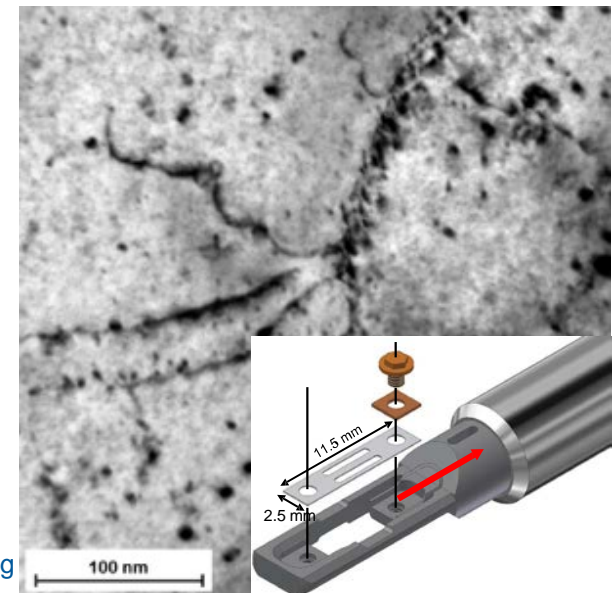
- High temperature materials for energy applications (e.g. ODS steels)
- Radiation defects in nuclear materials

### Scientific topics

- Concentration, size, degree of coherency and composition of nm-sized oxide particles
- Deformation phenomena such as plastic localisation and the underlying mechanisms
- Interaction of dislocations with nm-sized particles and lattice defects under mechanical stress (in-situ straining, long-term objective)



High performance EDXS mapping



Interaction of dislocations with defects



HELMHOLTZ

ENERGY MATERIALS  
CHARACTERIZATION PLATFORM

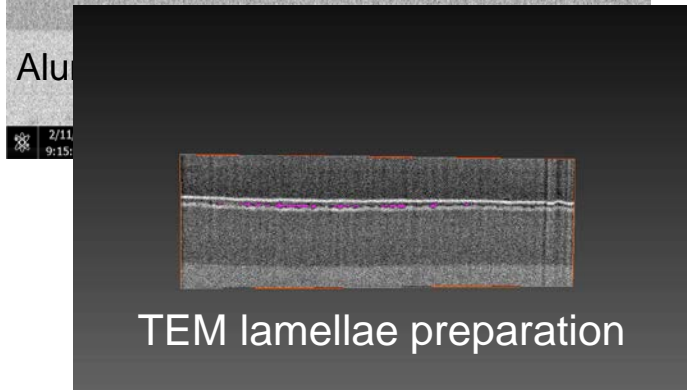
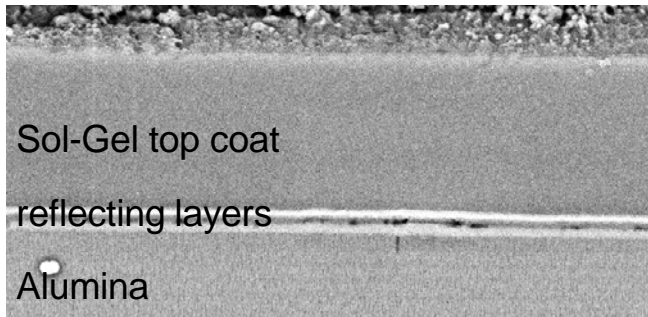
Artur Erbe | Institut für Ionenstrahlphysik und Materialforschung  
Transportphänomene in Nanostrukturen

# Instrumentation: FIB/TEM

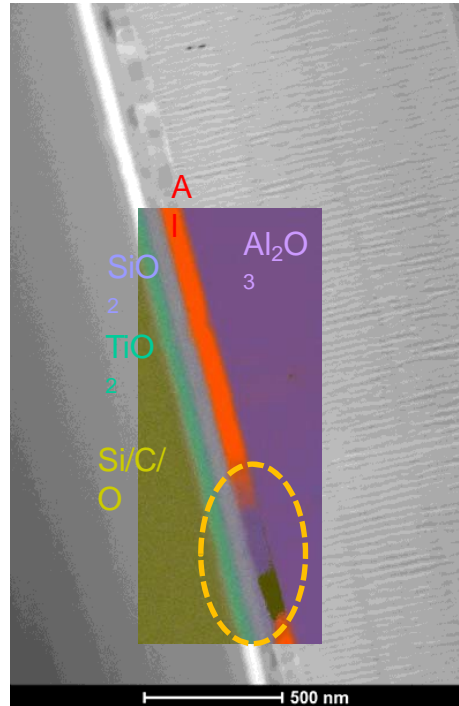
Corrosion phenomena in Al-mirrors  
Cross cut of corrosion spot with elemental analysis



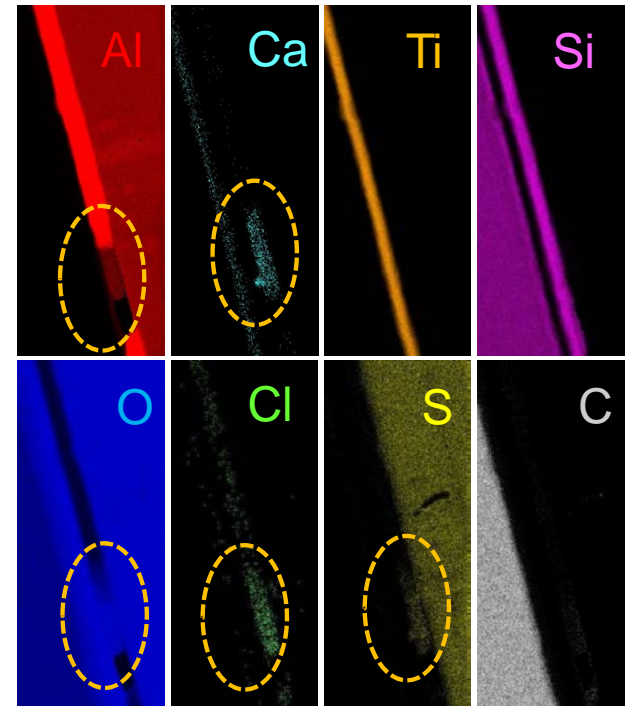
Focussed Ion Beam  
(FIB)



High Angle Annular  
Dark Field Imaging

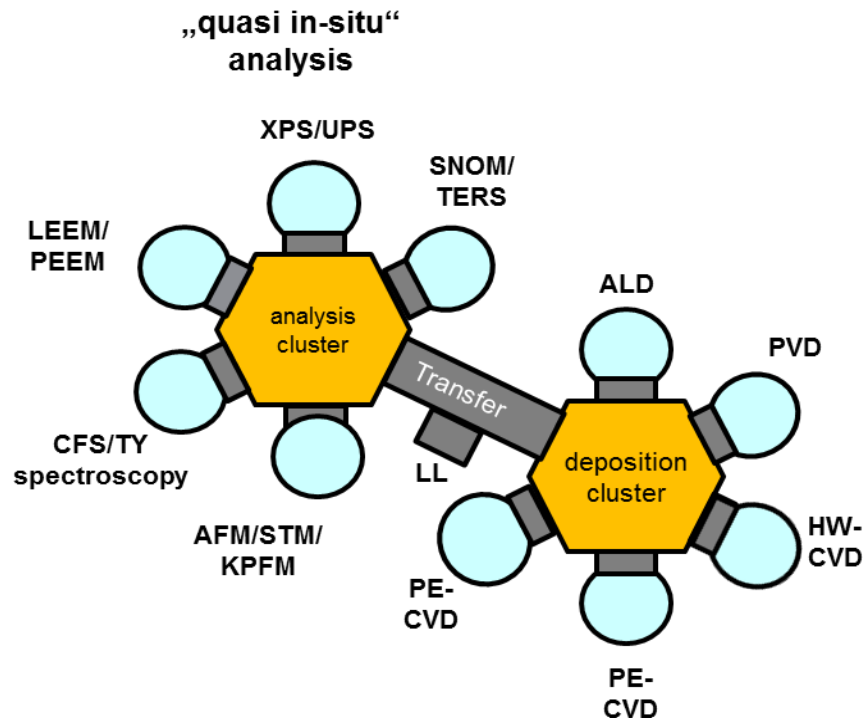


TEM-EDX on FIB-Lamellae



# Instrumentation: JOSEPH

## Juelich Online Silicon Growth Experiment for Photovoltaics



Preparation of complex layers and nanostructures

- In-situ growth analysis
- In-situ microstructure development and optical properties
- chemical composition and element distribution on nm scale
- electronic properties down to nm scale

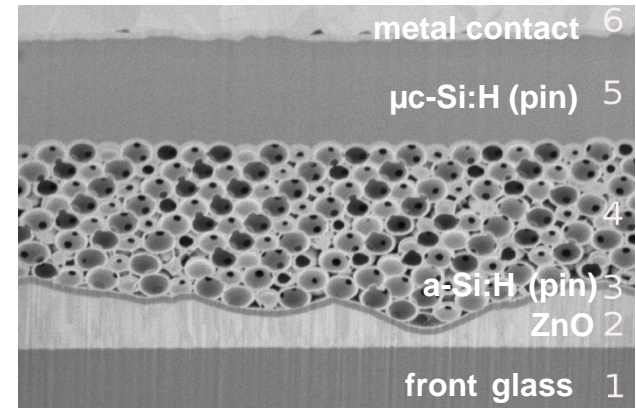
Growth and optoelectronic properties of:

- critical layers
- stacks and interfaces
- embedding of nanostructures for advanced materials and solar cells

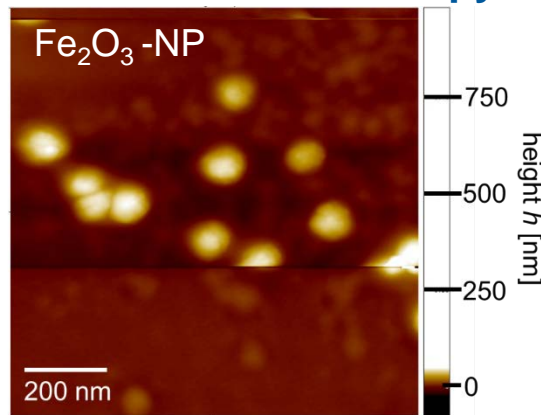
# JOSEPH: Tandem cells and First results of KPFM

Novel thin-film solar cells with  $\eta > 20\%$  will consist of multiple layer stacks combining various optoelectronic functionalities, i.e.

... nanoparticles with high absorption to boost efficiency

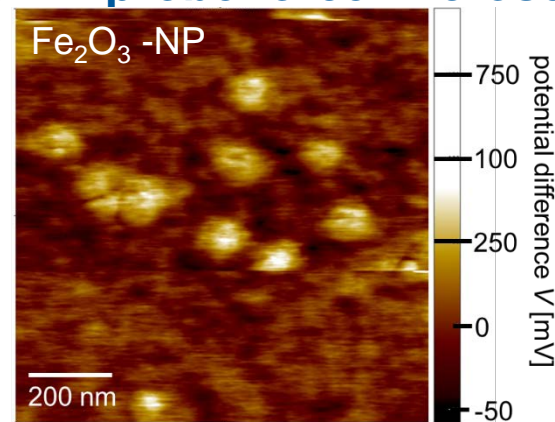


## Atomic force microscopy



Topography with nanoscale resolution

## Kelvin probe force microscopy

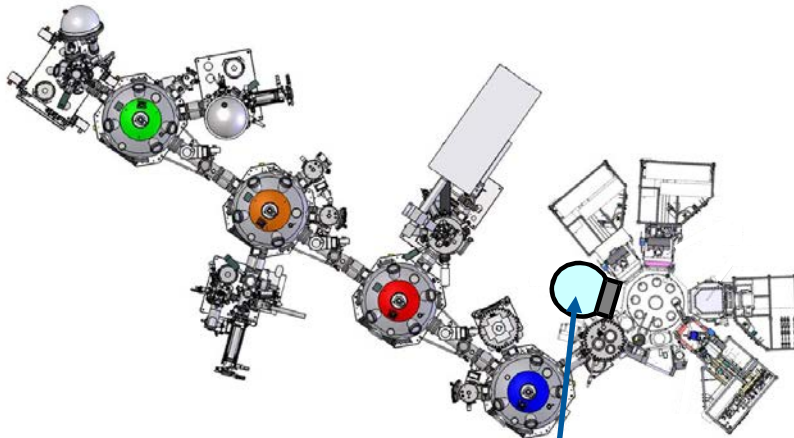


KPFM  $\rightarrow$  contact potential  
Reference material  $\rightarrow$  Work function

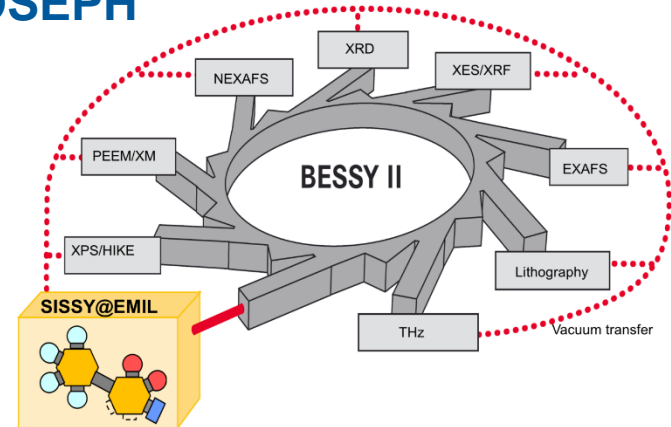
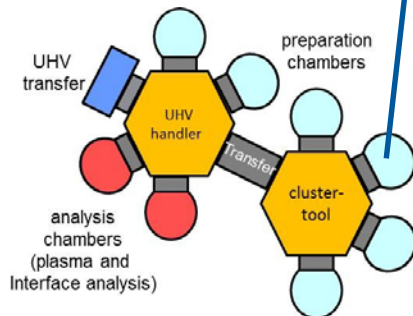
# Cooperation of centers

## SISSY@EMIL and JOSEPH

**SISSY** **HZB** Helmholtz Zentrum Berlin  
Silicon In-Situ lab at the Synchrotron



**JOSEPH** **JÜLICH** FORSCHUNGSZENTRUM  
Jülich Online Silicon growth Experiment for Photovoltaics



- High energy- and depth resolution
- High sensitivity



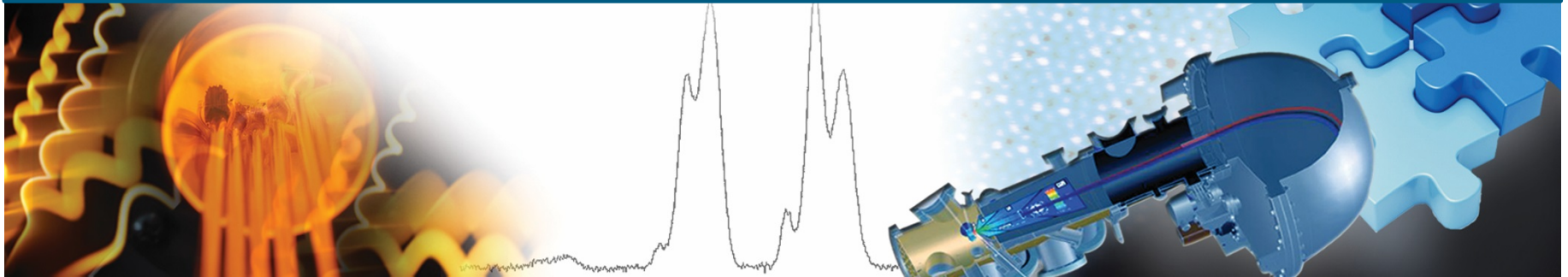
Identical preparation conditions by transfer of processes and chambers

- In-situ diagnosis of layer properties
- In-situ plasma analysis
- Excellent solar cells



- High throughput, broad spectrum of analytical methods
- Specific, industrially relevant production methods
- Preparation and analysis of complete devices

# HEMCP: Contact



[www.hemcp.de](http://www.hemcp.de)



**Prof. Dr. Lorenz  
Singheiser**  
Director of IEK-2 and  
HEMCP scientific  
representative

**Dr. Astrid Besmehn**  
HEMCP scientific  
coordinator and  
Team leader Molecular  
Physics (ZEA-3)