Analysis of Superconducting Thin Films in a Modern FIB/SEM Dual-Beam Instrument

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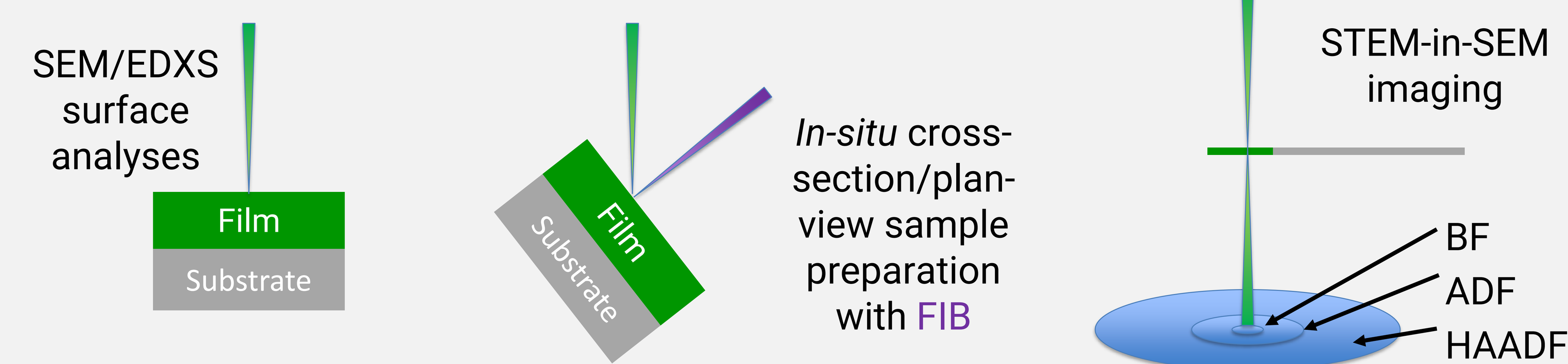
Introduction

- Epitaxially grown superconducting thin films are of interest for fundamental research and applications (e.g. coated conductors[1])
- nm-sized structural defects desirable for flux pinning[2]
  - Increased critical current density
  - Aim: Control of defect formation during film fabrication
- Combined focused-ion-beam/scanning electron microscope (FIB/SEM) instrument for high-throughput microstructural and chemical analysis

Materials & Methods

- Thin films: $Ba(Fe,Co)_2As_2$ (~50 nm) on CaF_2 substrate[3]
  $GdBa_2Cu_3O_{7-\delta}$ (~350 nm) on MgO substrate
- FIB/SEM: Thermo Scientific Helios G4 FX
  - Dedicated STEM holder for *in-situ* lift-out and subsequent STEM-in-SEM imaging (bright-field (BF) and (high-angle) annular dark-field (HAADF)) without breaking microscope vacuum
  - Energy-dispersive x-ray spectroscopy (EDXS): Bruker X-Flash 6|60



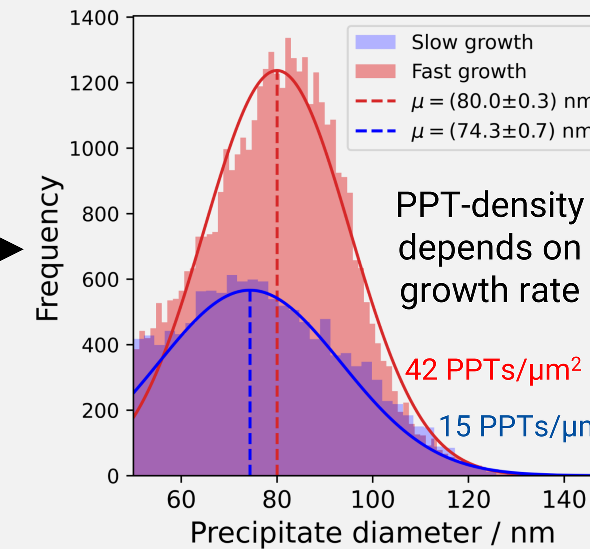
Summary

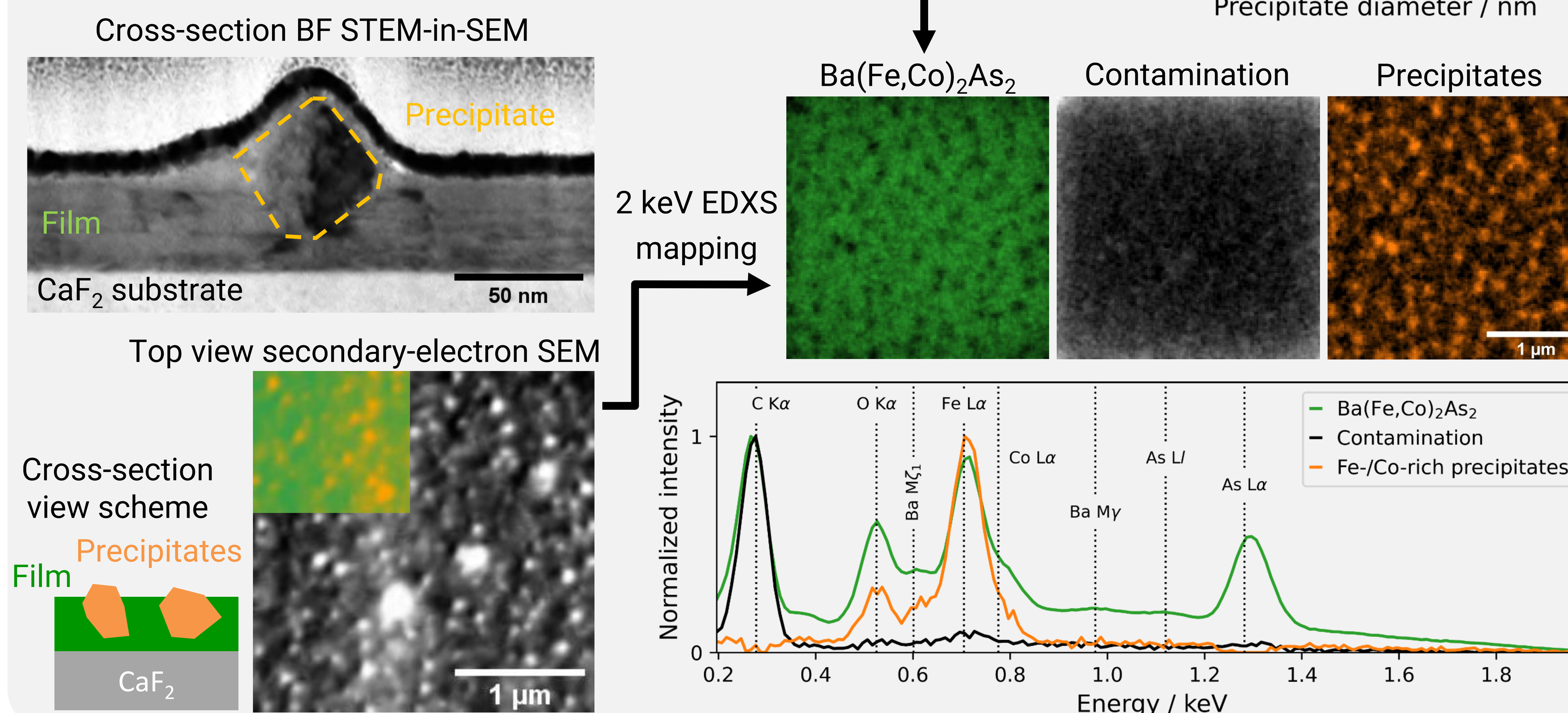
- Modern **FIB/SEM** instruments → **high-throughput** structural and chemical analyses down to nm-scale
- **Reactive materials** examined without exposure to ambient atmosphere

$Ba(Fe,Co)_2As_2$

Analysis of surface precipitates

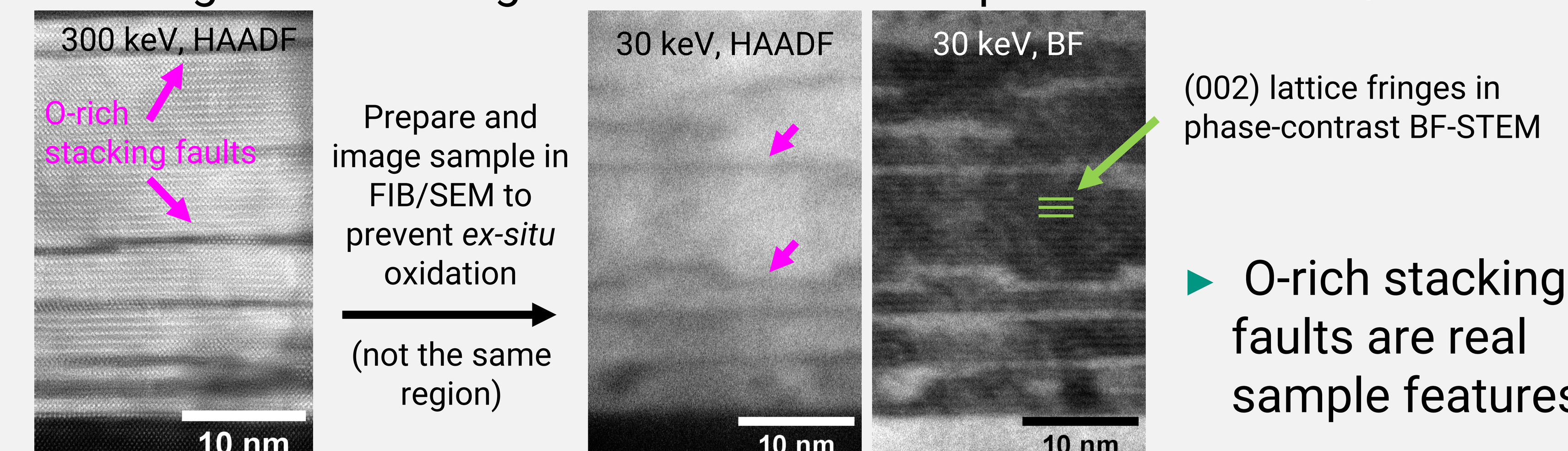
- Top-view SEM and cross-section STEM-in-SEM imaging of precipitates (PPTs)
- Analysis of size and surface coverage of PPTs with „Trainable Weka Segmentation"[3,4]
- Low-energy EDXS with non-negative matrix factorization[5] to separate PPTs/film signals





STEM-in-SEM of stacking faults

- TEM lamella preparation and subsequent STEM-in-SEM imaging without exposing the sample to air to exclude possible oxidation stacking faults[3] during transfer of TEM sample to dedicated S/TEM

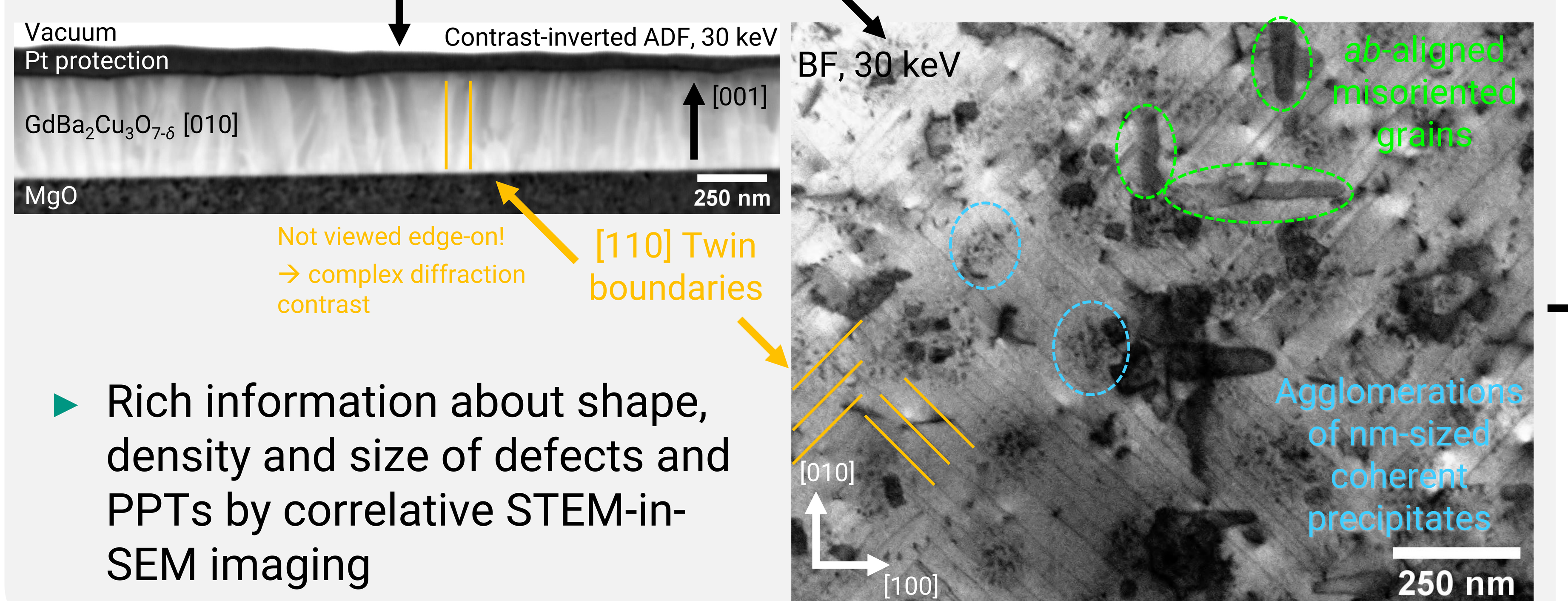


- O-rich stacking faults are real sample features

$GdBa_2Cu_3O_{7-\delta}$

Cross-section and plan-view imaging

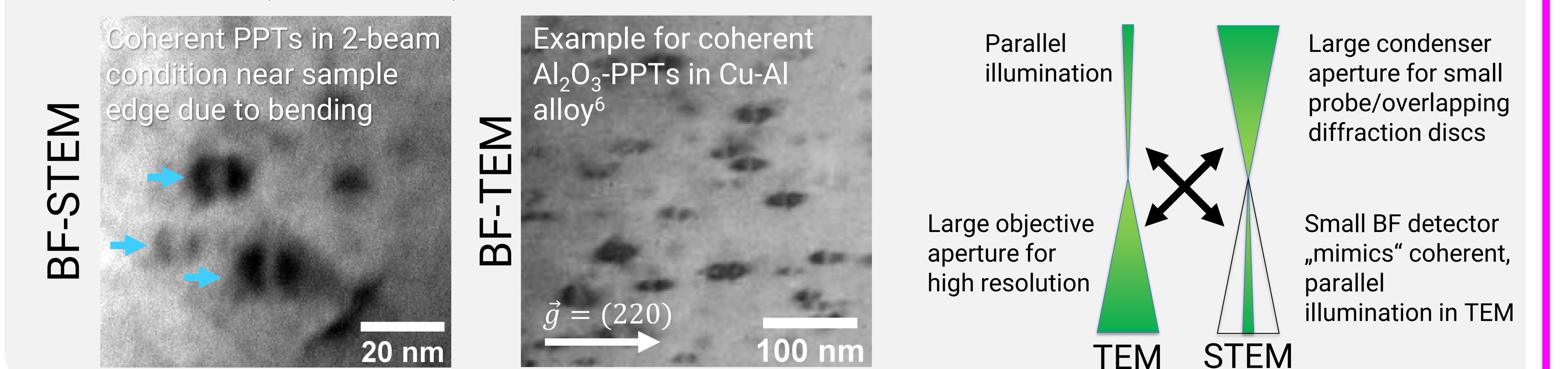
- Rapid TEM sample preparation by FIB and STEM-in-SEM imaging to correlate **cross-section** and **plan-view** imaging directions



- Rich information about shape, density and size of defects and PPTs by correlative STEM-in-SEM imaging

Coherent precipitates

- Ashby-Brown diffraction contrast[6] from coherent precipitates under two-beam condition in BF-(S)TEM
  - Same (diffraction-)contrast for STEM and TEM if reciprocity theorem is fulfilled



[1] Obradors and Puig, *Supercond. Sci. Technol.* **27** (2014) 044003
[2] Feighan et al., *Supercond. Sci. Technol.* **30** (2017) 123001
[3] Grünewald et al., *Supercond. Sci. Technol.* **34** (2021) 035005
[4] Arganda-Carreras et al., *Bioinformatics* **33** (2017) 2424–2426
[5] Jany et al., *Nano Lett.* **17** (2017) 6520–6525
[6] Ashby and Brown, *The Philosophical Magazine* **8** (1963) 1649–1676

More analyses on these defects and PPTs are on the poster **„Electron microscopic investigation of post-annealed superconducting $GdBa_2Cu_3O_{7-\delta}$ thin films on MgO"**

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