Leibniz Institute for Solid State and Materials Research Dresden

In situ and in operando structural studies by Leibniz IFW Dresden scientists at PETRA III.

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Overview

Research topics:

- ➢ Materials for electrochemical energy storage
- ➢ Solidification and phase transformations
- ➢ Additive manufacturing
- ➢ Short-range order in liquid and amorphous state

Experimental techniques:

- X-ray diffraction & absorption spectroscopy
- Predominantly in situ and operando studies
- Development of experimental setup

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Materials for electrochemical energy storage

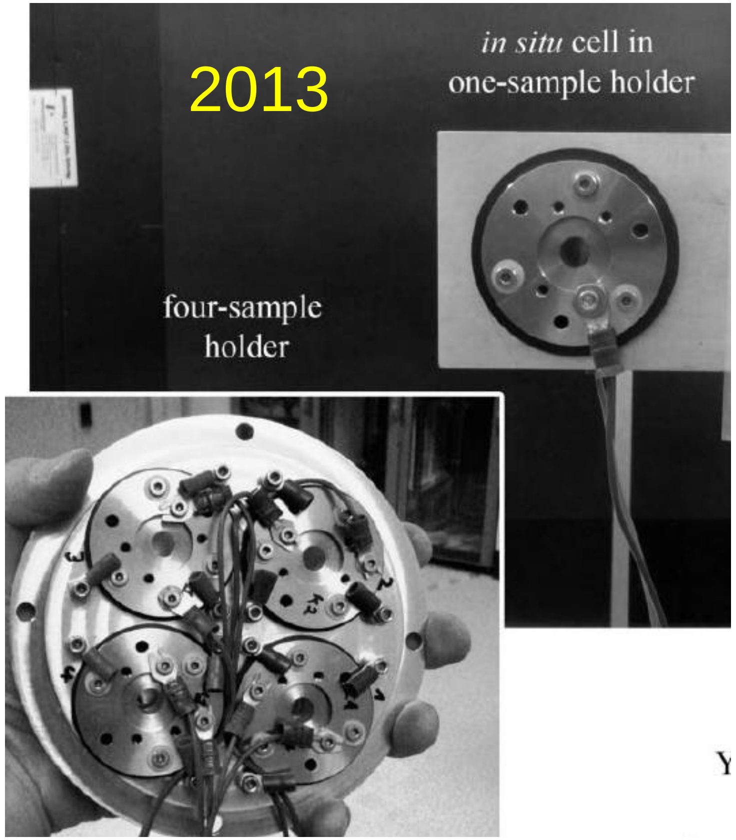
In operando studies of structural transformations and reaction mechanisms during charging and discharging

- XRD measurements at P02.1
- X-ray absorption spectroscopy at P64 & P65
- Instrumentation development:
  - high-resolution powder diffractometer at P02.1
  - sample holders

Contact: Dr. D. Mikhailova, IFW Dresden

Materials for electrochemical energy storage

Instrument design at IFW Dresden:
electrochemical cells & cell holders



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Advances in *in situ* powder diffraction of battery materials: a case study of the new beamline P02.1 at DESY, Hamburg

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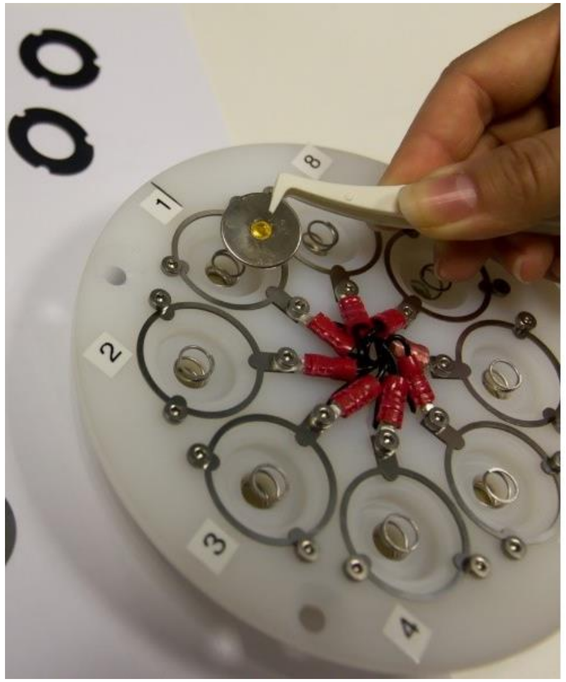
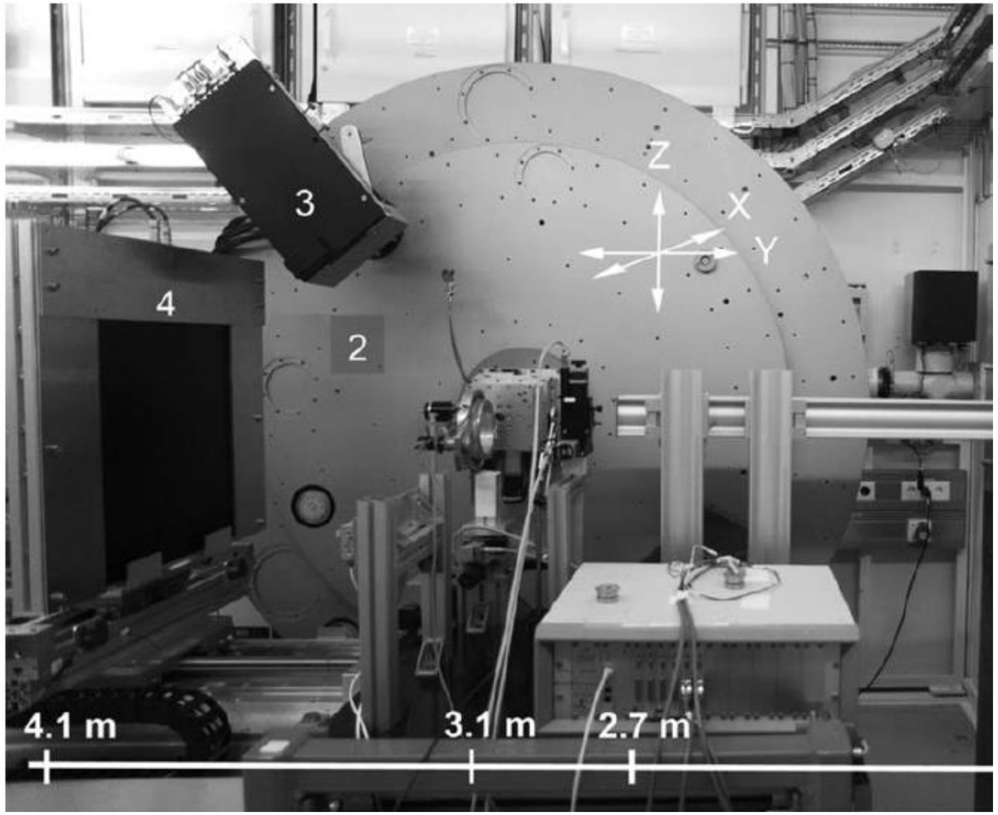


eight cells holder

(2023)

Materials for electrochemical energy storage

Instrument design at IFW Dresden:
2 – three-axis goniometer at P02.1
3 – multi-channel high-resolution detector with 10 analyzer crystals



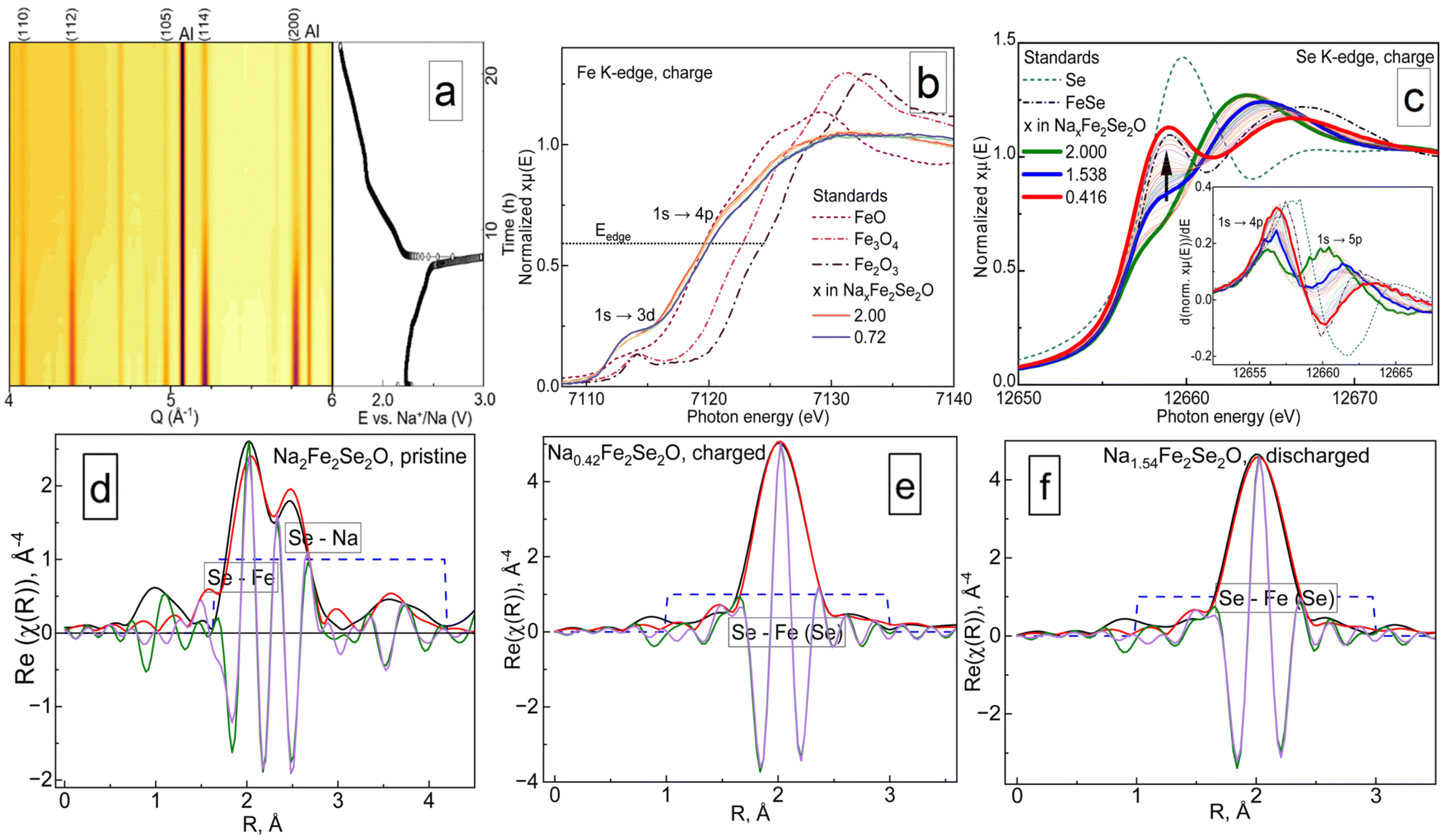
Multi-analyser detector (MAD) for high-resolution and high-energy powder X-ray diffraction

Alexander Schökel,[a,b] Martin Etter,[b] Andreas Berghäuser,[c] Alexander Horst,[d] Dirk Lindackers,[d] Thomas A. Whittle,[e] Siegbert Schmid,[e] Matias Acosta,[f] Michael Knapp,[a]* Helmut Ehrenberg[a] and Manuel Hinterstein[a]

Synchrotron JSR
JOURNAL OF SYNCHROTRON RADIATION

Materials for electrochemical energy storage

$Na_2Fe_2Se_2O$ double anti-perovskite: XRD at P02.1 & XAS at P64



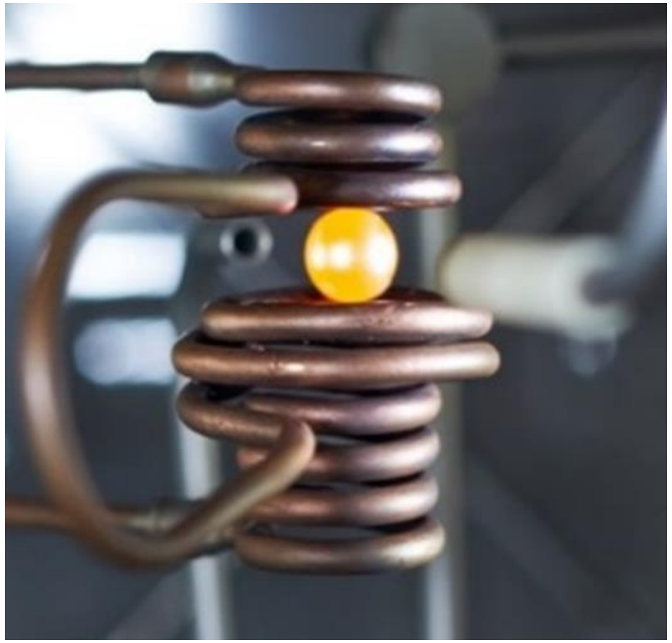
(a) XRD contour plots measured during charge-discharge cycle

(b) Fe and Se edges XAS spectra

(c,d,f) Se-edge Ksi(R)-curves for pristine, charged and discharged state

M.V. Gorbunov, T. Doert, D. Mikhailova, Chem. Commun., 2023

Solidification of undercooled metallic melts



Levitated metallic sample

Scope:

- Effect of melt undercooling on phase formation sequence
- Solid state transformations during cooling
- Crystal growth in dependence on undercooling

Electromagnetic levitation:

- inductive heating → high temperatures can be reached
- no crucible → no reaction, no heterogeneous nucleation

Contact: Dr. I. Kaban, IFW Dresden

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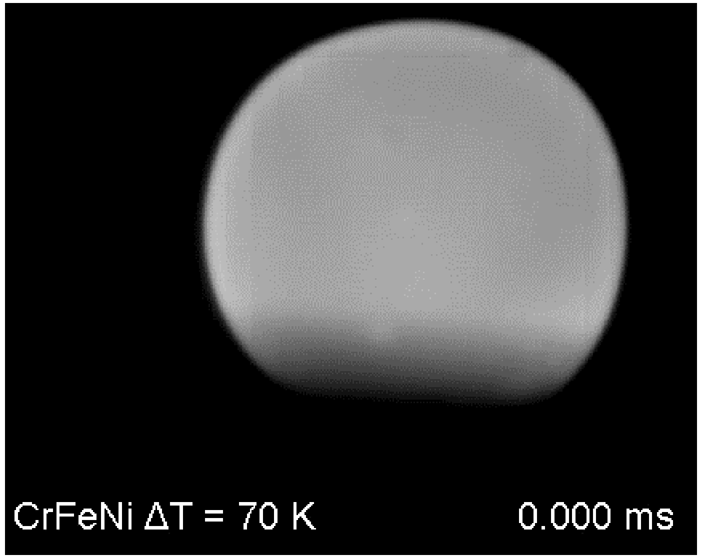
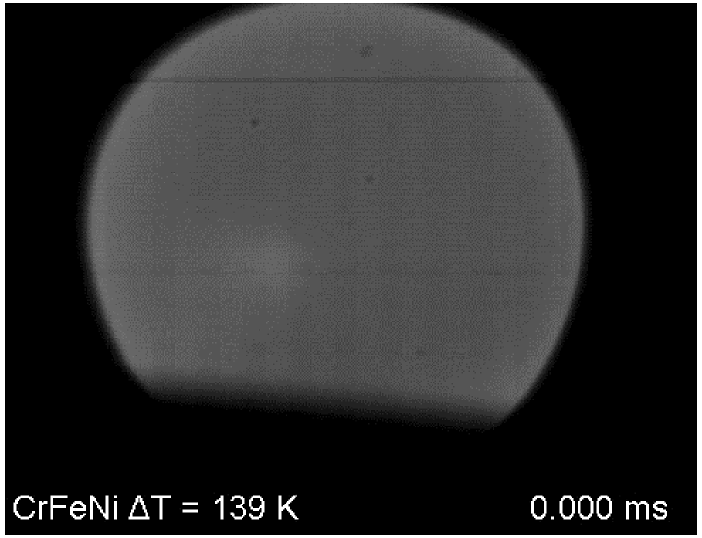
Solidification of undercooled metallic melts

High-speed videos taken from CrFeNi at 50.000 fps



small undercooling (70 K):

only one crystalline phase is observed



large undercooling (139 K)

there is a secondary phase

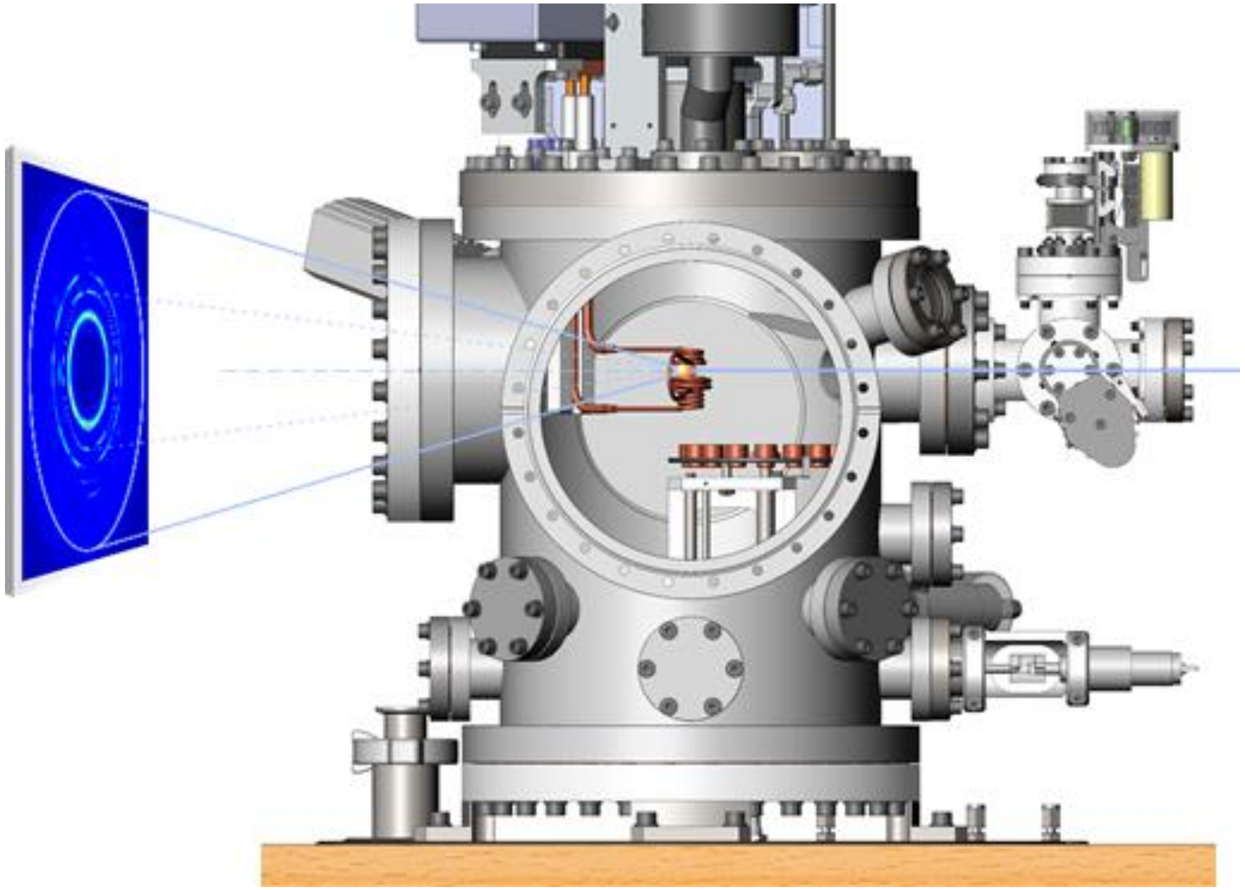
high-speed video → crystal growth rate

A. Andreoli et al. Acta Mater., 2021

What are the crystalline phases?

Solidification of undercooled metallic melts

EML facility of IFW Dresden



HE XRD

In situ measurements:

→ structure, phase formation and transformations

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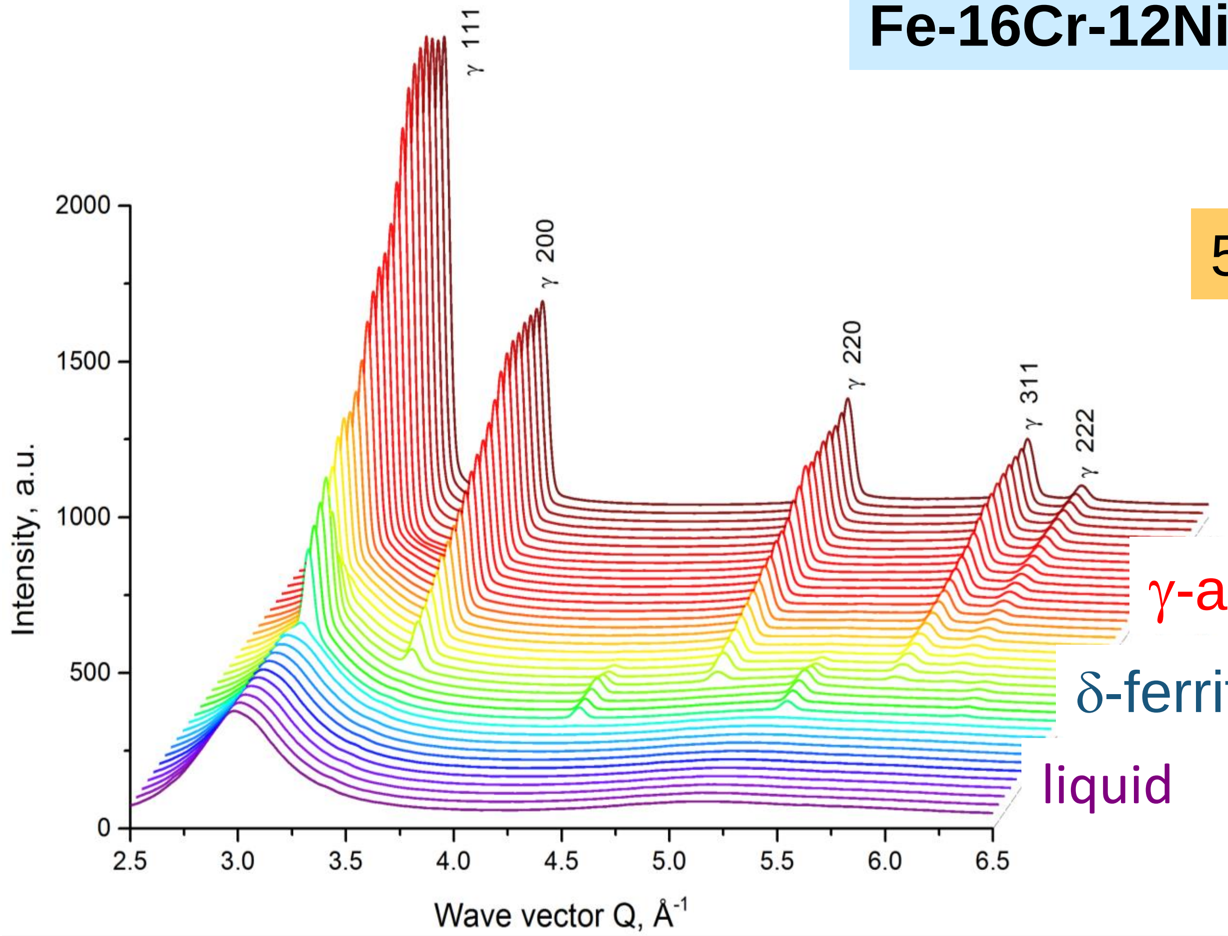
Solidification of undercooled metallic melts

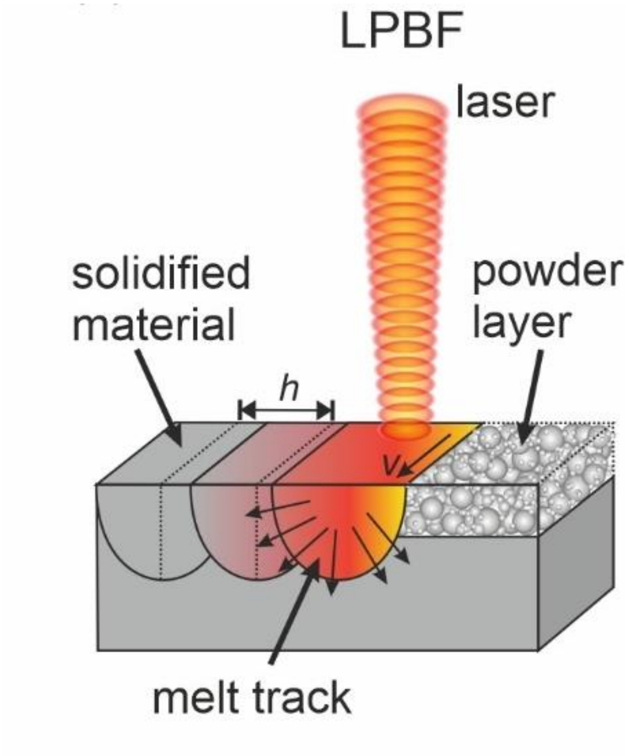
Fe-16Cr-12Ni: XRD at P07, PILATUS detector

50 K undercooling: primary phase δ-ferrite



D. Matson et al. JOM 2017

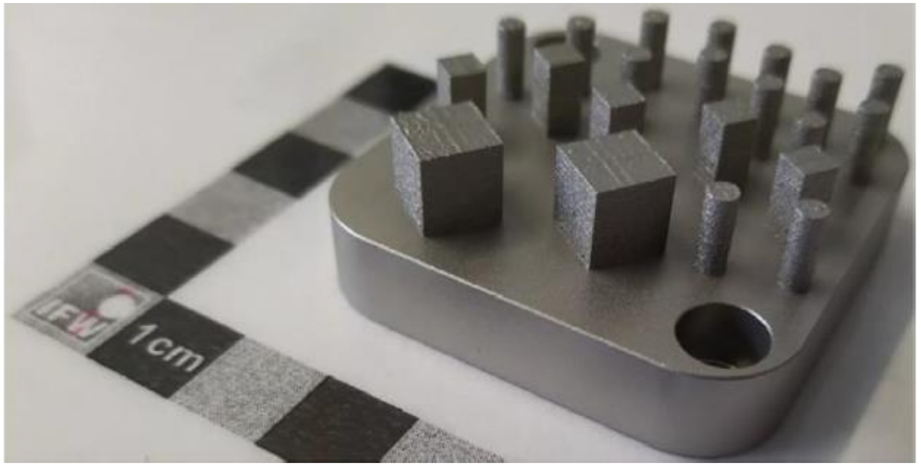
Additive manufacturing



Schematic of LPBF processing

Laser powder bed fusion

- Interplay of different physical and chemical phenomena
- Microstructure & properties depend on powder processing and solidification conditions in molten pool



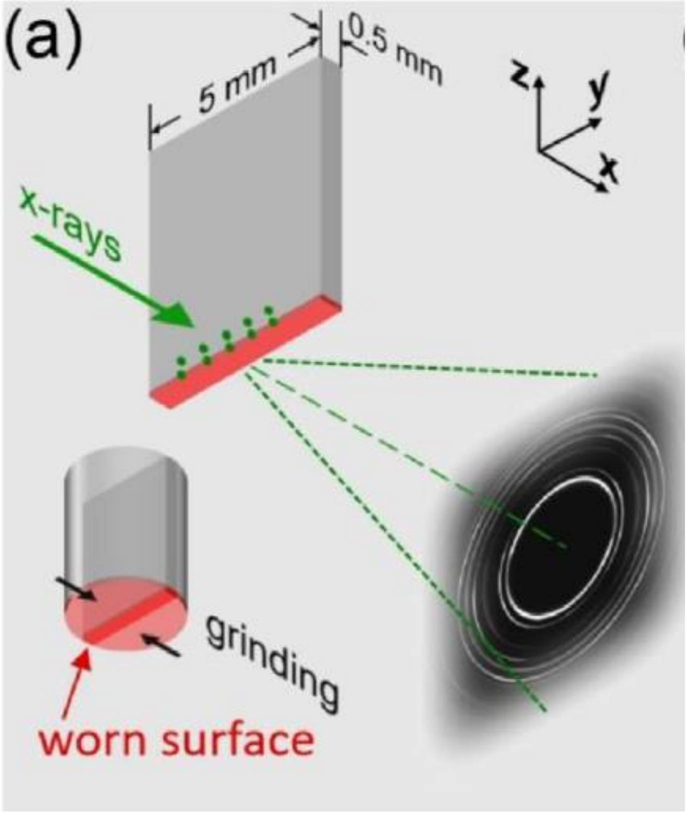
3D printed Fe85Cr4Mo1V1W8C1 samples

K. Kosiba et al., JMST, 2023

Contact: Prof. Dr. Julia K. Hufenbach, IFW Dresden

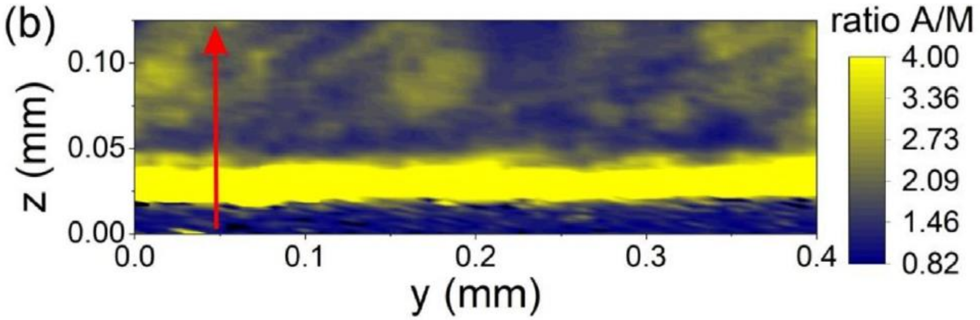
Additive manufacturing

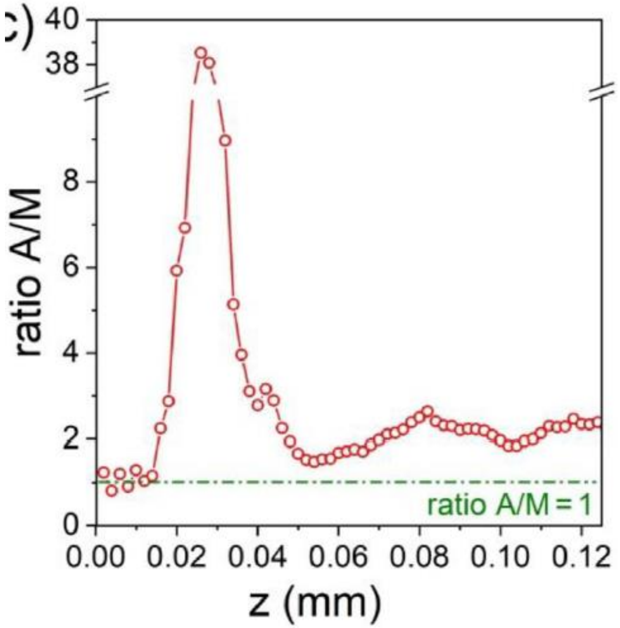
XRD micro-mapping of LPBF-fabricated Fe85Cr4Mo1V1W8C1 sample after wear test



Beamline: P21.2

Beam size: 2 x 9 µm^2

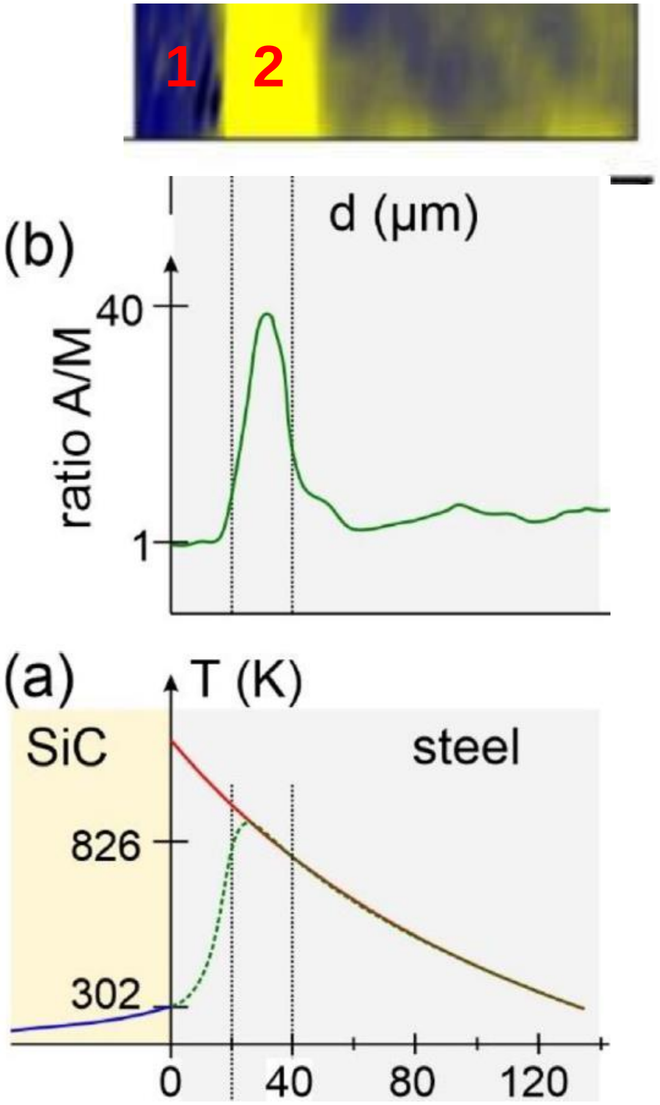




Ratio of austenite and martensite in the sample near worn surface

K. Kosiba et al., JMST, 2023

Additive manufacturing



Transformation mechanisms

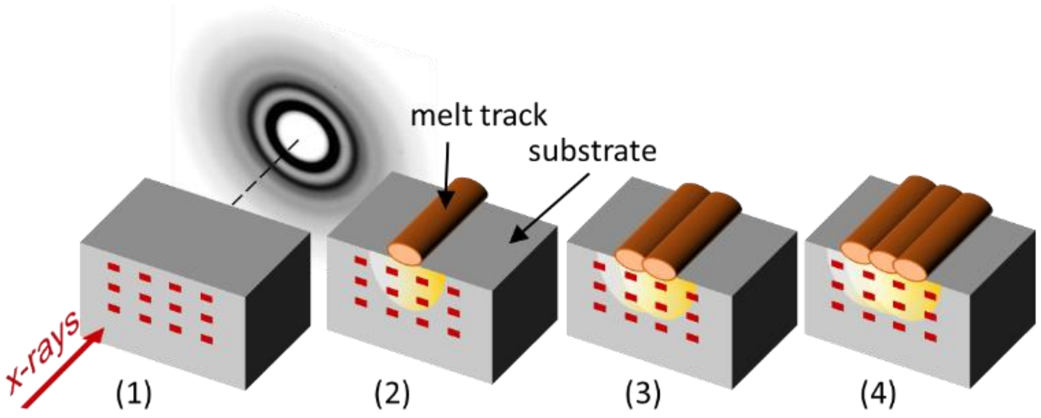
Region 1: Wear or friction-induced transformation of retained austenite into martensite near worn surface.

Region 2: M-A transformation in the region of elevated temperature

Possible temperature profile in the sample during wear test

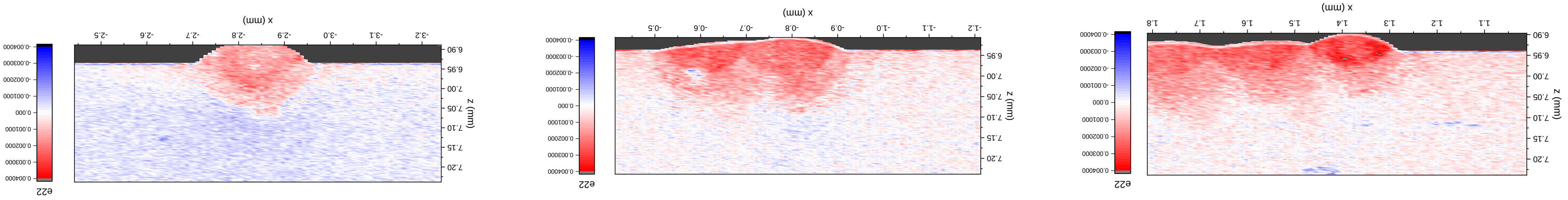
K. Kosiba et al., JMST, 2023

Additive manufacturing



Cumulative effect of melt trucks on residual stress

- HE XRD studies at P21.2 (unpublished)



Ex situ strain mapping of Zr-based BMG with increasing number of tracks

Contact: Dr. K. Kosiba, Dr. S. Scudino, IFW Dresden

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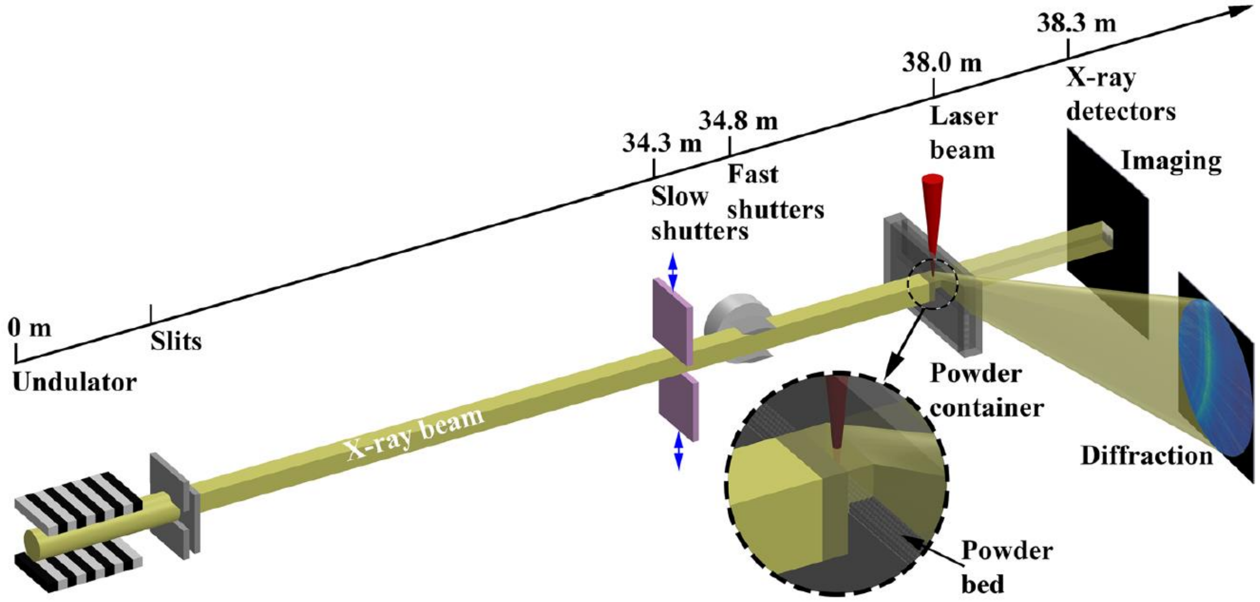
Additive manufacturing



X-ray imaging and diffraction measurements during 3D printing at Advanced Photon Source, USA [C. Zhao et al., Sci. Rep. 2017].

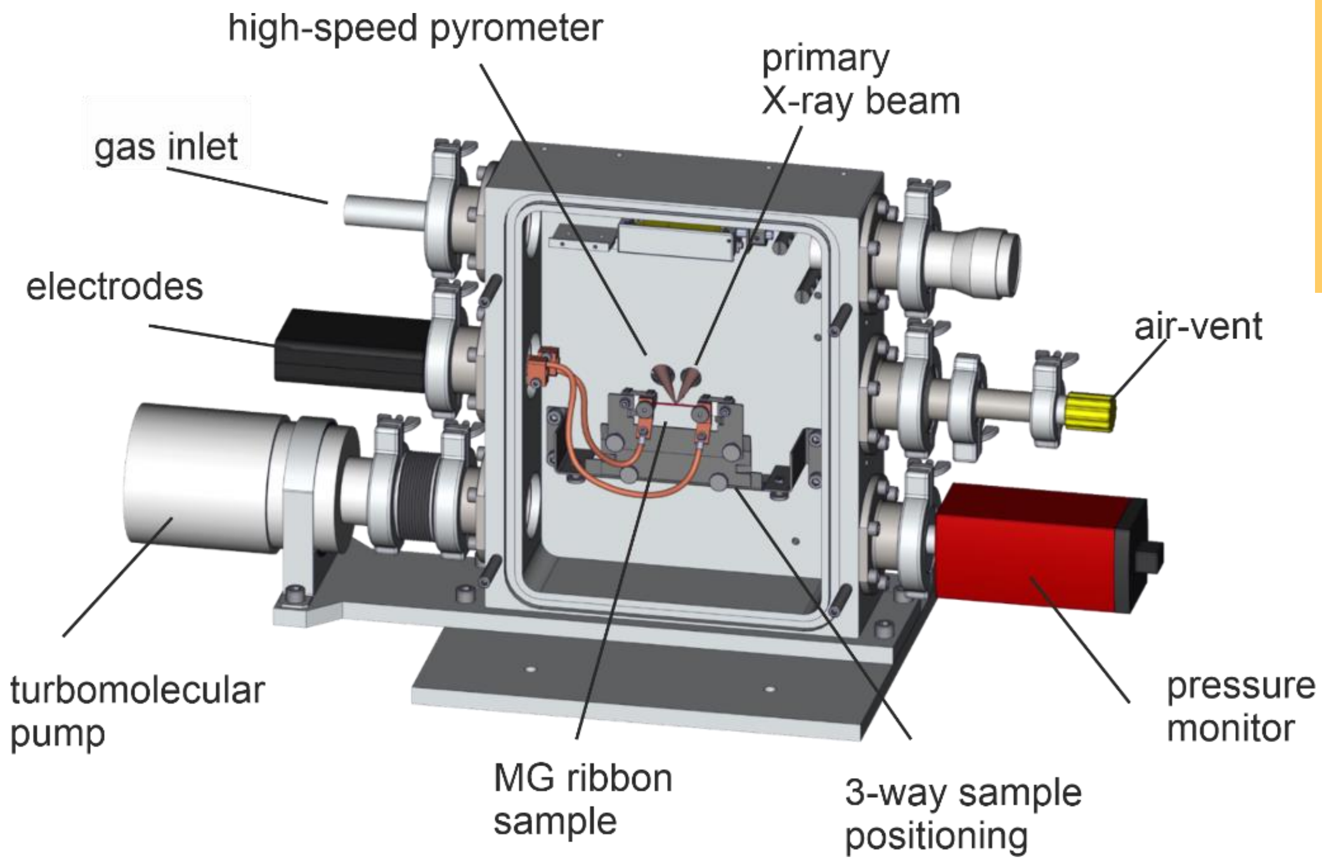
Future goals (wishes) for PETRA III:

- wide angle XRD
- small angle X-ray scattering
- X-ray imaging
- computed tomography (RAC project submitted)

Contact at IFW Dresden

- Dr. D. Mikhailova (electrochemical energy storage)
- Prof. Dr. J.K. Hufenbach, Dr. K. Kosiba, Dr. S. Scudino (additive manufacturing)
- Dr. I. Kaban (solidification, phase transformations)

Phase transformations upon rapid annealing



- Resistance
- Temperature (pyrometer or thermocouple)
- Structure (high-energy XRD & fast detector)

Key parameters:

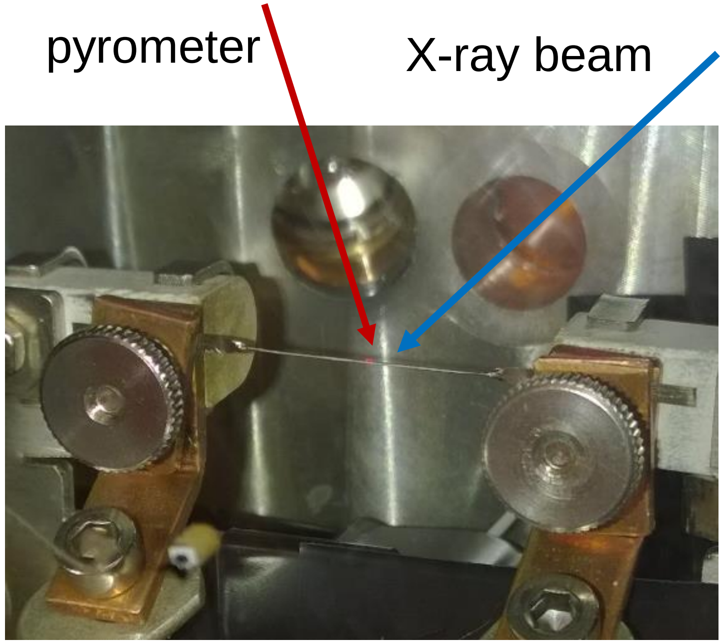
Vacuum (5×10^{-4} mbar) or inert gas (Ar, He)

Heating rate: ~10^1 – 10^5 K s^{-1}

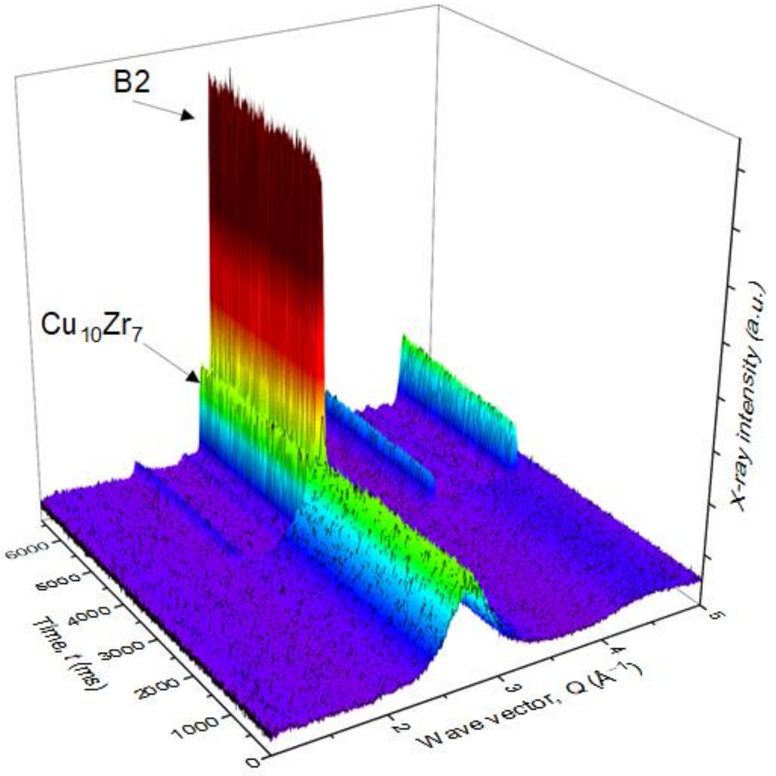
Cooling rate: 10^2 – 10^3 K s^{-1} (in He)

Contact: Dr. I. Kaban, IFW Dresden

Phase transformations upon rapid annealing



Rapid heating of $Cu_{47.5}Zr_{47.5}Al_5$ metallic glass (150 K/s)



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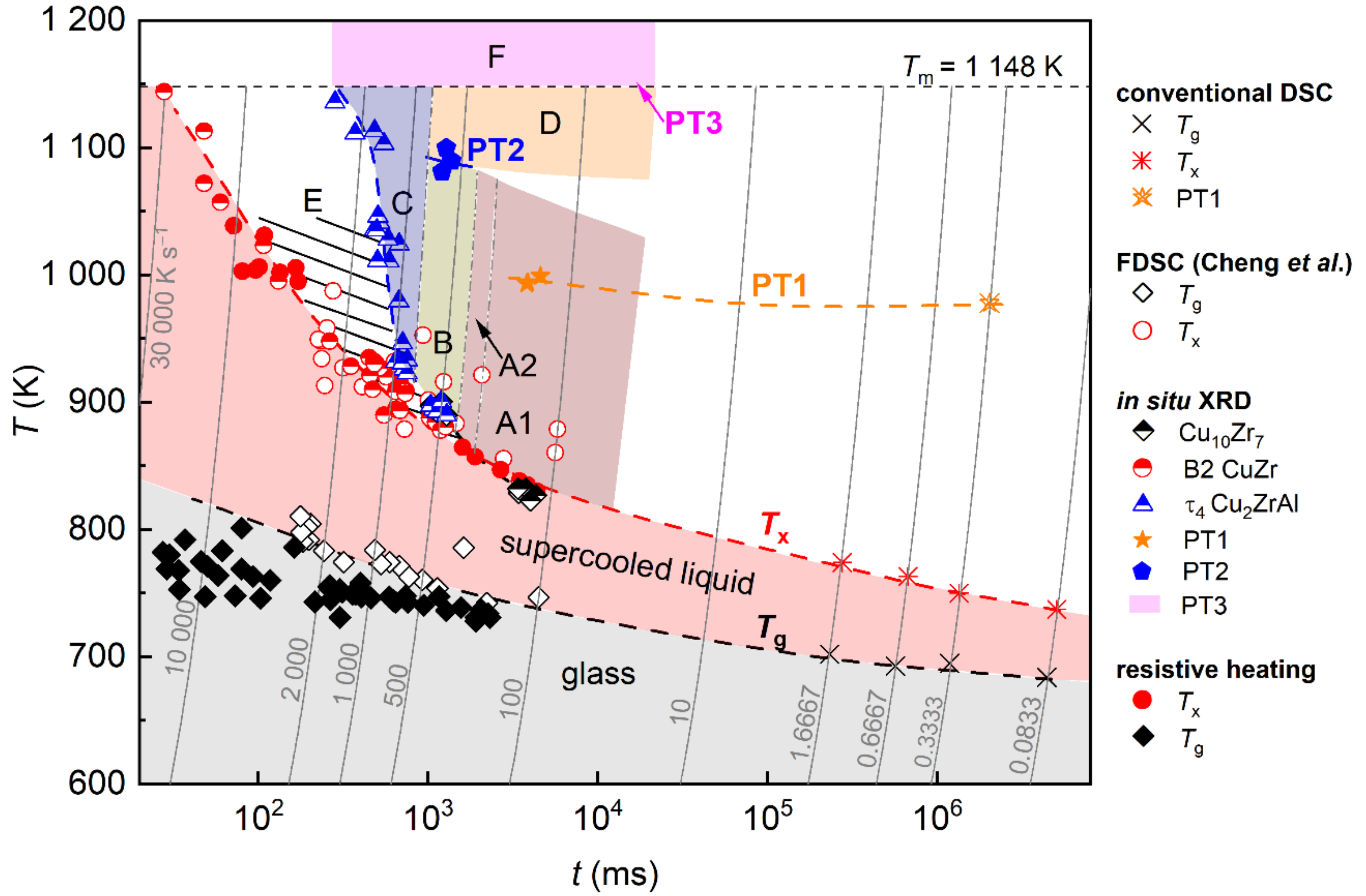
Phase transformations upon rapid annealing

CHT diagram for $Cu_{47.5}Zr_{47.5}Al_5$ metallic glass



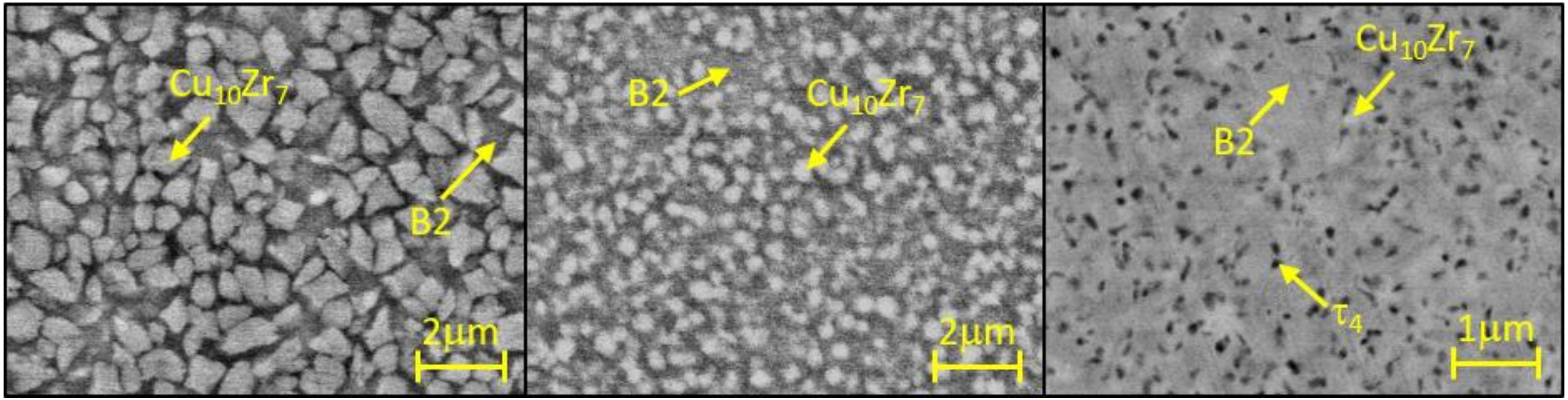
PT1: $Cu_{10}Zr_7 \rightarrow$ B2 CuZr

PT2: $Cu_{10}Zr_7 \rightarrow \tau_4$

PT3: B2 + $\tau_4 \rightarrow \tau_3$ + L

Phase transformations upon rapid annealing

Tailoring microstructure by rapid heating of $Cu_{47.5}Zr_{47.5}Al_5$ MG to a given temperature



T_x: MG → $Cu_{10}Zr_7$ + B2 CuZr

PT1: $Cu_{10}Zr_7$ → B2 CuZr

PT2: $Cu_{10}Zr_7$ → τ_4