

DAS X-RAY CORELAB AM HZB

Instrumentenpool und Röntgenschulen

Michael Tovar

Bruker-Anwendertreffen, Bremen

20.09.2022

Powder
Single crystal
Non-ambient

Outline

1. Das Helmholtz-Zentrum Berlin
2. Aufstellung und Ziele des X-ray Corelabs
3. Virtueller Rundgang
4. Weitere Methoden
5. Temperaturabhängige Röntgendiffraktion
6. Schulen
 - a) X-ray school
 - b) Rietveld school
7. Fazit

1. DAS HELMHOLTZ-ZENTRUM BERLIN

Forschungsschwerpunkte

- Photovoltaik
- Dünnschicht-Technologien
- Katalysatoren/Grüner Wasserstoff/CO₂-Umwandlung
- Solare Brennstoffe
- Batteriematerialien
- Quantenmaterialien
- Beschleunigerphysik & -technologien



WILHELM-CONRAD-RÖNTGEN-CAMPUS



LISE-MEITNER-CAMPUS

1. DAS HELMHOLTZ-ZENTRUM BERLIN

Corelabs

- Energy Materials In-Situ lab (EMIL)
- X-ray Corelab
- Correlative Microscopy and Spectroscopy
- Hybrid Silicon Perovskite (HYSPRINT)
- Kompetenzzentrum Photovoltaik (PVcomB)
- Quantum Materials



2. X-RAY CORELAB: AUFSTELLUNG UND ZIELE

- Institutsübergreifende Zurverfügungstellung von Mess- und Auswertemethoden der Röntgendiffraktion
- Steuerungskomitee berät über Investitionen und Laborentwicklung
- Jahresbudget: 120.000€

Lise-Meitner-Campus
(M. Tovar, R. Gunder / H. Prell)

- Klassische Pulverdiffraktion
- Textur, Epitaxie
- Einkristalldiffraktion
- Non-ambient



Wilhelm-Conrad-Röntgen Campus
(Ch. Genzel, R. Mainz, M. Klaus)

- Metal Jet Source
- Microstrain analysis

2. X-RAY CORELAB: AUFSTELLUNG UND ZIELE

Aufbau des Labors 2015



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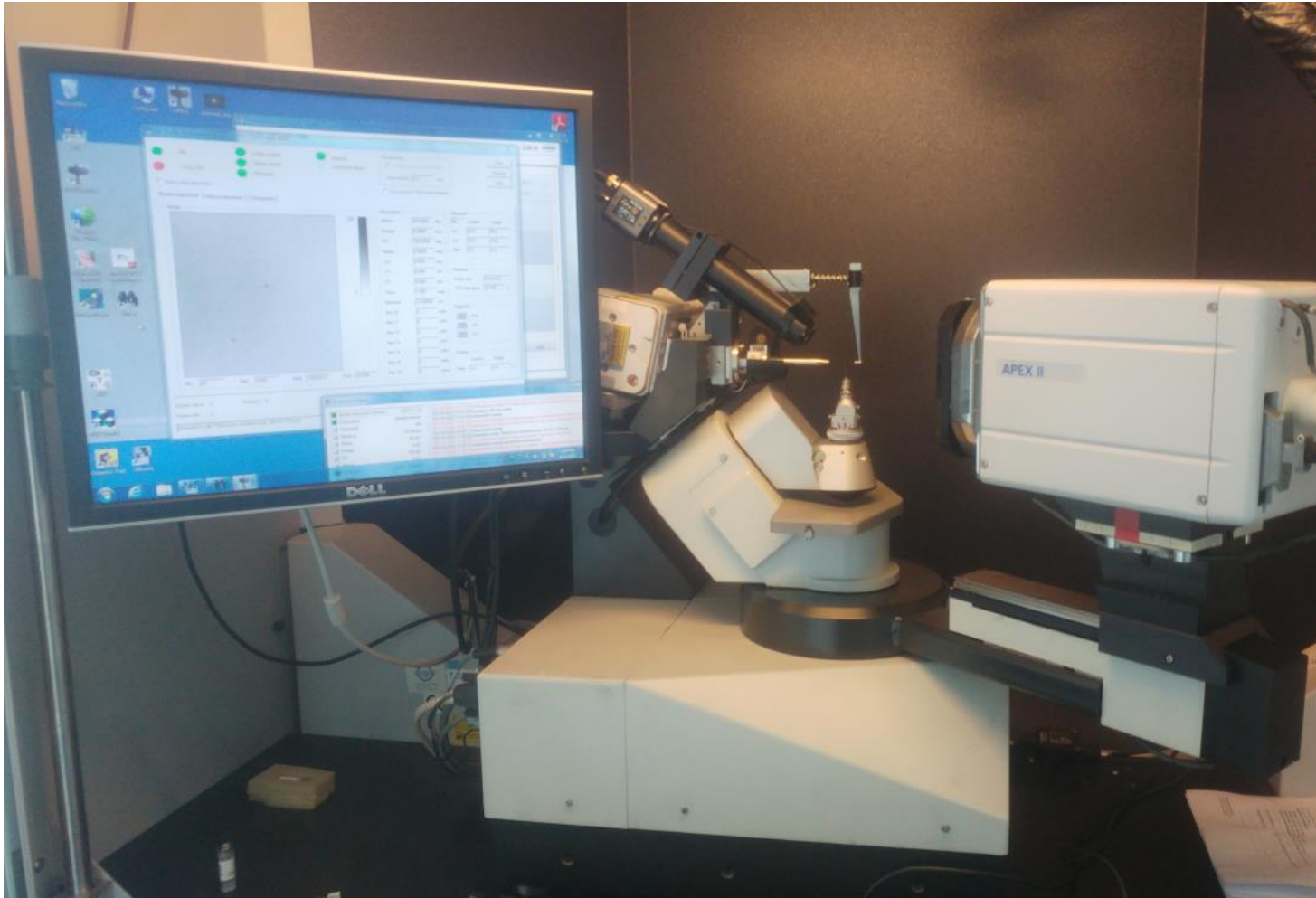


3. VIRTUELLER RUNDGANG

(Klick aufs Bild öffnet Labor-Webseite)



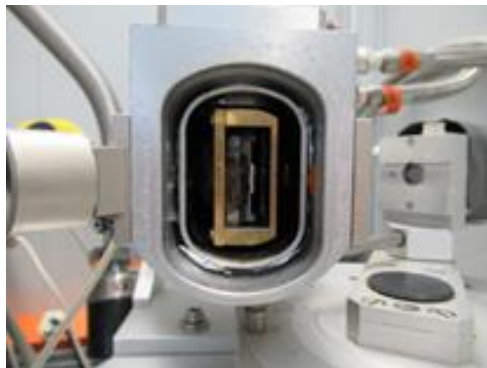
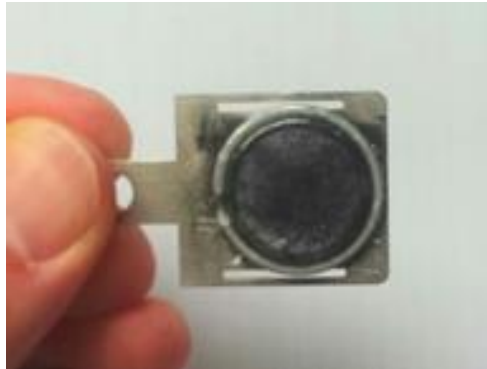
4. WEITERE METHODEN: BRUKER KAPPA APEX II



- Transfer von der Universität Glasgow
- Wiederaufbau und Modernisierung 2019-2021



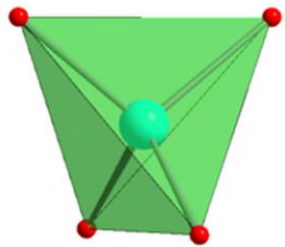
4. WEITERE METHODEN: TIEFTEMPERATUR-GUINIER-DIFFRAKTOMETER



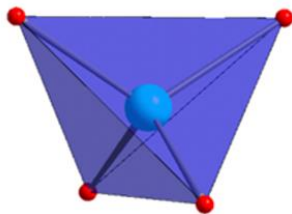
Tieftemperatur-Guinier-Diffraktometer: Huber-Goniometer in asymmetrischer Transmission, Cu-K_{α1}-Strahlung, He-Kryostat für Temperaturen von 12-300 K

5. TEMPERATURABHÄNGIGE DIFFRAKTION

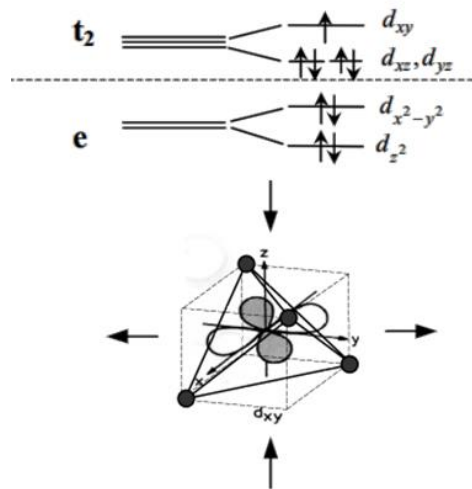
Structural distortions in mixed spinels $\text{Ni}_{1-x}\text{Cu}_x\text{Cr}_2\text{O}_4$



Ni^{2+}O_4



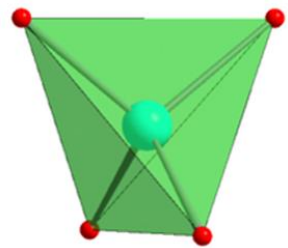
Cu^{2+}O_4



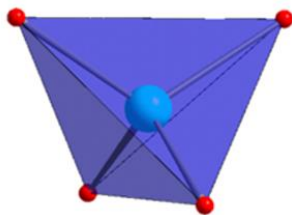
A. Wold, R. J. Arnett, E. Whipple, and J. B. Goodenough, J. Appl. Phys. 34, 1085 (1963)

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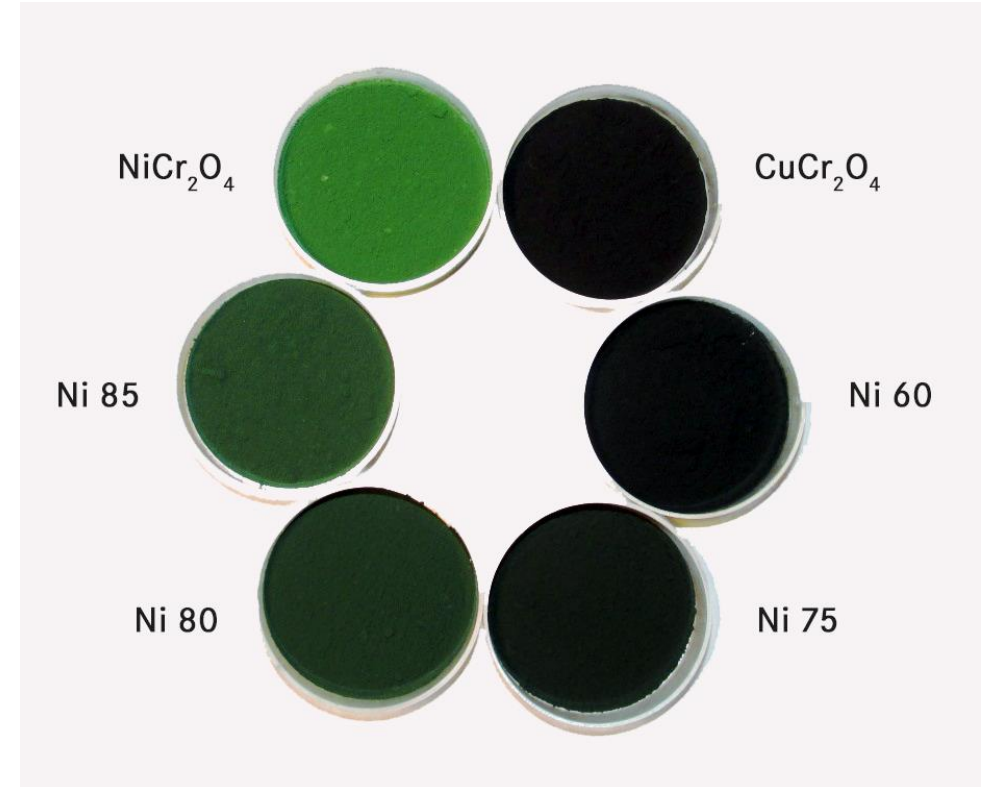
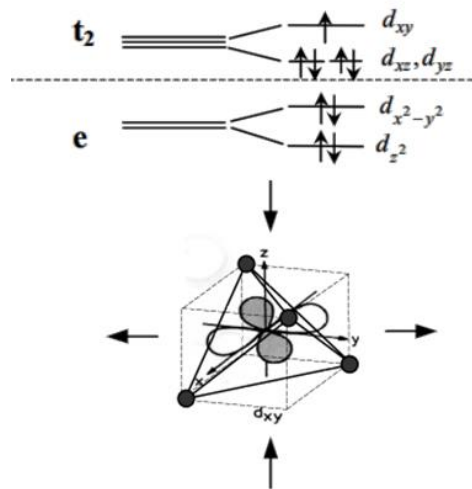
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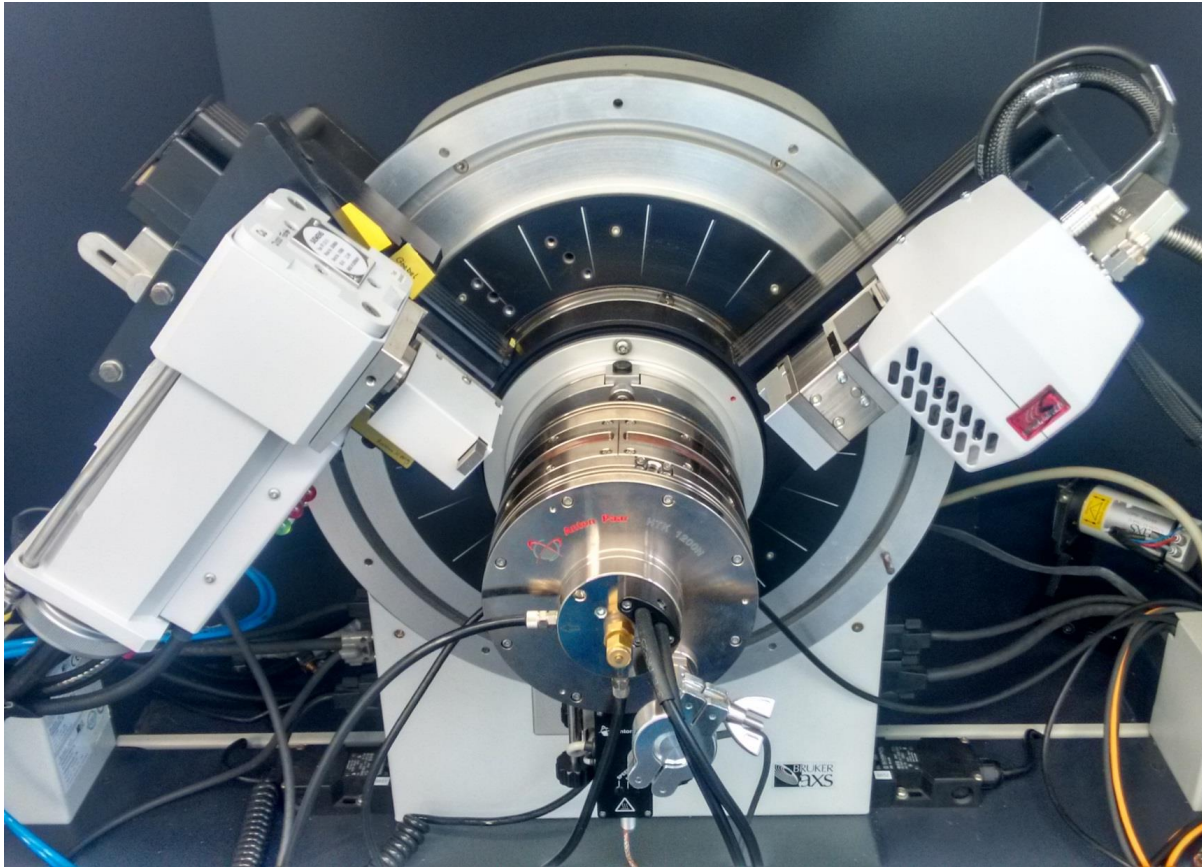


A. Wold, R. J. Arnett, E. Whipple, and J. B. Goodenough, *J. Appl. Phys.* 34, 1085 (1963)

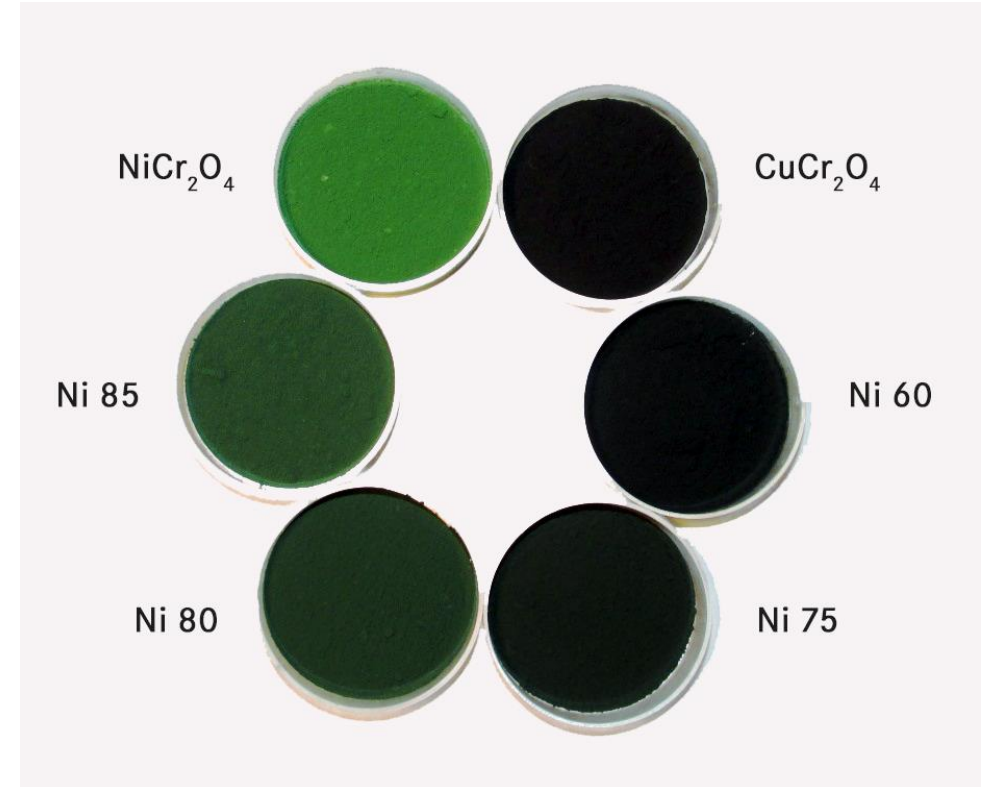
M. Tovar, R. Torabi, C. Welker, F. Fleischer, *Physica B*, 385–386, 196 (2006)

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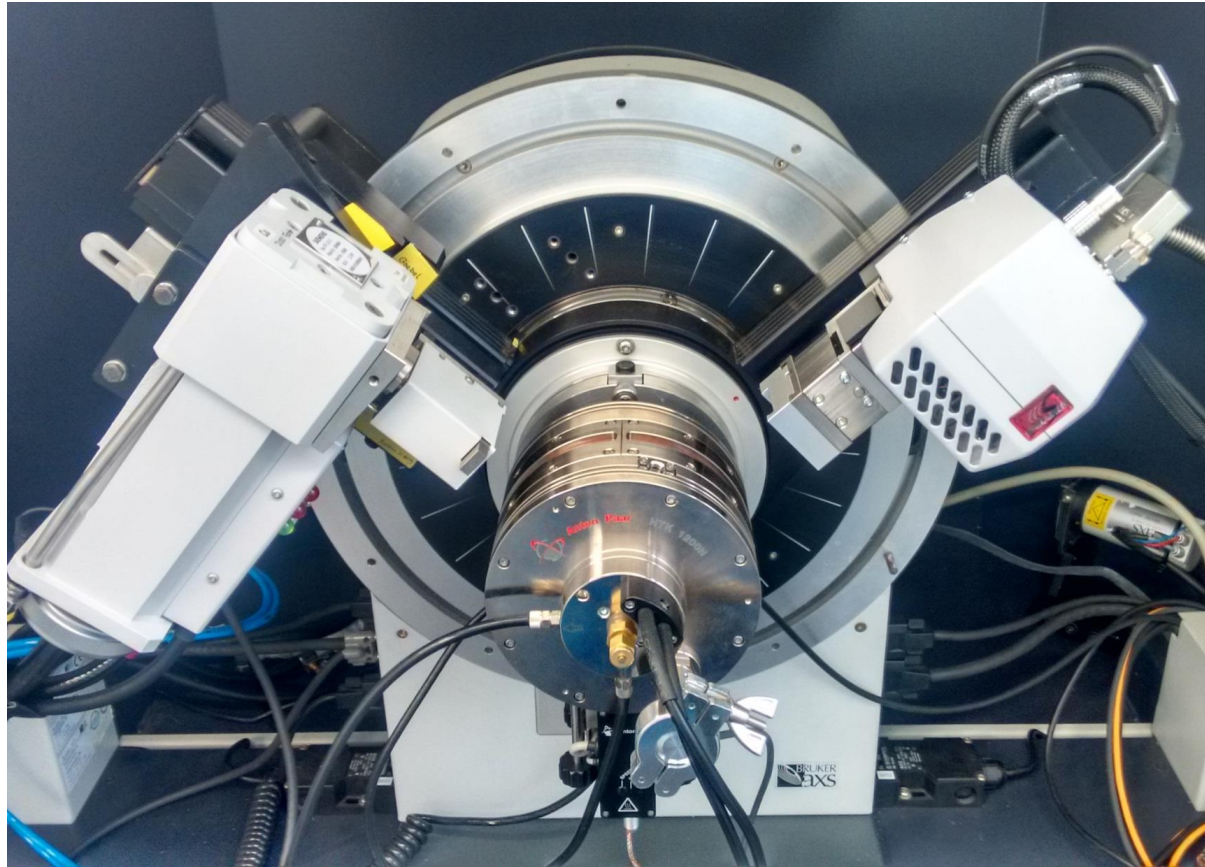


BRUKER D8 WITH ANTON PAAR HTK 1200N

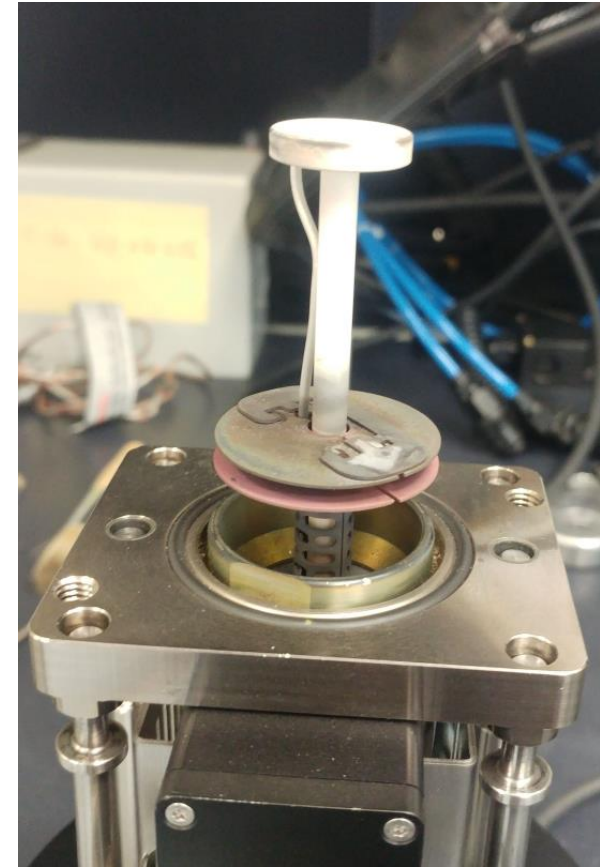


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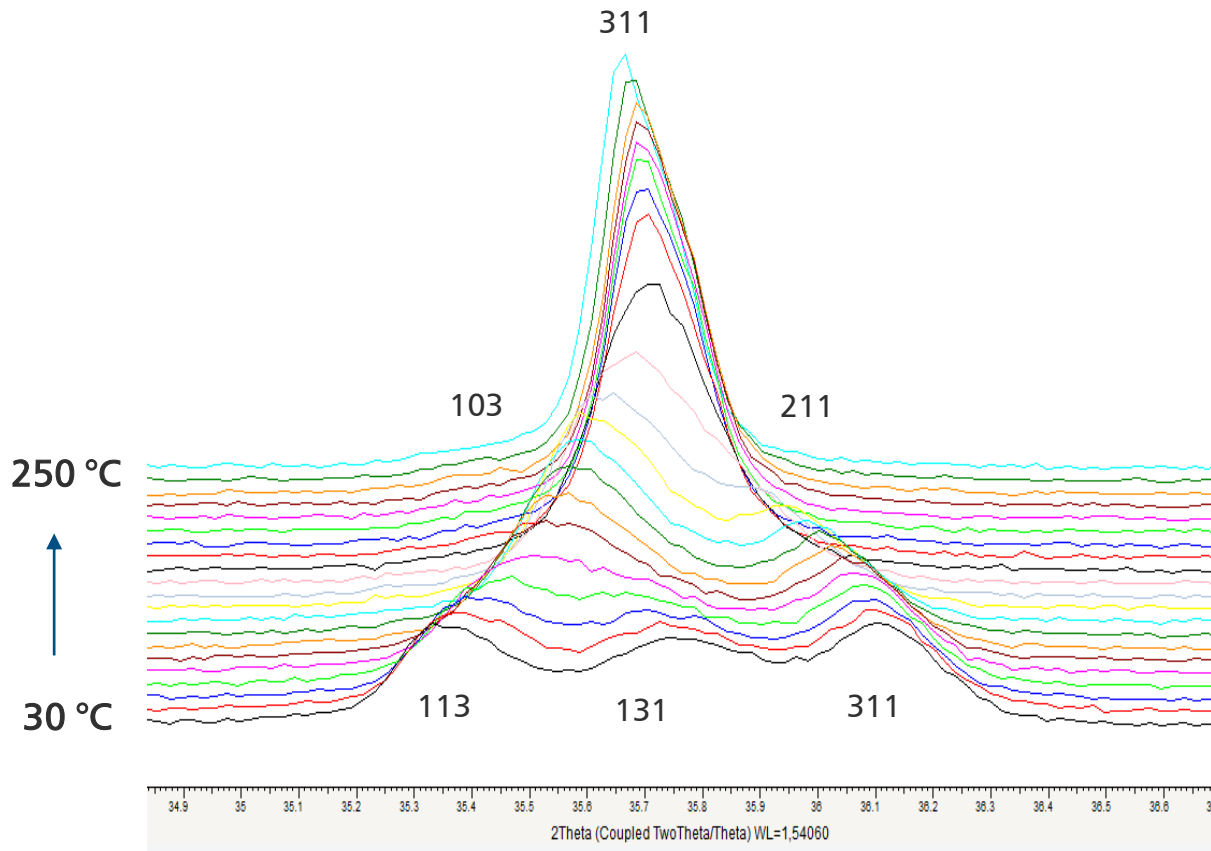
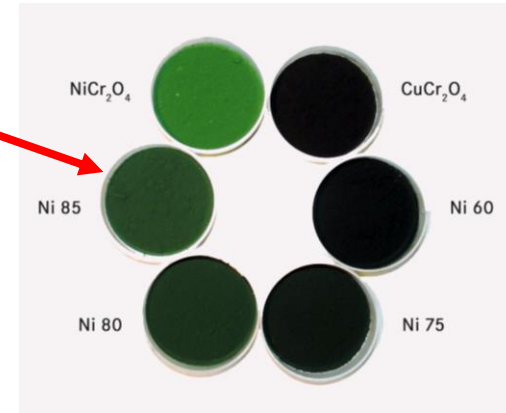
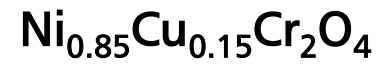


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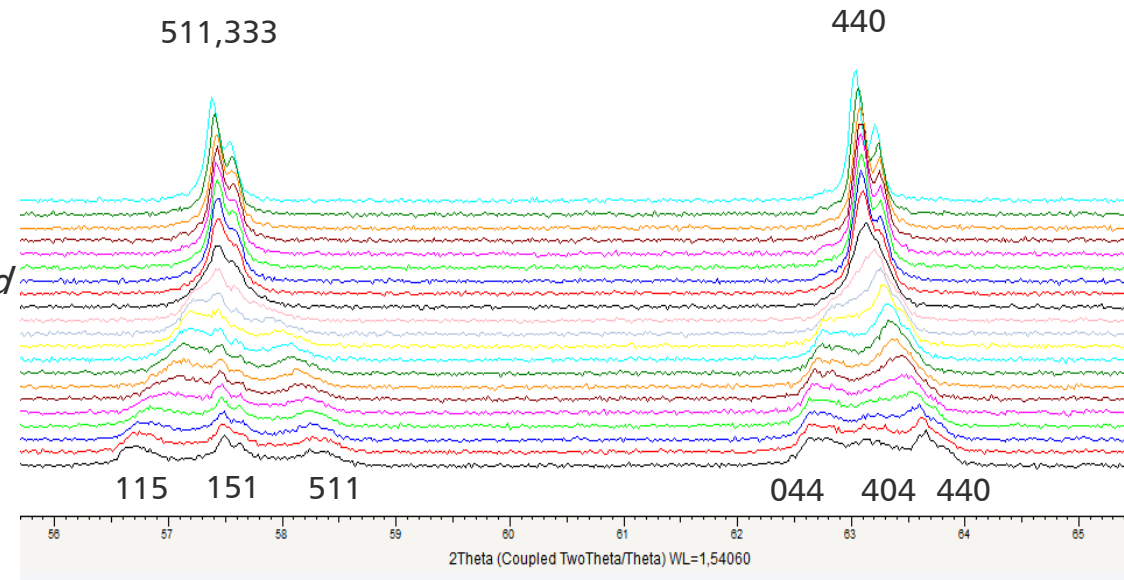


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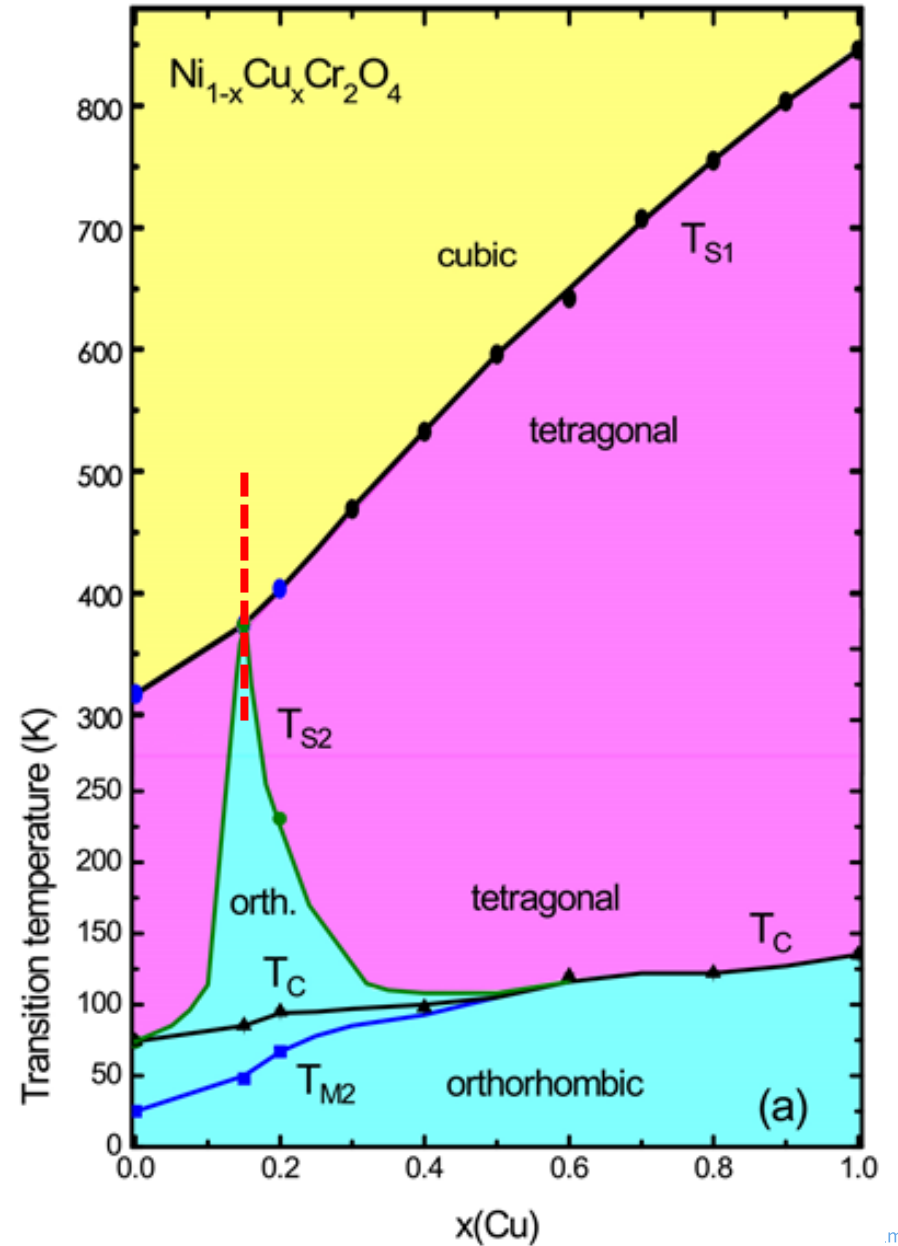
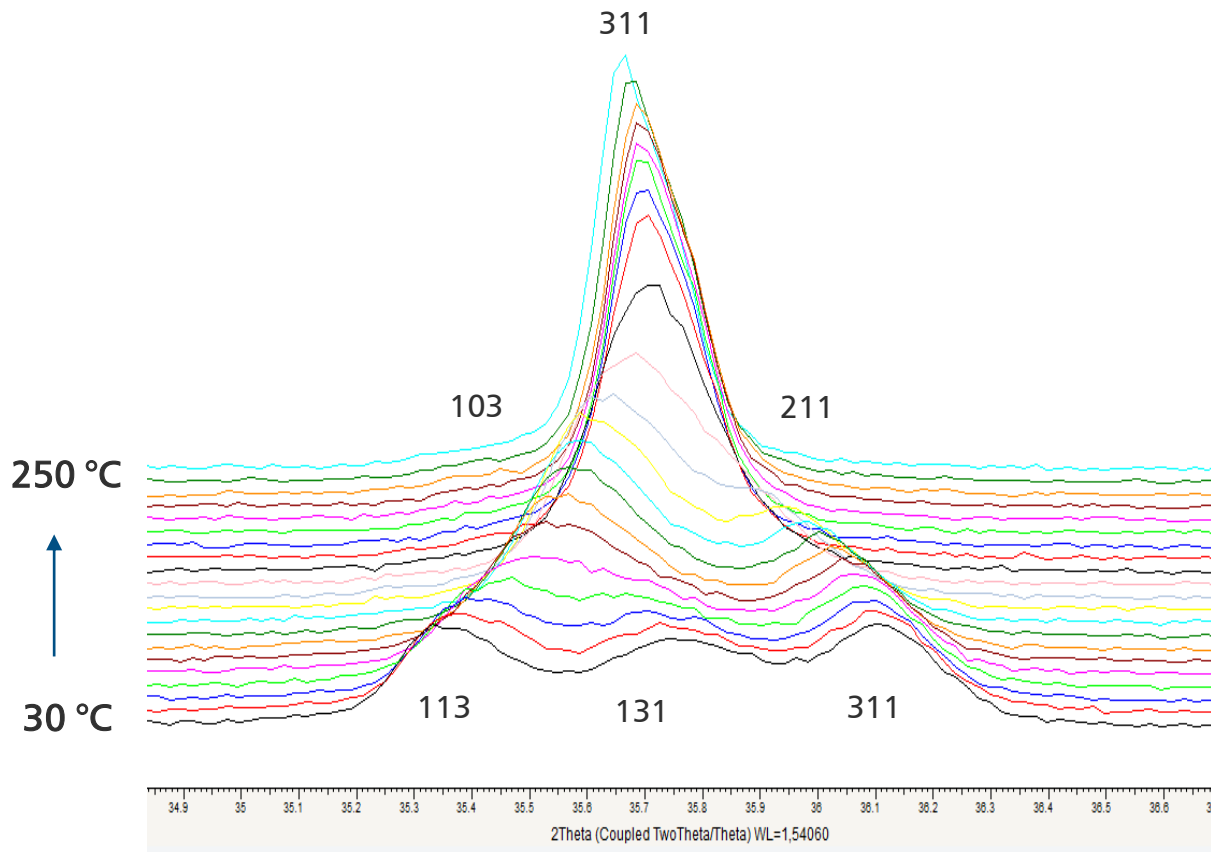


$Fd\bar{3}m$
 $I4_1/amd$
 $Fddd$



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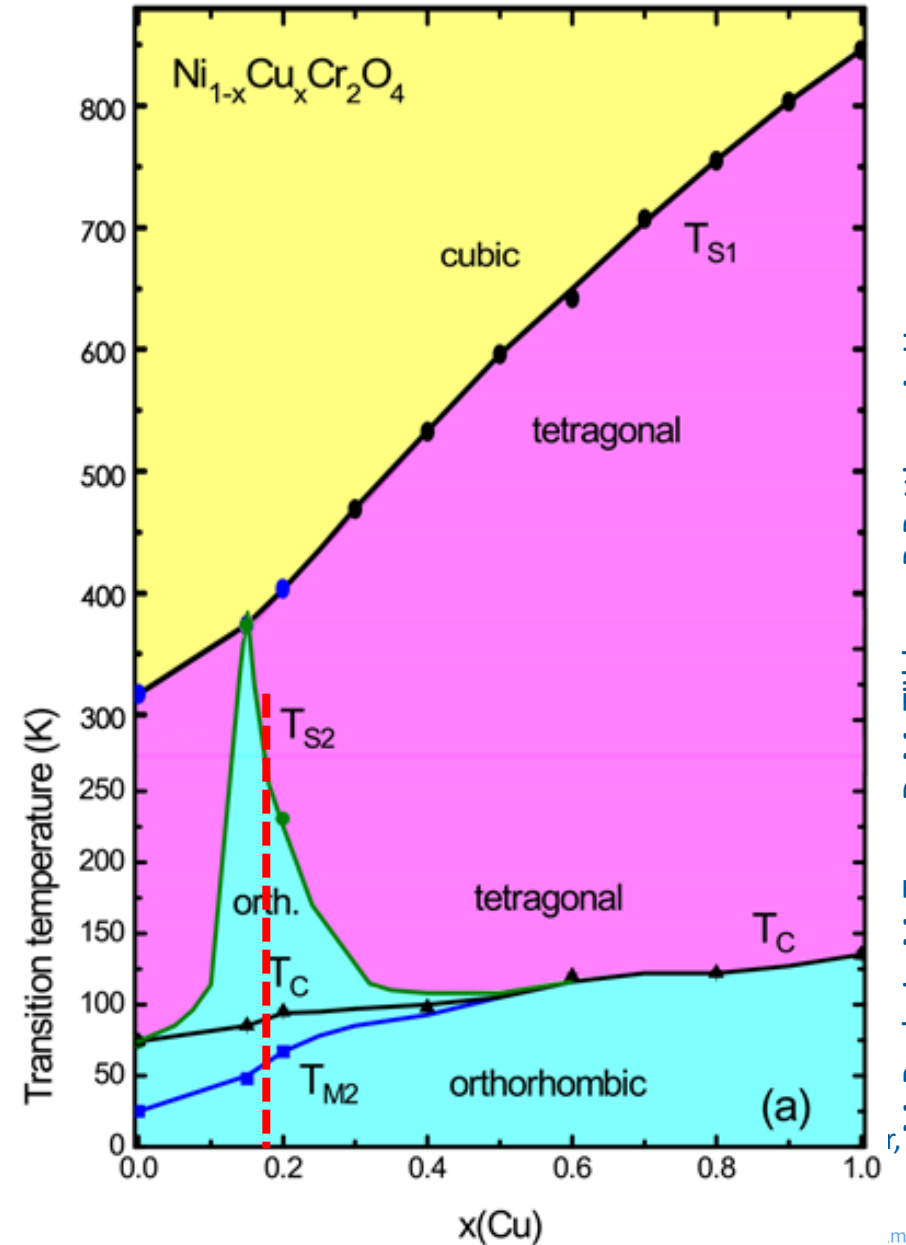
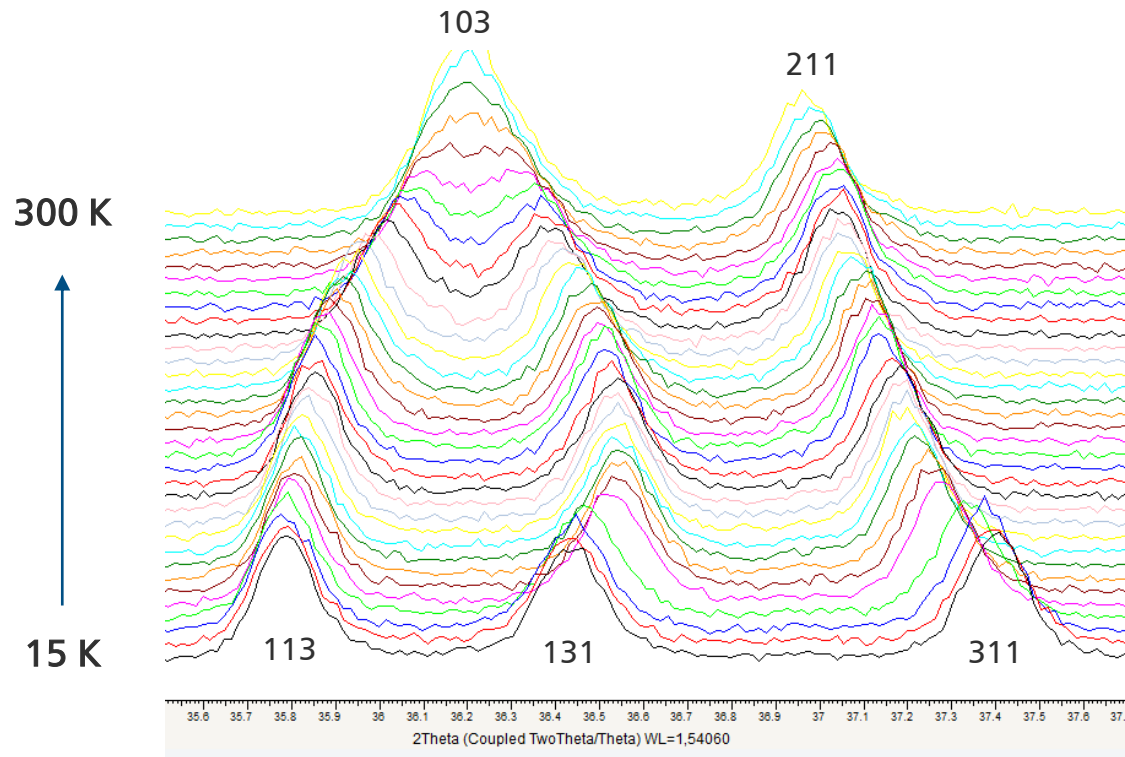


M. Reehuis, M. Tovar, D. M. Töbrens, P. Pattison, A. Hoser, and B. Lake: Phys. Rev. B 91, 024407 (2015)

5. TEMPERATURABHÄNGIGE DIFFRAKTION

Structural distortions in mixed spinels $\text{Ni}_{1-x}\text{Cu}_x\text{Cr}_2\text{O}_4$

LT-XRD of $\text{Ni}_{0.82}\text{Cu}_{0.18}\text{Cr}_2\text{O}_4$

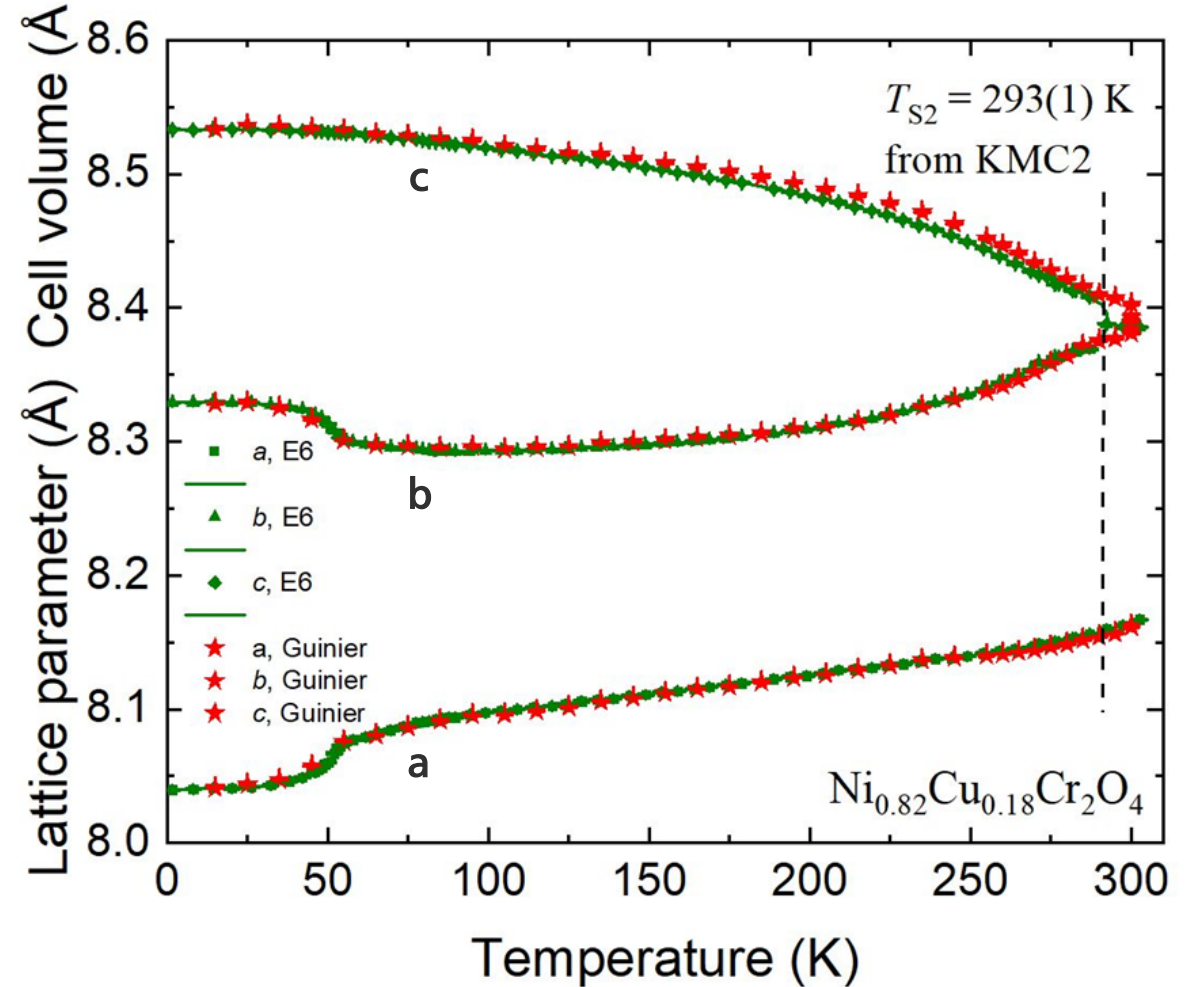
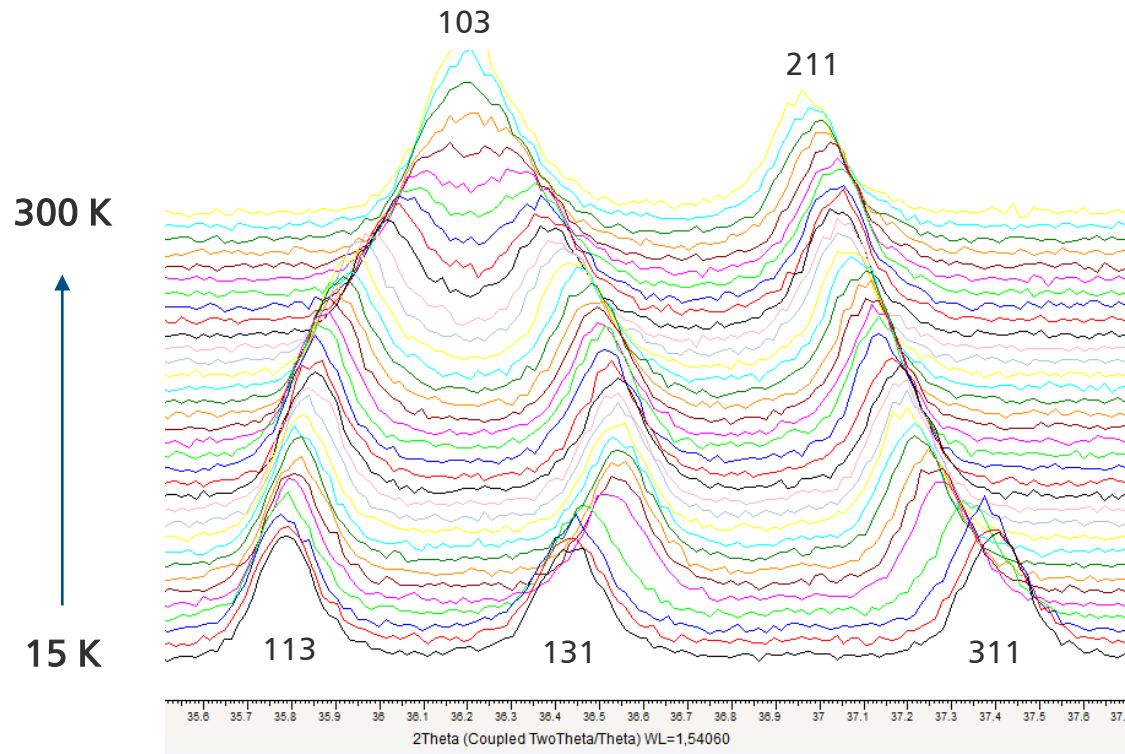


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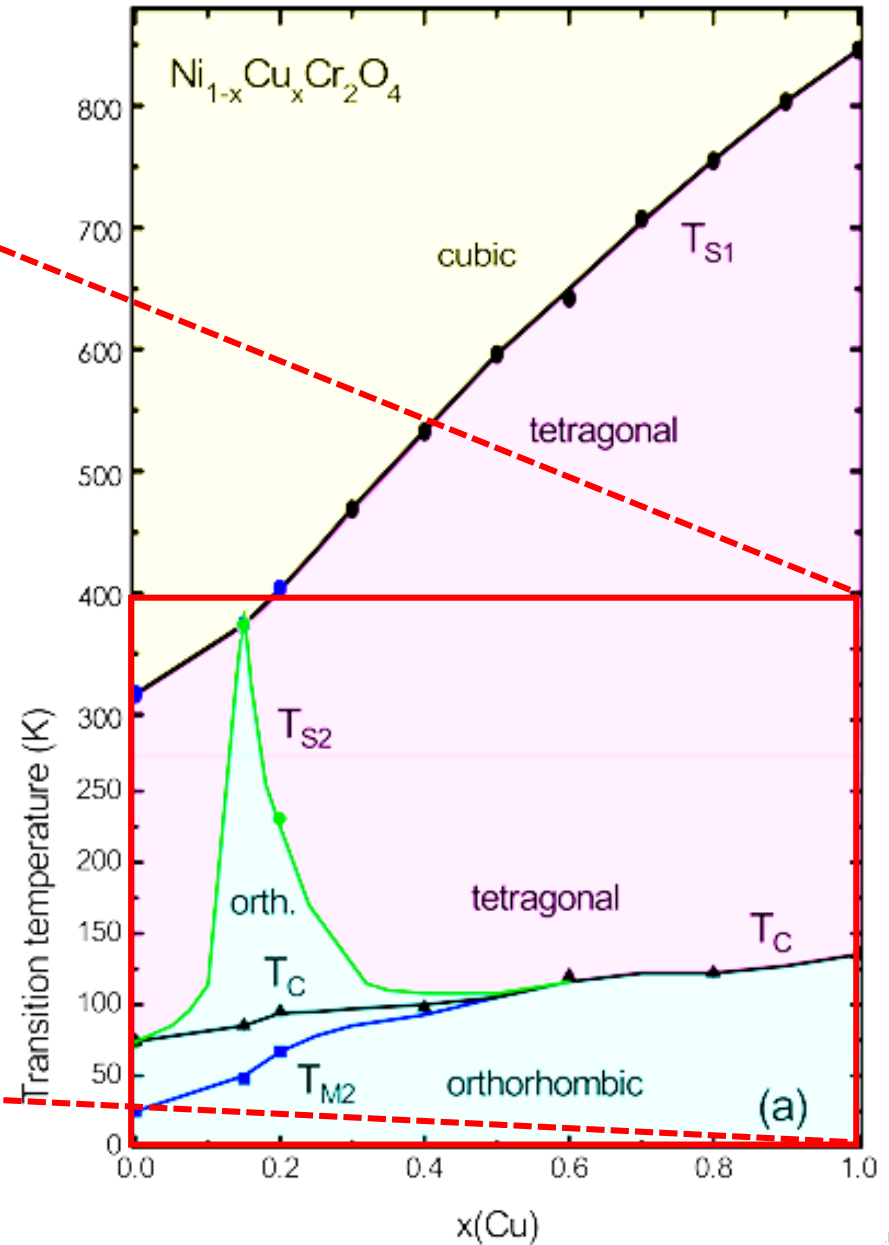
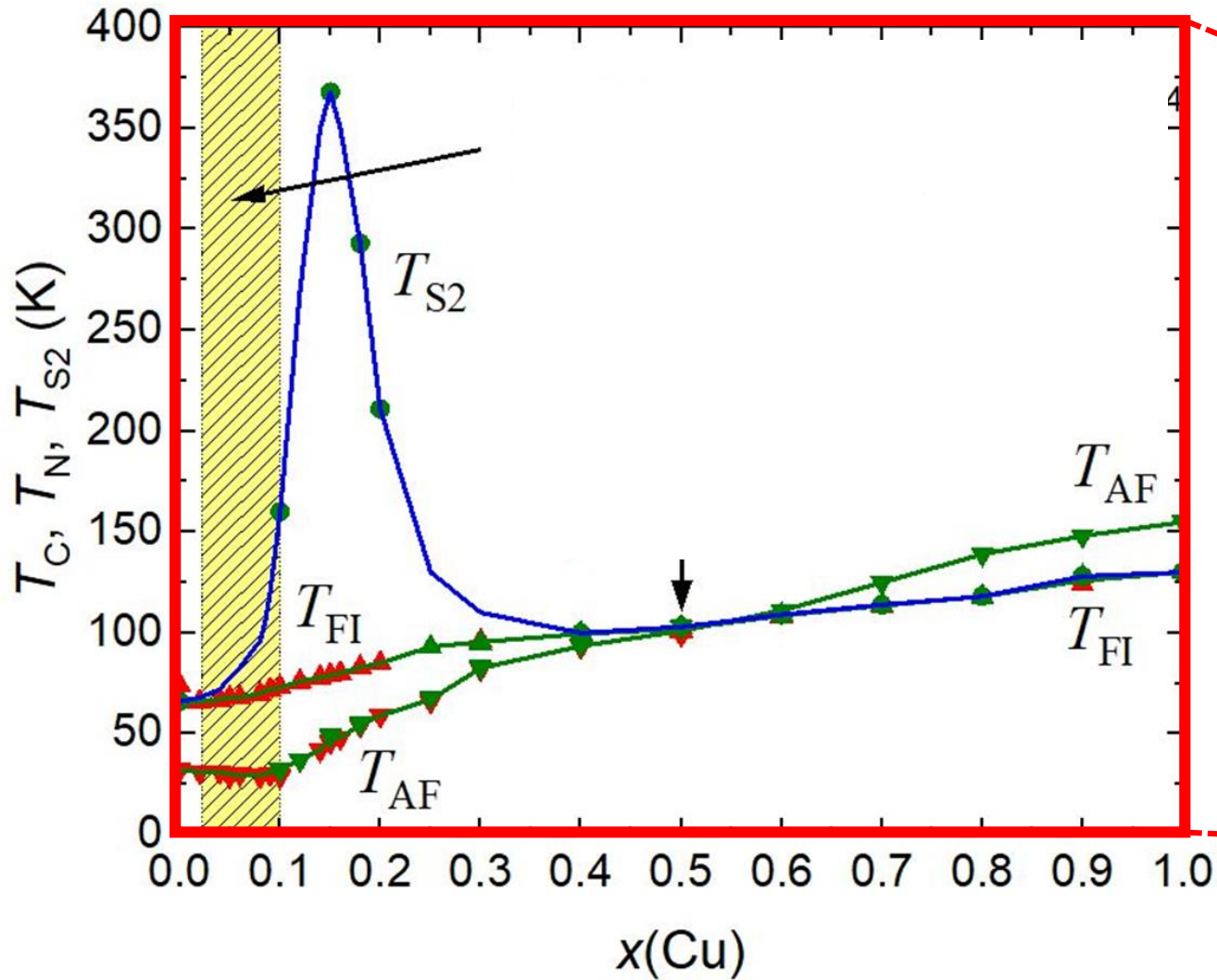
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M. Reehuis, M. Tovar, N. Stüßer (in preparation)

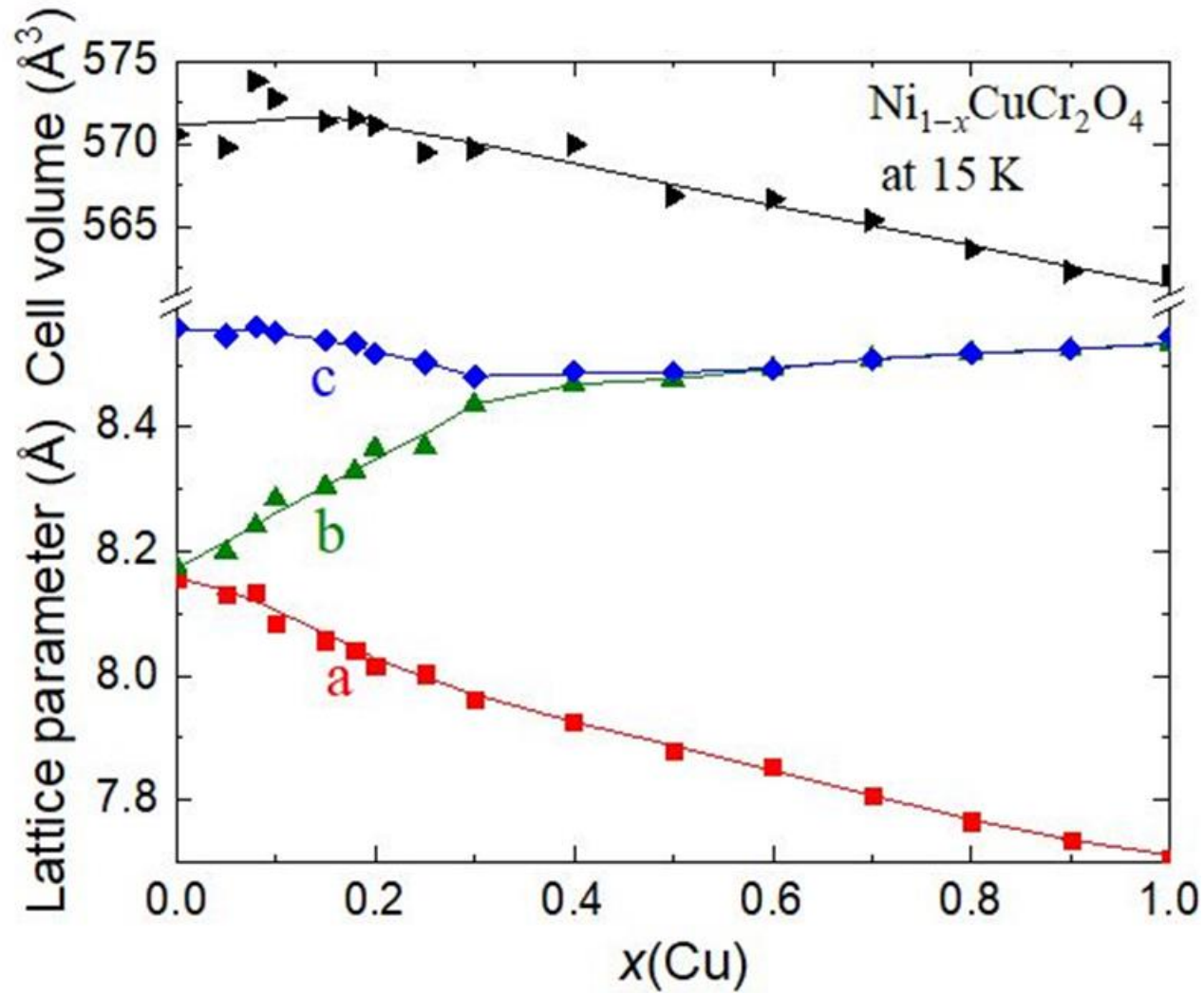
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M. Reehuis, M. Tovar, D. M. Többens, P. Pattison, A. Hoser, and B. Lake: Phys. Rev. B 91, 024407 (2015)

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M. Reehuis, M. Tovar, N. Stüßer (in preparation)

6. SCHULEN

- Schulungsraum mit 20 Plätzen
- Idealzahl 12

Fünf Röntgenschulen

Drei Rietveldschulen



6. SCHULEN

a) X-ray school

Program of the 5th X-ray school at HZB, 25.-29.04.2022 (Lise-Meitner-Campus)

| Zeit | Monday, 25th | Tuesday, 26th | Wednesday, 27th | Thursday, 28th | Friday, 29th |
|-------|---|---|---|--|--|
| 10:00 | Seminar room DV338 Welcome and introduction into crystallography S. Schorr | Seminar room DV338 X-ray powder pattern: Peak profile, background, FWHM, single peak fit Gunder | Practical training PT005/6 in rotating groups: - Instrument introduction - Optics, detection - Sample carriers - Preparation - Measuring parameters Tovar, Gunder | Practical training PT005/6 in rotating groups: 1) Grazing incidence XRD Gunder 2) High temperature XRD Tovar | Practical training PT005/6 in rotating groups: 1) Grazing incidence XRD Gunder 2) High temperature XRD Tovar |
| 11:00 | Exercise: Closest packing M. Tovar, R. Gunder - Coffee Break - | - Coffee Break - | | | |
| 12:00 | Fundamentals of X-ray diffraction S. Schorr | Extinction rules Schorr | Crystallographic databases (DV338) Tovar | Parallel session (DV338): Automatic indexation Többens | Parallel session (DV338) Automatic indexation Többens |
| 13:00 | - Lunch break - | | | | |
| 14:00 | Introduction to X-ray CoreLab Tovar | Organisation of lab practicals Tovar | Introduction into phase analysis applying search and match software EVA (Bruker) and powder diffraction databases | Introduction: LeBail refinement using Fullprof suite | Online talk: Modern X-ray detectors (30 min) Dubravka Sisak Jung, DECTRIS, Switzerland |
| 15:00 | <u>Powder pattern indexation:</u> 1 Introduction 2 Symmetry exercise 3 Pattern indexation by hand Tovar | Powder Cell for powder diffractogram simulation and analysis Gunder | Exercises in phase analysis Tovar | Evaluation: ZnS-CuInS ₂ mixed crystal series: Vegard's law Gunder | Metal Jet source at WCRC Roland Mainz Quiz + Closing |
| 16:00 | | | | | |
| | | | 17 Uhr: Get-together | | |



6. SCHULEN

b) Rietveld school

Program of the 1st Rietveld school of X-ray CoreLab, third group, 05.-09.07.2021

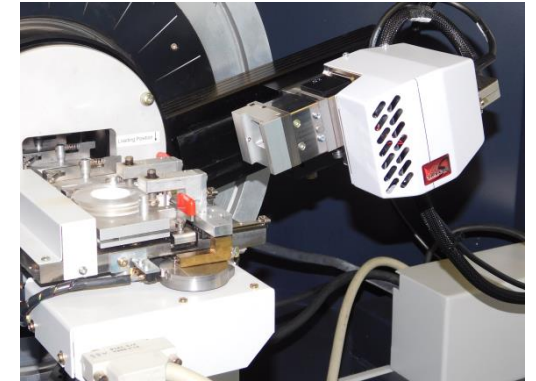
| Zeit | Monday, 05.07. | Tuesday, 06.07. | Wednesday, 07.07. | Thursday, 08.07. | Friday, 09.07. |
|-------|--|--|--|---|--|
| 10:00 | Welcome and school organization <i>S. Schorr, M. Tovar, R. Gunder</i> | Talk: Introduction into LeBail refinement | Full Rietveld analysis (guided): | Rietveld analysis of ZnS-CuInS ₂ mixed crystal series | Talk: Introduction into microstructure analysis |
| 11:00 | Introduction to the Rietveld analysis: history, basic idea of the procedure, refinement parameter, quality criteria - Break - Advanced Crystallography I: | Exercise: ZnS-CuInS ₂ mixed crystal series and Vegard's law | Introducing structure parameter, background treatment, refinement strategies | | Guided exercise in microstructure analysis |
| 12:00 | symmetry, classifications (systems, classes, 2D plane groups) Exercise: symmetry of 2D pattern <i>Schorr</i> | | | | <i>D. M. Többens</i> <i>D. M. Többens</i> |
| 13:00 | - 13:00 -14:00 Lunch break - | | | | |
| 14:00 | Hands-on Introduction into Fullprof suite and WinPlotr, Introduction into FIZ-ICSD <i>Tovar</i> | Exercise: LeBail refinement of GIXRD data of a CZTS thin film <i>Gunder</i> | Detailed view to Fullprof output files, error analysis, quality estimation <i>Tovar</i> | Multiphase refinement and quantitative analysis <i>Tovar</i> | Quiz + Survey + Closing <i>Tovar</i> |
| 15:00 | Break | Break | | Sequential refinement <i>Tovar</i> | |
| 16:00 | Creating a pcr file from ICSD and simulating an X-ray pattern (guided), background, FWHM... <i>Tovar</i> | Advanced Crystallography II: space groups and international tables <i>Schorr</i> | Rietveld analysis of a given diffraction pattern (ZnS) <i>Gunder</i> | | |
| | | | | 17:00 School barbecue! | |



7. FAZIT

Vorteile eines X-ray CoreLab (= zentrales Röntgenlabor)

- Abdeckung aller wichtigen **Methoden** der Pulverdiffraktion
- Flexible **Erweiterungsmöglichkeiten**
- Hohe **Durchlässigkeit** durch Methoden und Arbeitsgruppen
- **Vereinfachung** der Instrumentwartung und des Laborbetriebs
- **Softwarepool** zur Abdeckung verschiedener Analyseverfahren

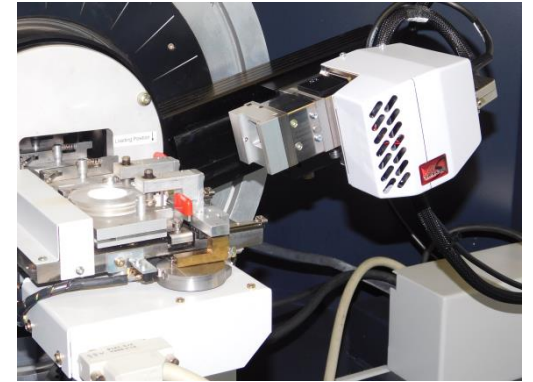


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- **Softwarepool** zur Abdeckung verschiedener Analyseverfahren

- **Basis-Schulungen** statt (zeitaufwändige) Einzeleinweisungen
- Schulungen **nach Bedarf**, der über das Labor ermittelt wird
- **Langzeitstabilität**: Instrumente geraten nicht in Vergessenheit
- **Community** mit gegenseitiger Unterstützung.



DANKE



- Susan Schorr: Head of steering committee, Röntgenschule
 - René Schwiddessen, Henrik Prell: Laborbetrieb, Röntgenschule
 - Joachim Breternitz: Bruker Kappa Apex II, Röntgenschule
 - Daniel Többens: Röntgenschule
 - Ralf Feyerherm: Guinier-Diffraktometer
-
- Maren Achilles: Budgetplanung
 - Bettina Wolter: Laborstatistik
-
- Reiner Giebler, Cees Baas+ Bruker-supportteam (!)

