MINIATURE AUXILIARY X-RAY DIFFRACTION SETUP FOR A SYNCHROTRON BEAMLINE END STATION

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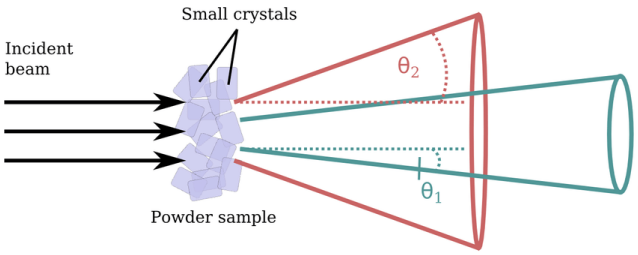
- Theory about Powder Diffraction, X-ray Absorption and Synchrotron Light
- Experiment with miniature (miniXRD) and large-area detector setup
- Results from this experiment
- Summary

RESEARCH QUESTION

To develop and characterize a miniature auxiliary X-ray diffractometer (XRD) for a synchrotron inelastic X-ray scattering beamline end station, with a focus on WO_3 (tungsten oxide) and Al_2O_3 (alumina) XRD studies as a function of photon beam energy and sample temperature.

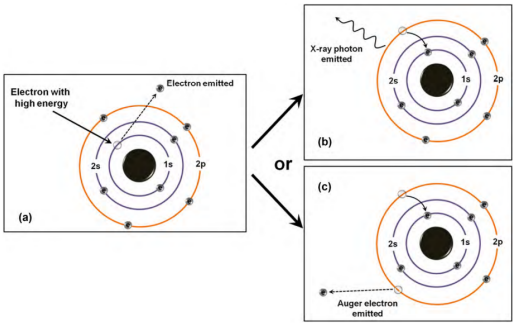
POWDER DIFFRACTION

- Used to determine crystal structures
- Measures diffraction patterns of polycrystalline samples
- Relies on Bragg's Law: $n\lambda = 2d \sin\theta$
- Applications: materials science and chemistry



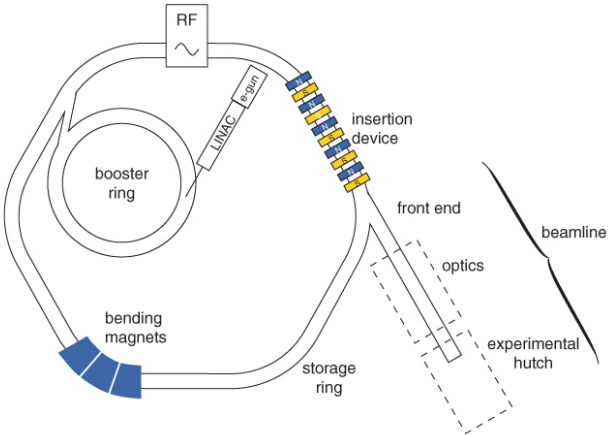
X-RAY ABSORPTION AND XAS

- Probes electronic and atomic structure via X-ray interaction.
- X-ray Absorption Spectroscopy (XAS) used for studying
- Key techniques in XAS:
  - X-ray Absorption Near Edge Structure (XANES)
  - Extended X-ray Absorption Fine Structure (EXAFS)
- Applications: material characterization, electronic structure, local atomic environment



SYNCHROTRON LIGHT

- Generated by accelerating electrons in a circular path
- High intensity and brilliance
- Broad energy range (infrared to hard X-rays)
- Applications: material science, biology, and chemistry



HYBRID PIXEL DETECTORS

- **Pilatus 300k-W (Pilatus):**
  - Detector area: 254 mm × 33.5 mm with a pixel size of 172 × 172 μm^2.
  - Energy range: 3–30 keV, with high quantum efficiency (e.g., 99% at 8 keV)
  - The sensor material silicon with a thickness of 320 μm.
- **MiniPIX TPX3 (Minipix):**
  - Detector area: 14 mm x 14 mm with a pixel size of 55 × 55 μm^2.
  - Uses Timepix technology for single-particle detection and high sensitivity.
  - The sensor material silicon with a thickness of 500 μm, optimized for detecting photons in a moderate energy range.

EXPERIMENT SETUP

- **Beamline:** ID20 at European Synchrotron Radiation Facility (ESRF), Grenoble, France
- **Sample:** WO_3-Al_2O_3 pellets with a diameter of 1 cm, thickness of about 0.3 mm each.
- **Detectors:** Pilatus and Minipix
- **Additional:** Two ion chambers for XAS measurements
- **Calibration:** Detectors will be calibrated with Cerium(IV) oxide (CeO_2) to ensure accuracy in measurements.

EXPERIMENT SETUP

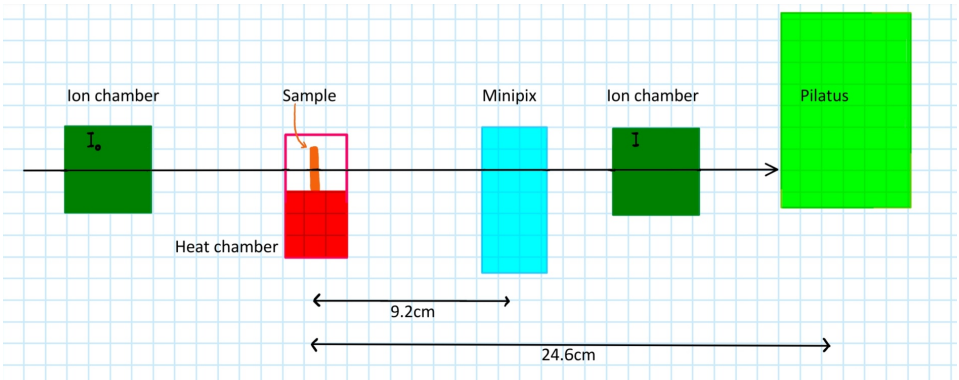
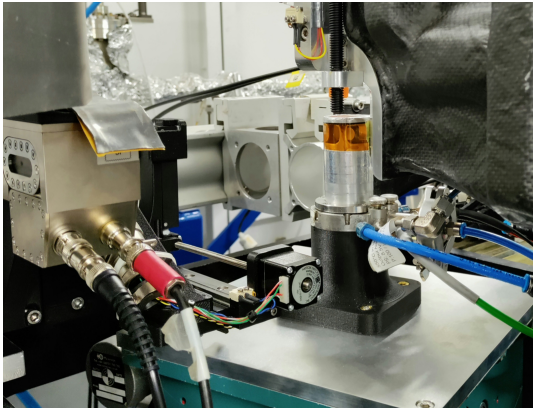


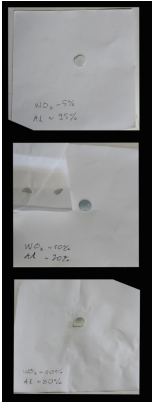
Figure: Not up to scale.

EXPERIMENT SETUP



EXPERIMENT

- Optimize WO_3-Al_2O_3 mixture for XRD measurements.
- **Room Temperature (22°C) Test:**
  - Initial tests with CeO_2 and 5% WO_3 concentration.
  - Alumina peaks dominated; WO_3 peaks were not clear.
  - Increasing WO_3 concentration to 10% improved WO_3 peak visibility.
- **Energy Calibration:**
  - Energies chosen: 10.180 keV, 10.190 keV, 10.195 keV, 10.200 keV
  - L_3 absorption edge of WO_3: 10.208 keV.

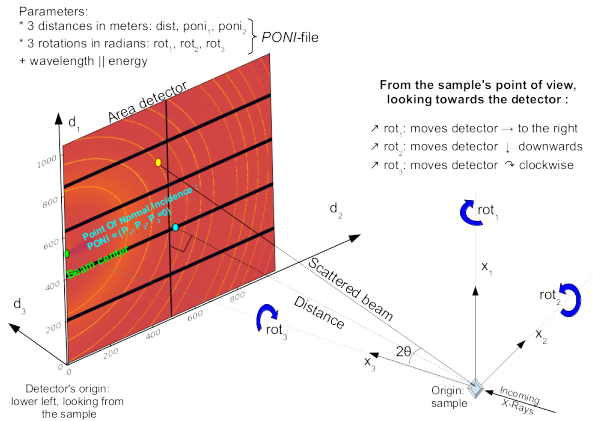


HIGH-TEMPERATURE AND VERIFICATION

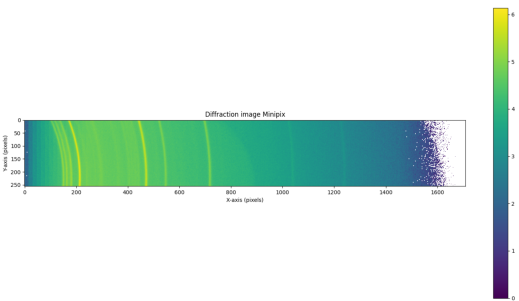
- **High-Temperature Measurements:**
  - Heat chamber used for 200°C, 400°C, and 600°C.
  - Three scans per temperature and energy point ensured accuracy.
- **Post-Experiment Verification:**
  - System cooled to room temperature.
  - CeO_2 used to verify detector alignment.
- **Setup Details:**
  - Minipix: 0.1 s/frame (170 frames), 17 s/scan; total acquisition 45 s
  - Pilatus: 0.1 s/diffraction pattern; total acquisition 30 s

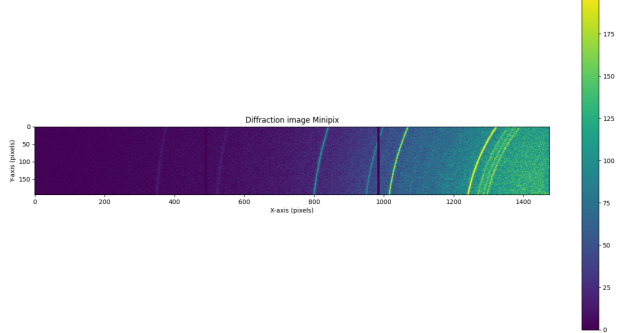
RESULTS: CALIBRATION

- PONI calibration was performed using pyFAI for both detectors to establish sample-to-detector distance and alignment.
- Minipix required image stitching; Pilatus captured patterns without stitching.



RESULTS: POWDER DIFFRACTION





RESULTS: POWDER DIFFRACTION

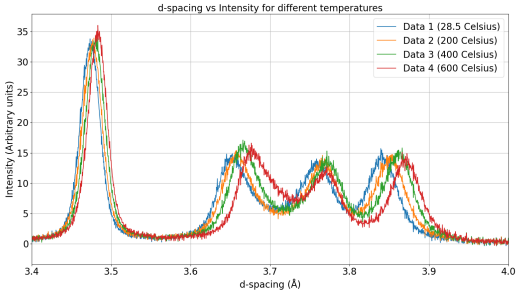


Figure: Powder diffraction pattern using Minipix.

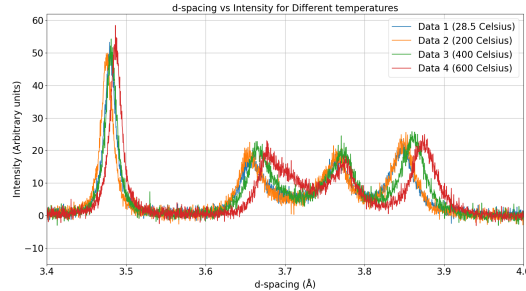


Figure: Powder diffraction pattern using Pilatus.

RESULTS: RESOLUTION

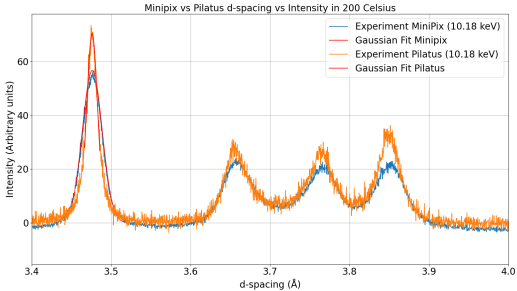


Figure: Pilatus and Minipix comparision

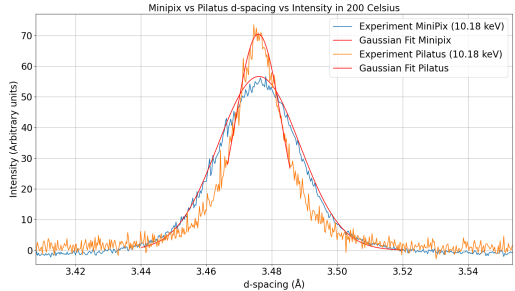


Figure: FWHM Pilatus: 0.0164 ± 0.003, FWHM Minipix: 0.0295 ± 0.003

RESULTS: XAS

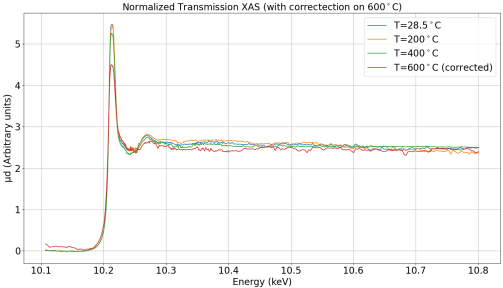


Figure: XAS measurements

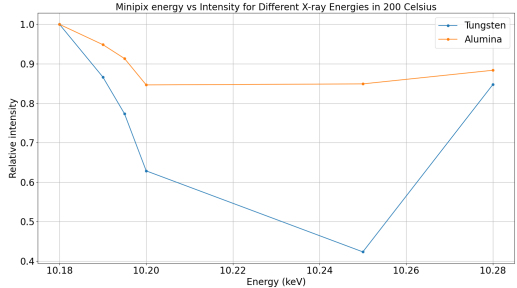


Figure: Peak intensities in different energies

RESULTS

- **Detector Performance:**
  - Minipix provided adequate resolution but required image stitching due to limited coverage.
- **Validation:**
  - Lattice parameters estimated from data match literature values.
  - The thermal coefficient for both material successfully evaluated.
  - XAS measurements correspond to literature.

SUMMARY

- Developed a compact X-ray diffraction (XRD) setup for WO_3-Al_2O_3 sample characterization at ID20.
- Validated the quality of XRD data by comparing patterns and data analysed results from the miniXRD setup (Minipix) and the large-area detector (Pilatus).
- Resolution proved to be sufficient enough for this kind of measurements, FWHM for Minipix 0.0295 ± 0.003 and for Pilatus 0.0164 ± 0.003.
- In the future, setup can be developed, e.g. by increasing the sample-to-detector distance and improving the stitching of images.

REFERENCES FOR FIGURES

Slide 3: Mabey, Paul. (2016). A study on dense plasmas using molecular dynamics and X-ray scattering techniques. 10.13140/RG.2.2.31961.42082.
Slide 4: Characterization of Biomaterials. Elsevier, 2013.
Slide 5: Philip Willmott. An Introduction to Synchrotron Radiation: Techniques and Ap- plications. John Wiley & Sons, Inc., Hoboken, NJ, USA, second edition, 2019.
Slide 12: Jérôme Kieffer. Fast Azimuthal Integration using Python. European Synchrotron Radiation Facility, Jan 2021.

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- Christoph J. Sahle et al. A miniature X-ray diffraction setup on ID20 at the European Synchrotron Radiation Facility. Journal of Synchrotron Radiation, 31(6), Nov 2024
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- Vitalij K. Pecharsky and Peter Y. Zavalij. Fundamentals of Powder Diffraction and Structural Characterization of Materials. Springer, 2nd edition, 2009.

Full reference list can be found from the end of the thesis.

Thank you for listening!
Any questions?