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Test results – Synchrotron Powder XRD measurements

Methods

Experiments and data processing

High-resolution synchrotron X-ray diffraction measurements were performed at beamline ID31 at the Synchrotron Radiation Facility. The sample powders were loaded into cylindrical slots (approx. 1 mm thickness) held between Kapton windows in a high-throughput sample holder. Each sample was measured in transmission geometry with an incident X-ray energy of 75.051 keV (λ = 0.16520 Å). Measured intensities were collected using an #### X-ray detector (xxxx × yyyy pixels, ppp × qqq μm^2 each) positioned with the incident beam in the corner of the detector. The sample-to-detector distance was approximately 1.5 m for the high-resolution measurements. Multiple measurements of the empty well with polyimide windows were measured, and summed together for improved statistics, for the background subtraction.

NIST SRM 660b (LaB_6) was used for geometry calibration for XRPD. Integration was performed with the software pyFAI, including flat-field, geometry, solid-angle, and polarization corrections. Invalid pixels were masked, and the sigma clipping integration algorithm was used to automask further azimuthal outliers. The summed background intensities were scaled respectively to each of the reference and sample measurements and subtracted. Rietveld refinements were performed using TOPAS v7 [1] to fit the structure with the reference lattice parameter fixed to the measurement to determine the instrumental profile contribution and correct for offset errors, including parallax [2]. The input files are available in the supplemental attachments upon request.

Sample preparations

The dough-like material was initially dried in an oven at 90 °C for 24 hours. For the unspiked sample, 200 mg of the dried material was homogenized using a Speca mill for 5 minutes. For the spiked sample, 200 mg of the dried material was combined with 22 mg of NIST SRM 674b TiO_2 standard, and the resulting mixture was subsequently homogenized with a Speca mill for 5 minutes. The milling is done in an agate vial (2.35 cubic centimeters in volume) with 2 agate balls, 4 mm in diameter, 180 mg each.

Data analysis

Phase ID was performed on identified peaks in both the as-sent and spiked samples using the ICDD PDF5+ database. Since no chemical information was available, an initial search was performed using common elements expected for the sample, and then a second round of searching was performed on expanded chemistry to account for the possibility of other metals. Identified phases were then fitted to the pattern using TOPAS v7. The instrumental profile was accounted for using the parameters determined from NIST LaB6 as described above. For phases with higher certainty, and where possible, lattice parameters and basic crystallite and strain broadening parameters were refined. For phases not well constrained by the pattern, parameters were kept fixed and only a scale factor was refined. A baseline was described using a 6th-order Chebychev polynomial for diffuse scattering and potential amorphous content.

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Results

Identified phases and composition are listed in **Table 1**. A high-resolution XRD diffractogram obtained from the sample is shown in **Figures 1-3**. Simulated phases from the sample are shown in **Figures 3a and 3b**.

Summary: Phase ID and composition

The **weighted profile R-factor (R_{wp})** is shown for all analyzed samples. R_{wp} is a statistical indicator that quantifies the agreement between the entire observed X-ray diffraction pattern and the calculated pattern based on a structural model. A lower Rwp value suggests a better agreement between the model and the experimental data. Identified phases, and their **estimated weight percentage (est. wt%)** from the spiked sample, as well as **original phase fractions/weight percentage (Orig. wt%)** determined without spiked phases are shown in the table.

Phase ID confidence is qualitatively based upon the ambiguity of diffraction peaks present. If the set of Bragg reflection for a given phase can be unambiguously seen, then confidence is given as high. Only phases marked as high confidence should be considered to be reasonably confirmed from the diffraction pattern alone. All phases marked medium and low confidence here only stand to provide suggestions for phases that appear to be consistent with the patterns but cannot be unambiguously confirmed from the patterns alone. Furthermore, all weight fraction values should be considered preliminary since not all phases have been identified. Errors reported are the uncertainty reported by the refinement.

Table 1. Identified phases and composition of analyzed samples. **Orig. wt%** represents the weight percentage of identified crystalline phases and amorphous phase in the original sample. Uncertainty in weight percentage is reported within parentheses.

Sample	Phase	Composition	ID confidence	est. wt%	orig. wt%
ID XXXXX-1 R_{wp} = pq.r %			high		
			medium		
			medium		
			medium		
			medium		
			medium		
			low		
			low		
			low		
			low		
			low		
			low		
	rutile	TiO_2	spiked phase	9.7910	–
	anatase	TiO_2	spiked phase	0.1183	–

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Sample: ID XXXXX-1

Figure 1. Comparison of separately prepared ID XXXXX-1 samples with and without spiking of the NIST reference material.

Figure 2. Top: Current state of fitting using the phases determined from the search-match process. A small number of weak peaks could not yet be identified.

Figure 3a. Comparison of simulated phases in log scale from sample ID XXXXX-1.

Figure 3b. Comparison of simulated phases in log scale from sample ID XXXXX-1.

Bibliography

[1] Alan A. Coelho. Topas and topas-academic: an optimization program integrating computer algebra and crystallographic objects written in c++. J. Appl. Cryst., 51:210–218, 2018.

[2] F. Marlton, O. Ivashko, M. Zimmerman, O. Gutowski, A.-C. Dippel, and M. R. V. Jørgensen. A simple correction for the parallax effect in X-ray pair distribution function measurements. J. Appl. Cryst., 52:1072–1076, 2019.

End of the test report

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