

XRD analysis of dropcast MXene suspensions - PANalytical X'Pert Pro II

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Tags: XRD XRD5 06/12/2021Synth KTH Collab

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Goal : Run typical, coupled $\theta:2\theta$ scan of dropcast Ti_3C_2 MXene synthesis product to confirm successful synthesis

Procedure :

Sample preparation

Prior to instrument booking, dropcast bulk concentration suspensions from synthesis on to zero-background holders (ZBH) made of oriented single-crystal silicon and leave to dry in fumehood. Ensure coverage of area 10 x 10 mm.

Instrument set-up

Geometry	Bragg-Brentano
Spinner	PW3064
Detector	1D X'Celerator (2.122 ° active length)
X-ray source	Copper line focus
Radiation	$K_{\alpha 1} = 0.1540598$ nm, $K_{\alpha 2} = 0.1544426$ nm, K_{α} ratio 0.5, $K_{\alpha \text{ av}} = 0.1541874$ nm
K_{β} absorber	0.02 mm Ni
Incident beam optics	0.04 rad Soller, 2 ° fixed anti-scatter, 10 mm incident beam mask, 0.03125° fixed divergence slit for ≤ 5 mm irradiated length
Diffacted beam optics	0.04 rad Soller
2θ start:finish:step / °	3:100:0.017
Dwell time / s	0.66
Stage oscillation (ϕ)	Yes

Data analysis

The methodology is much similar to that followed in [\[Experiment\] Quantitative Phase Analysis](#)

(QPA) of MXene synthesis product PXRD patterns, such that:

A structure is defined to represent Ti_3C_2 MXene, based on a model of 10wt% HF direct-etched Ti_3C_2 (<https://pubs.acs.org/doi/abs/10.1021/acs.chemmater.5b04250>)

The instrument and emission line profiles are carried forward from the Rietveld refinement to the LaB_6 calibrant in [Experiment] Quantitative Phase Analysis (QPA) of MXene synthesis product PXRD patterns, using a jEdit script which interfaces with TOPAS v5

In the absence of other phases (no internal standard), the XRD pattern has its fit optimised within physically justifiable limits to minimize errors using a .inp jEdit script named 2023BGIndividualScan.INP. A fifth order Cheybshev polynomial is fit to the background, minimum and maximum 2θ values ($2\theta_{\text{min}}$ and $2\theta_{\text{max}}$, respectively) are selected and the order of spherical harmonics is varied to improve the fit to account for crystallographic texture.

Results :

Sample	Filename
Dropcast Ti_3C_2 MXene synthesis product (see Experiment - MXene synthesis XI)	DropCastTi3C2_20211214 'KTHCollab'

.xy file is xy data of 2θ vs intensity, .xrdml file is the raw output from the diffractometer, .raw file is the TOPAS-legible direct conversion of the .xrdml file (converted with PowDLL Convertor), .inp is the jEdit input script for Rietveld refinement in TOPAS v5, .out file is the output of the same script, .txt files contain data of the fit phase hklm values, d-spacings, 2θ spacings and scaled intensities (suffixed hklm_d_Th2_IScaled.txt), columnar data of observed intensities, calculated fit intensities and the difference between these (suffixed Yobs_Ycalc_and_Difference.txt) and the .png file is an images of the plotted data, created using a Python script available on Github (XRD_TOPASfits_allphasehkl.py).

The goodness of fit parameters R-weighted pattern (R_{wp}) of the whole diffraction pattern is 30.55 and R_{Bragg} of the MXene phase is 8.29.

Conclusions:

No in depth analysis required, although qualitatively the sample seems to be of high Ti_3C_2 MXene purity in the absence of crystalline reflections. As noted throughout this analysis type, better fits would be achieved with the introduction of stacking faults, which is beyond the scope of this analysis.

Attached files

2023BGIndividualScan.out

sha256: d37093580d9991c247899b638d8616b520427458991cfccee73614aa9c38b51d

2023BGIndividualScan.INP

sha256: 92a2c1f29a9dc2dbf3dfe410a0df2a1fddd62b405dede1667cb94b839e92adf3

DropCastTi3C2_20211214.raw

sha256: d8e8c9ec6c71dda8efa147a3a42cae92847db19c9e281bda433134b305b360e2

KTHCollab_MXene_hklm_d_Th2_IScaled.txt

sha256: 2ff48818f43dec138e657a8298bc22e2dd9428e4e8f4eba74dd468e84936ad19

DropCastTi3C2_20211214.xrdml

sha256: 0d4ee4349a6458057f2e8e4f854121b6f3f600d5ff1a141eefd744b808b75961

KTHCollab_Yobs_Ycalc_and_Difference.txt

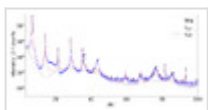
sha256: c8c47946daf4b01d46b57acd215b4c9debc23f8da8bca9dfb5e0e041dc198772

DropCastTi3C2_20211214.xy

sha256: 2466d3bb85653e333f043b463a405cdf9cd1798a19698d3d4d2c0e8980661d26

KTHCollab.png

sha256: 7d2eca5da81474d4ad361f3d4a95d18ae24c911e0cd8e3940bb403e2e1ba4c21



Unique eLabID: 20230708-467037d042f22c5905d13aa1c6e1b341427cfc9e

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=131>