

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

Date: 2020-03-17

Tags: XRD XRD5 11/03/2020Synth

Created by: James Bird

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Goal : Run typical, coupled θ :2 θ 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_{α} absorber	0.02 mm Ni
Incident beam optics	0.04 rad Soller, 2 ° fixed anti-scatter, 10 mm incident beam mask, automatic divergence slit (8 mm irradiated length)
Diffacted beam optics	0.04 rad Soller
2 θ start:finish:step / °	3.5:70:0.033
Dwell time / s	1.11
Stage oscillation (°)	Yes

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Results :

Sample	Filename
Dropcast Ti_3C_2 MXene synthesis product (see [Experiment] MXene synthesis V)	James Bird 10mm Mask Gonio Spin_Noeske Ti3_20200317

.xy file is xy columnar data of 2θ vs intensity, .xrdml file is raw output from diffractometer and .png is an image of the plotted data.

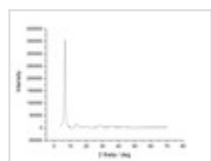
Conclusions:

No in depth analysis, although qualitatively the sample seems to be of high Ti_3C_2 MXene purity. Broad reflections at 6.8, 14.0, 20.8, 28.5, 35.9 & 43.4 ° 2θ are presumed to be harmonics of Ti_3C_2 MXene's (002) reflection givent the consistent intensity drop-off with increasing order (with the exception of (006)). Marked improvement in MXene purity with comparison to previous analysis. More complete data analysis (specifically quantitative phase analysis) still requires diffractometer calibration and phase pure MAX phase scan.

Attached files

20200317_DropcastTi3.png

sha256: 347ab1c665d2a1606cf1735ce56992dd38a881cfe0f124d99f2690e2a270f235



James-Bird-10mm-Mask-Gonio-Spin_Noeske-Ti3_20200317.xy

sha256: c3feb4ecc962624ff8ae43292c5532115fae46c98fd417aade3831e6d1766517

James-Bird-10mm-Mask-Gonio-Spin_Noeske-Ti3_20200317.xrdml

sha256: 8d5f4a7759b1c8bbf6f860049b1f7f151b15a137139bfbe1bc85ab2c8c2c6ccd

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Unique eLabID: 20221021-e11bb9093b91776d625ac99d8e6f1e58dfe47feb

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=56>