

XRD analysis of dropcast MXene and MAX phase/MXene suspensions - PANalytical X'Pert Pro (a.k.a. XRD5)

Date: 2019-07-22

Tags: Training 01/07/2019Synth XRD XRD5

Created by: James Bird

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Goal : Run typical, coupled $\theta:2\theta$ scans of dropcast MXene synthesis product and sediment to confirm successful synthesis

Procedure :

Sample preparation

Approximately 5 hours before 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)
Diffracted beam optics	0.04 rad Soller
2θ start:finish:step / °	5:70:0.033
Dwell time / s	1.22
Stage oscillation (°)	Yes

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Results :

Sample	Filename prefix
Sediment	Ti3C2_1
'Low quality' MXene product	Ti3C2_2
'High quality' MXene product	Ti3C2_3

X'Pert Highscore was used to fit the data profile with background type 1/x and using the minimum second derivative method to identify peaks. Phase identification proceeds by selecting which elements would be present in the material, after which an algorithm searches through powder diffraction files (PDFs) in a selected crystal database, such as ICSD (Inorganic crystal structure database) or ICDD (International centre for diffraction data), to determine which compounds bearing this composition best match to both the peak positions and their relative intensities.

Filetypes .xrdml are the raw output from the diffractometer, .xls are xy data of 2θ (°) vs intensity (a.u.), .txt is the output of identified peaks using X'Pert HighScore (PANalytical) software package and .DOC is a report from the same package. Plot saved as .png was produced in OriginPro software.

Conclusions:

Evidence of notable MAX phase presence remaining in all the suspensions, alongside typical (0002) MXene reflection at $< 9^\circ 2\theta$. MXene purity must be improved for further work. More complete data analysis (specifically quantitative phase analysis) not possible without diffractometer calibration and phase pure MAX phase scan.

Attached files

Ti3C2_2_20190722_1.xrdml

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sha256: 6cde08ef4326b108c0f539000ead0a641d3325dc0fb1c2bb7cc428e57010e942

Ti3C2_3_20190722-peak-list.txt

sha256: 03d970d622968284943dbef945bf6305f2ee48392cc3053c737bf70cbd2cc66d

Ti3C2_3_20190722.xls

sha256: 823a5f0f972231a722bb390a3e204585574239bd6d9ae32c7b5fd89fce283bd3

Ti3C2_3_20190722.DOC

sha256: 12c340524176b5bfc3a9d2f9d6f6949cbc52547d29f154a9ee0d10dcfaf501cb

Ti3C2_3_20190722.xrdml

sha256: eb59ff54818c480febaac32cfccb67376c8868affe50335b8e006a9654515429

Ti3C2_2_20190722_1.DOC

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Ti3C2_2_20190722_1.xls

sha256: c9c3f9daa847f2b4445280206a03fa800ec047aab508a4b0fc94a037fa418631

Ti3C2_1_20190722.xrdml

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Ti3C2_1_20190722.xls

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Ti3C2_1.DOC

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Ti3C2_1_20190722-peak-list.txt

sha256: 8011bf2c1954aec2c0b80f3e32ca9b856dfdbb39e7b02566196a4c39c56724cd

20190722_XRD5_FirstTi3C2Synthesis.png

sha256: f0ee16a9c3377de6a0e241609c9a330869b6f041619b32506fc67ba0fe4ea236

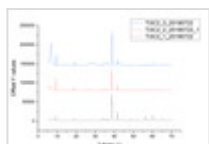
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Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=6>