

# XRD analysis of dropcast MXene and MAX phase/MXene suspensions - PANalytical X'Pert Pro I

Date: 2019-09-18

Tags: Training 01/07/2019Synth XRD XRD5 28/08/2019Synth

Created by: James Bird

1 / 4

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

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, $K_{\alpha}$ ratio 0.5, $K_{\alpha \text{ av}}$ = 0.1541874 nm
$K_{\alpha}$ 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 $\theta$ start:finish:step / °	5:70:0.033 or 0.017
Dwell time / s	1.22 or 2.86
Stage oscillation (°)	Yes

Smaller step size and longer dwell time used for both sediment samples.

# XRD analysis of dropcast MXene and MAX phase/MXene suspensions - PANalytical X'Pert Pro I

Date: 2019-09-18

Tags: Training 01/07/2019Synth XRD XRD5 28/08/2019Synth

Created by: James Bird

2 / 4

## Results :

Sample	Filename prefix
Sediment from 01/07/2019 synthesis	Ti3AlC2_03-07
Sediment from 28/08/2019 synthesis	Ti3AlC2_30-08
'High quality' MXene product	Ti3C2

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  $2\theta$  (°) 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. No notable increase in oxidation due to storage.

## Attached files

Ti3AlC2\_03-07\_20190918-peak-list.txt  
sha256: 350546b74f1984d14dbb57fe4b3f22d83b6f260a26a55951a979106f8c724a1b

# XRD analysis of dropcast MXene and MAX phase/MXene suspensions - PANalytical X'Pert Pro I

Date: 2019-09-18

Tags: Training 01/07/2019Synth XRD XRD5 28/08/2019Synth

Created by: James Bird

3 / 4

Ti3AlC2\_03-07\_20190918.DOC

sha256: 95f8899948c5304c904cb565f4c4bc1c439565b8b312170ca4e2abefc153fa6e

Ti3AlC2\_03-07\_20190918.xls

sha256: 537ea8d0c2e85df547e2845814e54fbe37bc215a3946f73b856667c2c51f14d8

Ti3AlC2\_03-07\_20190918.xrdml

sha256: ff22b262057243414069a4ddac6600d15654d43370f522376be27fe7f3480b67

Ti3AlC2\_30-08\_20190918-peak-list.txt

sha256: 6c4629b083572b829116e955f8c17dbc0a5da49cb07eadf868024b698a701690

Ti3AlC2\_30-08\_20190918.DOC

sha256: 5bef6e3881d1a965b263f45add6d301a139fdd273fce93e6a05100e3c9180fc3

Ti3AlC2\_30-08\_20190918.xls

sha256: 9a27fa343551814b2a94f41d934c99c0ee544eebe776734748fefda327689a02

Ti3AlC2\_30-08\_20190918.xrdml

sha256: df53d8f67d08482d663b36375c8ffa4849df7acdada5dd9aebaa46ad4304251b8

Ti3C2\_20190918.xls

sha256: ebd844ef3e2751b1bf7582e156934e8f36d1508148227537f7642b7633377a99

Ti3C2\_20190918.DOC

sha256: e44c77bff8fb54eb290d85ece166b7d31099333336e33aff60591d2fbbf35a01

Ti3C2\_20190918.xrdml

sha256: c44aba0c1a6e2527c68e5ff1c6e4097b2d262cd85adddb3015ddbe98eaaa156

Ti3C2\_20190918-peak-list.txt

sha256: c13af2dde26e11e156613713836e984e5aada83daf83be13b6818985f98d08ed

G20190918.png

sha256: 202525baf5e070daecc2a17ce6fd5a940500f1bf10ea7c6b3e91f8b05f978cdb

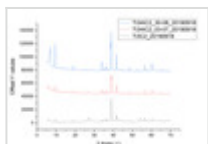
# XRD analysis of dropcast MXene and MAX phase/MXene suspensions - PANalytical X'Pert Pro I

Date: 2019-09-18

Tags: Training 01/07/2019Synth XRD XRD5 28/08/2019Synth

Created by: James Bird

4 / 4



Unique eLabID: 20221009-1743a79e38e5cee4923754d9ea29a670d5e02f20

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=16>