

# XRD analysis of vacuum-filtered MXene film - PANalytical X'Pert Pro

Date: 2021-10-13

Tags: XRD XRD5 Texture 18/08/2021Synth

Created by: James Bird

1 / 4

**Goal : Characterise crystallographic texture of a vacuum-filtered  $\text{Ti}_3\text{C}_2$  MXene film and verify composition/purity using Rietveld refinement**

Procedure :

## Sample preparation

Vacuum filtered film ( $\varnothing < 47$  mm, produced in [\[Experiment\] Vacuum-filtration of MXene nanoparticle suspension](#)), still attached to the filter membrane, is held flush with the sample holder surface using a spring-loaded sample holder (see IMG\_1652.JPEG)

## 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, 0.03125° fixed divergence slit for $\varnothing$ 5 mm irradiated length
Diffacted beam optics	0.04 rad Soller
$2\theta$ start:finish:step / °	3:100:0.017
Dwell time / s	0.66
Stage oscillation ( $\varnothing$ )	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:

# XRD analysis of vacuum-filtered MXene film - PANalytical X'Pert Pro

Date: 2021-10-13

Tags: XRD XRD5 Texture 18/08/2021Synth

Created by: James Bird

2 / 4

- A structure is defined to represent  $\text{Ti}_3\text{C}_2$  MXene, based on a model of 10wt% HF direct-etched  $\text{Ti}_3\text{C}_2$  (<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  $\text{LaB}_6$  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 another .inp jEdit script, named MXeneFilmNoNegPO.inp. A fifth order Cheybshev polynomial is fit to the background, minimum and maximum  $2\theta$  values ( $2\theta_{\text{min}}$  and  $2\theta_{\text{max}}$ , respectively) span the whole  $2\theta$  measurement range and 2nd order spherical harmonics are used to improve the fit to account for crystallographic texture.

## Results :

Sample	Filename
Vacuum-filtered $\text{Ti}_3\text{C}_2$ film	James Bird 10mm Mask Gonio Spin_VacuumFilteredFilm_20211013 JamesBird10mmMaskGonioSpin_VacuumFilteredFilm_20211013

.xls file is  $2\theta$  vs intensity, .xrdml file is raw output from diffractometer, .raw file is a 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 is the output of the same script, .txt files contain data of the fit MXene hklm values, d-spacings,  $2\theta$  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 .png is an image of the plotted data.

The goodness of fit parameters R-weighted pattern ( $R_{\text{wp}}$ ) of the whole diffraction pattern and  $R_{\text{Bragg}}$  for the phase are 31.32 and 4.56, respectively.

## Conclusions:

Much similarly to the analyses conducted in [\[Experiment\] Quantitative Phase Analysis \(QPA\) of MXene synthesis product PXRD patterns](#), the fit to the background is imperfect, especially in the very low angle range ( $< 30^\circ 2\theta$ ). The unit cell definition used for the vacuum-filtered  $\text{Ti}_3\text{C}_2$

# XRD analysis of vacuum-filtered MXene film - PANalytical X'Pert Pro

Date: 2021-10-13

Tags: XRD XRD5 Texture 18/08/2021Synth

Created by: James Bird

3 / 4

MXene film matches less well to the observed diffraction pattern than the mixtures analysed previously, as indicated by the large  $R_{wp}$  for the phase-pure sample. A notable difference is of course the introduction of presumably much greater crystallographic texture in the vacuum-filtered film compared to when powdered  $Ti_3C_2$  MXene forms part of a mixture. Thus fewer notable reflections are observed in the film. It is likely that the restacking of the two-dimensional nanomaterials leads to the  $P6_3/m\ m\ c$  space group no longer providing a suitable definition of the structure, hence the offset peak locations when the structure is constrained to that of perfectly stacked particles.

## Attached files

IMG\_1652.JPEG

sha256: ec7c0cdabfcef1457a1b8124543a831c718de8491c3897e38bc7ee9f42f6c287



James-Bird-10mm-Mask-Gonio-Spin\_VacuumFilteredFilm\_20211013.xls

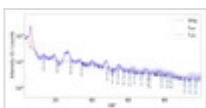
sha256: 45690408ebaced91a5dea7d8ea04b88e20aab02206d773c7e6a74fa113f2a48f

James-Bird-10mm-Mask-Gonio-Spin\_VacuumFilteredFilm\_20211013.xrdml

sha256: d81707b73a8d245a97c9a7581bec5f6c9d1b6b136f8b1e878fc08781d7025b96

Ti3C2\_VacFilterFilm.png

sha256: eb414d3e88726d2e6a943340a3e987fe53521b555c62f04627bc1f6183f3c1ae



JamesBird10mmMaskGonioSpin\_VacuumFilteredFilm\_20211013.raw

sha256: ac791f5d3a4678a63027a32fa02716937d380008ce4c3eee232bc79686b3efd6

# XRD analysis of vacuum-filtered MXene film - PANalytical X'Pert Pro

Date: 2021-10-13

Tags: XRD XRD5 Texture 18/08/2021Synth

Created by: James Bird

4 / 4

MXeneFilm\_NoNegPO.out

sha256: d4148e1eb75866ea431aafda838fb786e9805ba5f4712ae47a5980fe99b7c9da

MXeneFilm\_NoNegPO.inp

sha256: 25c76eb0b571f3619b0bbe76dcba2ccee357d28601a2fcb610eae765213a8266

MXene\_hklm\_d\_Th2\_IScaled.txt

sha256: b84e28c793b0896b72073ee69a12651a09a25fb1608dce94a01b3d44d8260232

Yobs\_Ycalc\_and\_Difference.txt

sha256: 6f19fcb58b6e267de47865c164087c11ed31c6f36b895b2450b936e8d80bdb53



Unique eLabID: 20230320-82ad9c257119ca807630e47dba6b340bd9a2b5dd

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=100>