

XRD analysis of vacuum-oven dried MXene film - PANalytical X'Pert Pro

Date: 2019-11-28

Tags: Training XRD XRD5 07/10/2019Synth

Created by: James Bird

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Goal : Characterise vacuum-oven dried MXene film

Procedure :

Sample preparation

Large, torn piece of vacuum oven-dried film ($10 < \ell / \text{mm} < 16$, produced in Exp. 'MXene synthesis yield acquisition') attached to silicon zero-background holder (ZBH) using double-sided sticky tape outside of the illumination zone ($\ell = 8 \text{ mm}$). An attempt was made to flatten the film as much as possible by using a combination of a spatula and forceps when placing the film, in order to render the film flush with the ZBH surface.

Instrument set-up

Geometry	Bragg-Brentano
Spinner	PW3064
Detector	1D X'Celerator (2.122 ° active length)
X-ray source	Copper line focus
Radiation	$K_{\alpha 1} = 0.1540598 \text{ nm}$, $K_{\alpha 2} = 0.1544426 \text{ nm}$, K_{α} ratio 0.5, $K_{\alpha \text{ av}} = 0.1541874 \text{ nm}$
K_{α} absorber	0.02 mm Ni
Incident beam optics	0.04 rad Soller, 2 ° fixed anti-scatter, 10 mm incident beam mask, fixed divergence slit (0.5°, 140 mm from sample giving 8 mm irradiated length)
Diffacted beam optics	0.04 rad Soller
2θ start:finish:step / °	5:70:8.4x10 ⁻³
Dwell time / s	2.86
Stage oscillation (°)	Yes

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Results :

Sample	Filename
Vacuum-oven dried film of Ti_3C_2	Refinement_Ti3C2 vac dried film_20191128

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

Conclusions:

The sharp peaks of the parent MAX phase are visible in the scan of the MXene film, suggesting the etch and delamination was incomplete under the synthesis parameters. The (002) MAX phase height is however much smaller relative to the (002) of the MXene, so qualitatively it is apparent that much MAX phase has been converted to MXene, especially considering the relative peak areas. The MXene (002) peak corresponds to a c lattice parameter (c-LP) of 2.598 nm. These results highlight the difficulty in indexing a $\text{Ti}_3\text{C}_2\text{T}_z$ diffraction pattern, where so few, but broad peaks are present which correspond to the MXene. The issue is complicated by the unavoidable introduction of texture in such a sample-type, where the nanoparticle basal planes will predominantly align parallel to the substrate.

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Attached files

Refinement_Ti3C2-vac-dried-film_20191128.xrdml

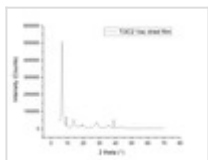
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Refinement_Ti3C2-vac-dried-film_20191128.xls

sha256: f33c28a81f9611a996c288217f1966a29a2862878bb9814f2cc1563b674120fc

Ti3C2_VacOvenDriedFilm.png

sha256: fb44cd35854602ae68df6b711fa1b870889a7c5d53ccfcc350538c00713692be



Unique eLabID: 20221012-2f77c0e5b3369dc228724d956d29550bb5ada18a

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=31>