

XRD analysis of freeze-dried MXene powder - PANalytical X'Pert Pro

Date: 2020-10-02

Tags: XRD XRD5 11/03/2020Synth

Created by: James Bird

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Goal : Run typical, coupled θ : 2θ scan of freeze-dried and powdered Ti_3C_2 MXene synthesis product to confirm successful synthesis and lack of decomposition during long-term storage

Procedure :

Sample preparation

Freeze-dried Ti_3C_2 (synthesised in [\[Experiment\] MXene synthesis V](#)) was ground into a powder with a pestle and mortar and loaded into a back-loading powder sample holder, ensuring powder flush with holder surface.

Instrument set-up

| | |
|---------------------------------|--|
| Geometry | Bragg-Brentano |
| Spinner | PW3064 |
| Detector | 1D X'Celerator (2.122 ° active length) |
| X-ray source | Copper line focus |
| Radiation | $K_{\alpha 1} = 0.1540598$ 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 |
| 2θ start:finish:step / ° | 3.5:70:0.033 |
| Dwell time / s | 1.11 |
| Stage oscillation (°) | Yes |

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Results :

| Sample | Filename |
|--|---|
| Ti ₃ C ₂ freeze-dried, ground powder | 200916 KaiKai98 Ti3C2Tx Bremen_20201002 |

.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:

Both this and the previous XRD analysis were ran on the same batch of MXene ([\[Experiment\] XRD analysis of dropcast MXene suspensions - PANalytical X'Pert Pro](#)) although the sample preparation techniques were different - the previous was dropcast whilst this was freeze-cast and powdered. Observations of interest is the shift in presumed MXene peak intensities of MXene peaks in (00l), where l is even, harmonic series to 7.2, 14.5, 28.7, 35.4, 43.5° 2θ (from 6.8, 14.0, 20.8, 28.5, 35.9 & 43.4° 2θ), the (006) reflection appears missing and the peak shapes have changed substantially. MAX phase reflections at ~ 9.6, 19.2 and 38.8° 2θ are still present, unshifted and at similar relative intensities. The scan conditions being equivalent, these can only be accounted to the transition from a more textured (dropcast) sample to an isotropic (freeze-dried powder) sample. There is of course also the possibility that oxidation after 205 days in storage has contributed.

A unique observation is the introduction of a fairly intense peak at 60.8° 2θ in this powder diffraction pattern. Most intriguingly, this reflection aligns with the first, and only reflection in this scan range which is perpendicular to the Ti₃C₂ basal plane, with hkl values (2 -1 0). The appearance of this particular reflection is entirely consistent with the change in sample preparation method, as these reflections were previously inaccessible when particles are predominantly aligned parallel to the ZBH when dropcast. It's relative sharpness (comparative to (00l) reflections) is testament to intact Ti₃C₂ polyhedra. More complete data analysis (specifically quantitative phase analysis) still requires diffractometer calibration and a phase pure MAX phase scan.

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Attached files

200916-KaiKai98-Ti3C2Tx-Bremen_20201002.xrdml

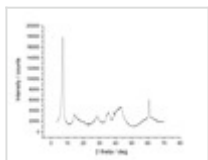
sha256: ff56d1c178f3ceedf198bb0f1d9945f7dcd4a6b8f2053bef1ddfe8f055fa7adb

200916-KaiKai98-Ti3C2Tx-Bremen_20201002.xy

sha256: e2a902c9b7ca1ac793ed4ad98f69153b2e247abadb8858ba8834317f2d0d7ef0

20201002_Ti3C2Powder.png

sha256: 27a011e131b899cdef187cab27b4503e10bd22093c7db28c7e8df59a9fdc7b58



Unique eLabID: 20221028-ab59efdf5598cbb501af13333b19a468f36589d2

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=60>