

EDXRF of MXene synthesis product

Date: 2020-10-02

Tags: 11/03/2020Synth Purity EDXRF

Created by: James Bird

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Goal : Perform EDXRF on Ti_3C_2 MXene product to verify composition and purity

Procedure :

Sample preparation

- Ti_3C_2 MXene product synthesised in [\[Experiment\] MXene synthesis V](#) is ground into a powder using a pestle and mortar
- ~ 250 mg is submitted for analysis (here 258.3 mg) to be able to cover the entirety of a Mylar ($\text{C}_{10}\text{H}_8\text{O}_4$) film of 28 mm diameter (3.6 μm thick and density 1.37 g cm^{-3})
- Sample loaded into the EDXRF instrument (Panalytical MiniPal 4)
- Argon flow ensured for duration of all four 'Omnians' - each Omnian constitutes a series of settings under which characteristic X-ray detection is performed during which time the sample is illuminated with X-rays produced from a rhodium anode:

Omnian	Anode voltage / kV	Current / μA	Filter	Medium
1	30	300	Silver	Helium
2	20	151	Aluminium	Helium
3	12	148	Thin aluminium	Helium
4	5.5	679	None	Helium

Instrument operation/data processing

In MiniPal software program:

- Sample mass and thickness input (measured with balance and calipers) - 250 mg and 1 mm
- Omnian sample parameters chosen are:

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1. Sample preparation type: Loose Powder
 2. Required sum is not used
 3. Correct for finite thickness effects
 4. Correct for fluorescence volume geometry (FVG)
- Most recent Omnian calibration loaded
 - Default, elemental compound list loaded
 - All visible line groups (characteristic X-ray emission lines)
 - Background fit models are axial filter with square root preprocessing, with 24, 20, 16 and 10 iterations for Omnians 1-4, respectively.

Results :

Element	Conc / %
Al	0.538
P	0.153
Cl	7.097
Ca	0.082
Ti	81.308

The calculated theoretical weight percentage (concentration) for both Ti_3AlC_2 MAX phase and Ti_3C_2 are also given below.

Element	Mass / amu	MAX phase stoich	Amu contribution	MAX wt%	MXene stoich	Amu contribution	MXene wt%
Ti	47.867	3	143.601	73.79	3	143.601	77.11
C	12.011	2	24.022	12.34	2	24.022	12.90
H	1.008	0	0	0.00	0.06	0.06048	0.0325
Al	26.982	1	26.982	13.87	0	0	0.00
Cl	35.45	0	0	0.00	0	0	0.00
O	15.999	0	0	0.00	0.85	13.59915	7.30

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F	18.998	0	0	0.00	0.26	4.93948	2.65
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.txt files are spectra from each Omnian, .qan is the concentration of each element present and .pngs are various plots of the dataset.

Conclusions:

If all of the aluminium present can be attributed to remaining Ti_3AlC_2 present in the etch product, then it follows mathematically that the proportion of each phase present can be calculated, based on the assumption of there being only the two phases present ($\text{Ti}_3\text{C}_2\text{T}_z$ and Ti_3AlC_2). The titanium : carbon stoichiometry allows the calculation of the concentration of the (undetectable) carbon also. As can be seen, the calculation leads to a sum of concentrations > 100 %, which suggests there are no oxygen, fluorine or hydrogen (hydroxyl) terminating groups present on the MXene surface. This is of course contrary to the literature. Errors associated with PANalytical's Fundamental Parameters algorithm are unfortunately opaque. Qualitatively, it can be deduced that the MXene purity is high, and the addition of T_z contributions to mass would only increase the stated purity value.

Component	Conc / wt%	Purity / %
[Ti]_MXene	78.44	
[Ti]_MAX	2.86	
[C]	13.60	
[Al]	0.54	
[Cl]	7.10	
[O + F + H]	-2.54	
[MXene]	98.66	96.48

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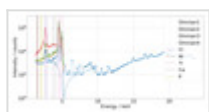
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[MAX]	3.88	3.52
SUM	102.54	100

Attached files

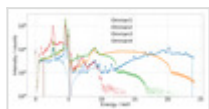
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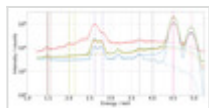
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2020-10-02_Ti3C2_logyEDXRFplot_SharedOmnianRange_Elements.png

sha256: 87e730106e56f9e01c6baf233bae8fb5e1780c2bdfadfb01f401cae96ebb3c7a



BremenSpectrumOmnian1.txt

sha256: 5198fdb7d2c151ab80d4476b3c00a1f2e16b10992bc99c5a47feeccd47e72972

BremenSpectrumOmnian2.txt

sha256: 745cd440c49ad89cf665bc33c0a9428f327f81917dba73876c8ab0e2e1b73c17

BremenSpectrumOmnian3.txt

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BremenSpectrumOmnian4.txt

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2020-10-02_Ti3C2_EDXRFplot.png

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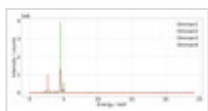
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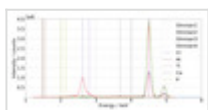
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2020-10-02_Ti3C2_EDXRFplot_OmnianRange_Elements.png

sha256: 91839cac970059551e4cf72d133fbb83abae798324db5beb2dd069ac57bcd8



2020-10-02_KaiKai98_Ti3C2_Bremen.qan

sha256: ea1e308927b69ac92d4eb975197633d3a5247df47510de5490080479c17fb40f



Unique eLabID: 20221103-1db4d1e0362c3d77667f25c5ee9b903c9e12343d

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=66>