SEM - Zeiss EVO 50 I

Date: 2019-07-26
Tags: *Training 01/07/2019Synth SEM PSD EDX EVO50*
Created by: James Bird

Goal : Confirm morphology of MXene suspension (product) and sediment components align with XRD findings using EDX

Procedure :

- Samples (see Experiment SEM stub preparation for PSD I) loaded into SEM chamber and vacuum acquired (< 1.3 x 10^{-4} mbar)
- 20 kV accelerating voltage set for electron beam
- WD either 13 or 13.5 mm (within recommended EDX WD range of 9 < WD / mm < 15)
- Secondary electron detection only (Everhart-Thornley)

Results :

Sample	Filename prefix
Higher concentration sediment	Ti3AlC2
Higher concentration 'High quality' MXene product	Ti3C2

- More particles (presumed MAX phase - somewhat corrobated with EDX and definitely with XRD) present in sediment sample than product
- Some dispersed MXene particles (dark on light silicon background) visible with micron-scale lateral dimensions (and smaller) at higher magnification of ~ 3,000 x
- Unable to image clearly at higher magnification due to poor selection of imaging conditions
- Area, point scan and mapping with EDX in more ideal conditions (recommended WD) showed aluminium presence in crystallites, again alluding to unetched MAX phase presence
- EDX point scan in presumed MXene region (dark, continuous regions below crystallites) failed to show notable titanium presence due to X-ray interaction volume at high accelerating voltage

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Detailed EDX findings

The maps and image correspond well to expected observations: silicon gives a strong signal from the substrate, across all areas bar those where crystallites are visible, which instead strongly emit X-rays of energy corresponding to the Ti $K_{\square}$ electron transition, along with the weaker emission of Al $K_{\square}$ X-rays. Both carbon and fluorine $K_{\square}$ X-rays are most strongly emitted from the crystallites. Oxygen however shows the contrary - there is a distinct sparsity of X-rays emitted from the location of particles, suggesting instead that oxygen is present in the substrate. When plots are overlay, areas of high X-ray density for Ti and Al correlate well, whilst C and F also correlate well. This observation insinuates that more carbon-dense MAX particles are more inclined to harbour fluorine or fluorinated terminating groups if etching without delamination occurred. We would expect oxygen to be found wherever MXene is present, exisiting as a terminating group, as is concordant with the O $K_{\square}$ EDX map map. Although, there is no clear reduction in the oxygen in the area where the MXene is absent on the SEM micrograph. Note that the general abundance of aluminium indicated by the EDX map is likely to be an overestimate due to the peak overlap with silicon.

Attached files

Ti3AlC201.tif
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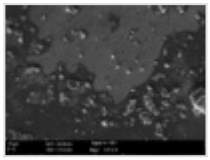

Ti3AlC202.tif
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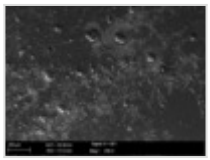

Ti3AlC203.tif

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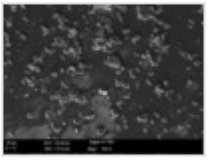

Ti3AlC203_from_EDX_mapping.png
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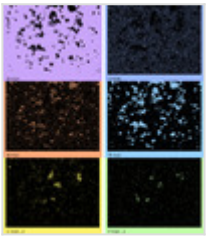

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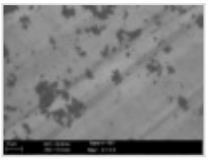

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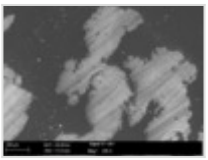

EV050-EDX-Sediment.docx
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Link: https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=8