

SEM - Zeiss EVO 50 II

Date: 2019-07-30

Tags: Training 01/07/2019Synth SEM PSD EDX EVO50

Created by: James Bird

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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 ($< 1.3 \times 10^{-4}$ mbar)
- 15 or 20 kV accelerating voltage set for electron beam
- WD between 7.5 & 10.5 mm (analytical working distance (AWD) is 8 mm)
- Secondary electron (SE) detection only (Everhart-Thornley)

Results :

Sample	Filename prefix
Lower concentration sediment	JB-Ti3AlC2
Lower concentration 'High quality' MXene product	JB-Ti3C2-004
Higher concentration 'High quality' MXene product	JB-Ti3C2-003

Detailed EDX findings

SE micrographs and supporting EDX maps of the same area were taken at the AWD for the lower concentration sediment sample. EDX mapping was ran for a longer period of time (933.89 seconds) than in the subsequent experiment (Scanning Electron Microscope - Zeiss EVO 50 I). The data support the findings in the previous experiment.

Confirmation that the black flakes appearing on the SE micrographs were MXene was sought with EDX of the lower concentration MXene product - region selected is 'coffee ring'. A shorter X-ray collection time of 732.78 seconds was used. WD

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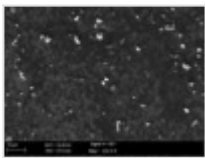
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was approximately AWD. 20 kV accelerating voltage too high as the X-ray count from the silicon substrate caused the aluminium peak to be absorbed by the bremsstrahlung shoulder of the Si K_α peak. Despite the lack of X-ray counts from the sample itself and the shorter collection time, there does appear to be a faint correlation between the dark region on the micrograph and Ti and C presence, with an absence of Al above the background level.

Attached files

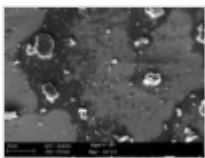
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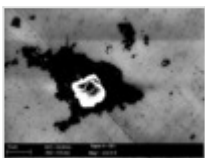
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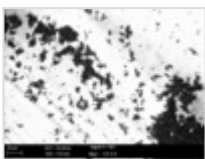
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JB-Ti3C2-003-03.tif

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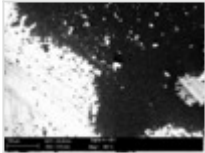
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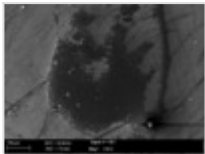
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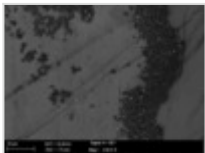
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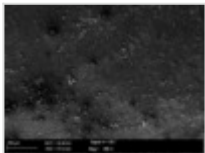
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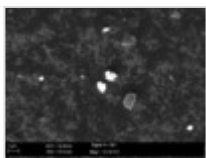
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EDX_DropcastComponents.docx

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JB-Ti3C2-003-04SumSpectrum.txt

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Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=9>