

SEM - Zeiss EVO 50 III

Date: 2019-11-20

Tags: SEM EDX EVO50 KU Leuven 07/10/2019Synth

Created by: James Bird

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Goal : Investigate morphology of MAX phase particles and vacuum oven-dried MXene film with imaging and EDX

Procedure :

- Samples (see Exp. 'SEM stub preparation for film and MAX phase characterisation') loaded into SEM chamber and vacuum acquired ($< 1.3 \times 10^{-4}$ mbar)
- 15 kV accelerating voltage set for electron beam
- WD set at either 7, 9, 9.5 or 10 mm (analytical working distance (AWD) is 8 mm)
- Secondary electron (SE) detection only (Everhart-Thornley)

Results :

Sample	Filename prefix
Ti ₃ AlC ₂ MAX phase powder (KU Leuven, $d > 75 \mu\text{m}$, $> 98\%$)	Ti3AlC2 powder
Vacuum oven-dried film from 07/10/2019 MXene synthesis	Ti3C2 vac dried film

.tif files are micrographs, .docx is a report generated in Inca and .txt is xy data of the reported sum spectrum.

SEM

Reasonable imaging of both film and MAX phase particles to reveal rough surface of MAX particles and layered film edge.

EDX

EDX conducted at close to AWD, for a total collection time of time of 1179.65 s. Titanium, fluorine and chlorine appear to be correlated, although the count time should have been extended to confirm this. A large carbon signal is given by the substrate (appearing as empty space in the micrograph). Sulfur and sodium should not be present as they do not feature in the processing route - viable alternative elemental assignment should be explored.

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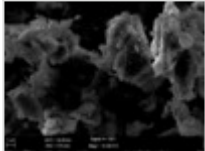
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Attached files

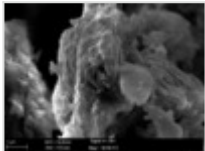
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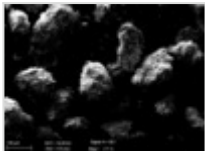
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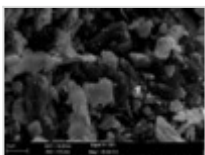
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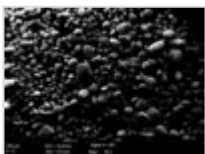
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Ti3AlC2-powder005.tif

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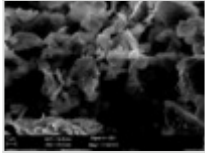
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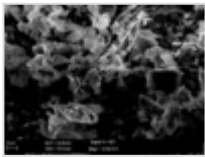
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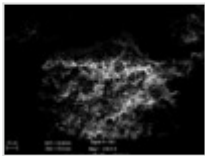
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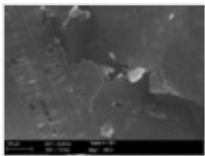
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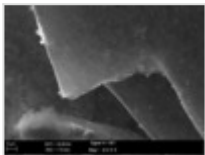
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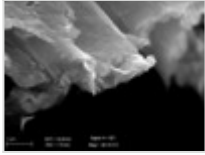
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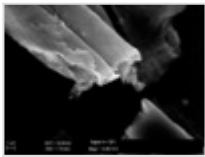
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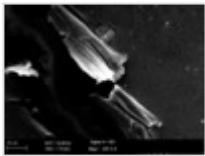
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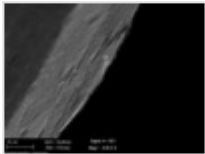
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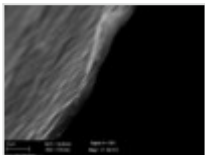
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Ti3C2-vac-dried-film007.tif

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201119-Ti3C2Tx-film.docx

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201119-sum-spectrum.txt

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Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=30>