

SEM stub preparation for PSD II

Date: 2019-09-09

Tags: 01/07/2019Synth SEM PSD Sample preparation 28/08/2019Synth

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Goal : Dropcast diluted Ti_3C_2 on to silicon wafer to enable particle-size distribution (PSD) to be produced from scanning electron microscope (SEM) imaging

Materials :

- Sediment from 01/07/2019 and 29/08/2019 synthesis
- 'High quality' MXene (product) from 01/07/2019 synthesis and only (single) product from other synthesis.

Procedure :

- Use micropipette (Pipetman P1000) to transfer small aliquot (~ 50 μL) from Ti_3C_2 bulk suspension to glass vial
- Dilute with deionised water until translucent (light grey) for a higher (unknown) relative concentration suspension - labelled as such
- Take small aliquot of just-prepared suspension and add to second glass vial
- Dilute until suspension entirely transparent for lower (again unknown) relative concentration suspension - labelled as such
- Take ~0.5mL of suspension with a Pasteur pipette and drop on to polished silicon wafer (Alpha Nanotech) without spilling over edge
- Silicon wafer previously attached to aluminium stub with carbon tape of equal diameter
- Samples loaded into vacuum oven and vacuum pump (Vacuubrand MD 4 NT) exhausted oven to ≈ 1 mbar
- Oven temperature set at 40 °C and left to dry for < 1 hour

Results :

- Samples froze when vacuum applied (room and oven temperature 9 °C), in some cases ejecting frozen suspension from atop silicon with expansion.
- 8 samples - lower and higher concentration dropcast samples of both sediment and product for each synthesis

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Attached file

VacuumOven_FrozenSuspension.JPG

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