

Freeze-casting of Ti₃C₂ MXene suspension II

Date: 2021-10-22

Tags: Freeze-cast Aerogel Nanoplexus 200 2021 19/10/2021Synth

Created by: James Bird

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Goal : Freeze-cast Ti₃C₂T_z aqueous nanoparticle suspensions to give well-aligned nanoparticles in a sample within the target dimensions and tolerances

Procedure :

- Add necessary quantity of lyophilizer beakers (Labconco) to ultra-low temperature freezer at - 81 °C and note room temperature (16.1 °C)
- MXene synthesised in [\[Experiment\] MXene synthesis IX](#) is speed-mixed with the protocol below, to give a smooth, homogeneous paste with no signs of oxidation

Spin rate / rpm	500	1000	1500	2250	3000
Time / s	30	30	30	120	120

- Assemble template using Vaseline jelly to seal joints and to adhere to aluminium top-plate
- Ensure excess Vaseline is removed with a clean cotton bud
- Pour homogenous suspension into a section of the template, flatten with a spatula and place atop the freeze-plate
- Set target temperature to - 70°C on liquid nitrogen/water flow controller with a ramp rate of - 5 K/min
- Allow uni-directional freezing to complete, so that the freeze-front has visibly reached the entire sample surface
- Don waterproof cryo gloves
- Remove a lyophilizer beaker from freezer
- Remove top-plate with the attached template, deconstruct the frame and remove the sample
- Add sample to beaker and return to freezer - remove cryo gloves
- Set target temperature to room temperature on flow controller with a ramp rate of +10 K/min

The two samples have an initial suspension concentrations of 3.17 ± 0.04 wt% (32.7 mg/mL).

Prior to lyophilisation, each sample is cleaved along a plane using a hammer and

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chisel. Sample #1 (see below) is fractured parallel to the freeze-front growth direction (□ to freeze-plate) while Sample #2 is fractured perpendicular (// to freeze-plate). Once all samples have been made, they are attached to the lyophilizer in their beakers (22/10/2021 15:22) and left to dry for approximately three days (25/10/2021 12:25).

Results :

The attached .JPEG is a photographs of both samples prior to freeze-casting, hence the suspension held in the template atop the top plate. The measured ramp rate was found to be -4.90 K/min. The entirety of each sample was frozen when the target temperature was reached. Details of the lyophilized samples are summarised in the table below, where 'dim.' is an abbreviation for 'dimension':

Sample / #	Length (long dim. // to freeze-plate) / mm	Width (short dim. // to freeze-plate) / mm	Height (□ to freeze-plate) / mm	Volume / cm ³
1	21.5	15	15	4.84
2	26	15	15	5.85

Conclusions :

- No leakage occurred now that vaseline is frequently used to seal template joints
- Ramp target temperature of -70 °C appears to be sufficient for complete freezing of sample 15 mm in height
- New freeze-template needs designing to prevent sample cracking
- If in any doubt of liquid nitrogen supply it must be replenished

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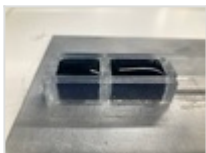
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Attached file

IMG_1726.JPEG

sha256: 4adcdf912fa68e5de1238e6d43488af3209e5e78ae116a8d70b1e011e49fa01c



Unique eLabID: 20230416-01ffea8568385abf17727e43584e8cd82e643154

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=108>