

Freeze-casting of Ti₃C₂ MXene suspension IV

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Tags: Freeze-cast Nanoplexus 400 2021 Aerogel 06/12/2021Synth KTH Collab Nanoplexus Ti₃C₂T_z 2021

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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 (17.7 °C)
- Ti₃C₂T_z MXene suspension at 48.2 mg/mL (see [Experiment - MXene mixing, dilution, homogenization and freeze-drying](#)) is speed-mixed immediately prior to use with the protocol below, to give a smooth, homogeneous paste with no signs of oxidation.

Spin rate / rpm	1500	2250	3500	1500	300
Time / s	5	120	120	5	30

- 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 (- 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
- Once all samples have been made, they are attached to the lyophilizer in their beakers and left to dry for approximately three days.

Results :

A smaller version of the newer 15x15 mm internal diameter freeze-template design (see [Experiment - Freeze-casting of Ti3C2 MXene suspension III](#)), with internal dimensions of 10 x 10 mm instead, is fabricated by 3D printing and is used here. The associated .ipt, Autodesk Inventor Part files are attached for this smaller frame. The design suffers from the same issues as discussed previously, although the two freeze-cast samples produced here correspond to the smallest target dimensions (31.75 x 10 x 10 mm (L, W x H)) meaning they are unaffected by this design flaw. The freeze-front in the first sample travelled at an average of 18.8 $\mu\text{m/s}$ and the second at 18.3 $\mu\text{m/s}$, as both froze within about 9 minutes of the freeze-plate passing 0°C.

Attached files

MouldDivider10x10internal.ipt

sha256: 0653b9e304a875c4d4fe57f653cad43601f72732a2beb3d38657f61439fce3c0

MouldEdge6x10x139.ipt

sha256: bae870afbc4081997fe8132b8f4af2d73ec2087eb0c2af4f532add0b628163a0

MouldSeal2x3x10mm.ipt

sha256: 6f7994b707e44839fce223e50331a45212471b0e6289bf7c102f4f95de2ef21d



Unique eLabID: 20230527-68ad4783c3eec8b6c807a9d6d373b894514772e0

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=126>