

SEM - Zeiss Sigma VP FEG-SEM (SDD + EBSD) I

Date: 2021-09-09

Tags: SEM EDX Sigma 18/08/2021 Synth Nanoplexus 400 2021

Created by: James Bird

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Goals :

1. Image microstructure of freeze-cast (FC) and lyophilised $\text{Ti}_3\text{C}_2\text{T}_z$ aqueous suspension
2. Use EDX on freeze-cast sample to verify nanoparticle composition
3. Image dropcast dilute $\text{Ti}_3\text{C}_2\text{T}_z$ aqueous suspension to obtain a particle-size distribution with subsequent image analysis

Sample preparation:

Dropcast samples:

- MXene synthesis products are produced in [\[Experiment\] MXene synthesis VII](#) and diluted in [\[Experiment\] Dynamic Light Scattering \(DLS\) for nanoparticle size-distribution acquisition IV](#)
- The two least concentrated suspensions, at 9.4×10^{-3} and 1.9×10^{-3} wt% are dropcast using the methodology as described elsewhere (see [\[Experiment\] SEM stub preparation for PSD VII](#))

Freeze-cast samples:

- Sample #1 produced in [\[Experiment\] Freeze-casting of \$\text{Ti}_3\text{C}_2\$ MXene suspension](#) is sliced with a scalpel into smaller fragments using a scalpel, such that the exposed top faces which can be imaged in the microscope are detailed in the table below:

Sample	Upward face
A	// to freeze-direction and temperature gradient (i.e. plane \perp to freeze-plate)
C	Same as above, but cut at 90° around the axis \perp to freeze-plate
D	\perp to freeze-direction and // to freeze-plate
E	Same as above

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- Each fragment is mounted atop an aluminium SEM stub, using sticky carbon tape as a conductive adhesive between both the stub and the sample

Procedure :

- Load samples into SEM chamber and acquire vacuum
- Accelerating voltage set at 5 keV and WD at 3 mm for dropcast nanoparticle imaging or at 5 - 20 keV and 8.5 - 15 mm WD for freeze-cast microstructure imaging
- Working distance set at AWD of 8.5 mm for EDX
- Everhart-Thornley secondary electron (SE) detector used for FC microstructure imaging and in-beam SE detector employed for dropcast nanoparticle imaging

Results :

Sample	.tif filename prefix(s)	'LineScan'/'Sum Spectrum' .docx/.txt N° / #	.png 'Electron Image' N° / #
MXene synthesis product dropcast at 1.9×10^{-3} wt%	ActualCofefeStainEdge CoffeeStainEdge FlakeOverlap OutsideCoffeeStainEdge OutsideStan		
Freeze-cast Sample A	FC_SampleA	1-3	1-3
Freeze-cast Sample C, D or E	FC_SampleCDE		

.tif files are micrographs taken directly in the SEM, .png files are electron micographs with annotations, captured via the EDX software program Aztec 5.0, .dat are EDX data files, .docx are reports generated in Aztec with zoomed micrographs depicting linescan regions, .txt files are xy column data of the sum

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spectra of each linescan and .oip is the Aztec project file.

Freeze-cast samples:

Micrographs of the freeze-cast and cut sample, consisting of $\text{Ti}_3\text{C}_2\text{T}_z$ aqueous suspension FC at 14.83 wt% (~ 150 mg/mL), show the interconnected network of nanoparticles formed as a result of the freeze-casting and lyophilization process. Away from the smoother surfaces seen at low resolution, which may have been smoothed by the scalpel slicing, the structure is seen to be highly porous, as is typical of aerogels. Note that in all images of FC Sample A, the bottom of the image is aligned with the surface where the freeze-plate was situated. Hence, when freeze-casting under these particular conditions (suspension concentration, temperature ramp, PSD) there is no visually-identifiable orientation preference on nanoparticles.

When using EDX, the first linescan conducted at 5 keV again supports the conclusions found previously in [\[Experiment\] SEM - Zeiss Sigma VP FEG-SEM \(SDD + EBSD\)](#) that 5 keV accelerating voltage is insufficient to provoke measurable Ti K_α X-ray emission (at 4.5 keV), as nanoparticles are clearly visible on the sample surface, yet no measurable titanium concentration is found. Increasing the accelerating voltage to 15 keV for the second linescan (actually the sum of multiple linescans) over the same region gave the average values stated in the table below, returning an average Ti/C atom ratio of 1.13 compared with the theoretical 1.5 from stoichiometry:

Element	Weight %		Atom %
Ti	62.7	0.1	35.3
C	13.9	0.0	31.2
O	11.5	0.1	19.4
F	7.7	0.0	10.9
Cl	4.1	0.0	3.1
Al	0.1	0.0	0.1

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For the third linescan, a seemingly more dense region of MXene nanoparticles is chosen and the accelerating voltage is set at 20keV. The average element concentrations are listed in the table below, where the Ti/C atom ratio is 0.89. These findings suggest that either the Ti/C stoichiometry is indeed $< 3:2$ or that carbonaceous contamination is present in the microscope chamber on the sample surface. There was no improvement in optimising the deadtime on increasing accelerating voltage from 15 to 20 keV. Note that aluminium presence in both linescans 1 & 2 is negligible, but chlorine is not.

Element	Weight %		Atom %
Ti	56.8	0.2	29.8
C	16	0.1	33.5
O	13.3	0.2	20.9
F	9.6	0.1	12.7
Cl	4.3	0.0	3.0
Al	0.1	0.0	0.1

Dropcast samples:

Nanoparticles are clearly visible across many micrographs. Lateral dimensions seem to vary from hundreds of nanometres to microns, although particle overlap in micrographs renders image analysis for PSD production challenging, so the dropcast suspension can be deemed too concentrated. Oxidation (small, bright regions) appear consistently across nanoparticle surfaces and edges, as anticipated due to the 22 day separation between the synthesis beginning and SEM analysis.

Conclusions :

- 15 keV accelerating voltage EDX seems optimal for FC structure compositional analysis
- Aluminium concentration is negligible in the MXene synthesis product used in freeze-casting, whilst chlorine concentration is less negligible and can be

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presumed to be a terminating group following the literature

- Fracturing FC structures must be done when samples are frozen to avoid smoothing of surface during incision via shear
- The suspension at ~ 150 mg/mL is too high in concentration for lamellar ice-growth which would lead to visible nanoparticle alignment in the lyophilized FC structure
- When dropcasting suspensions for PSD production, the concentration should be $< 1 \times 10^{-3}$ wt% to enable image analysis

Attached files

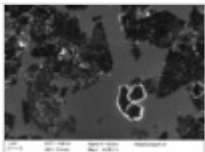
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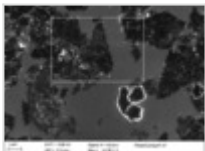
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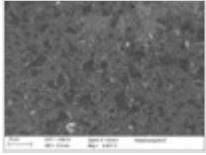
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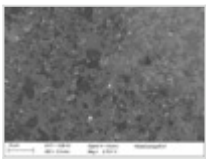
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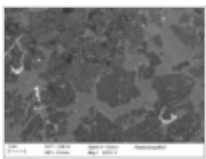
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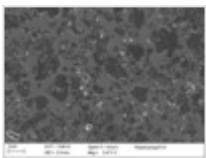
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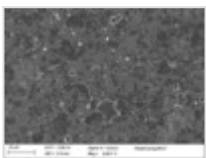
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FlakeOverlap08.tif

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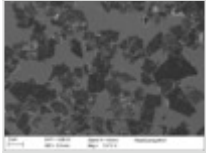
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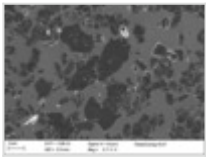
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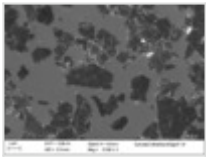
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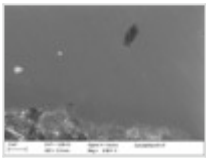
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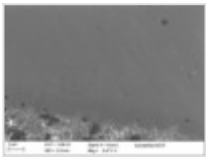
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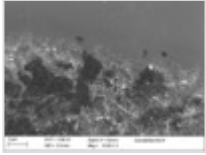
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OutsideStain04.tif

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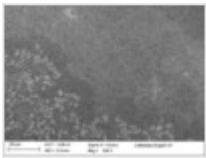
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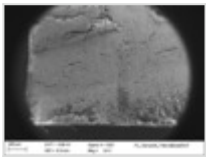
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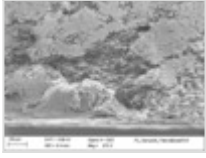
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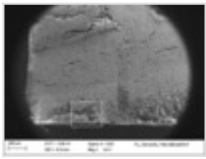
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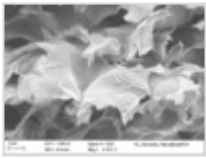
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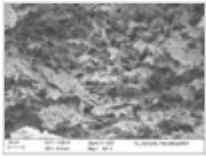
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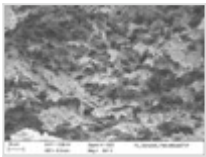
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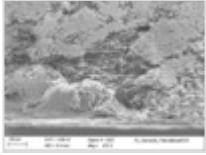
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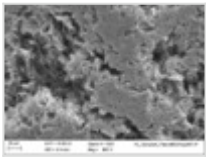
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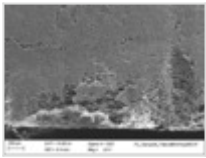
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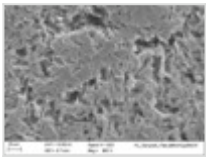
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FC_SampleCDE_Centre_NoPO01.tif

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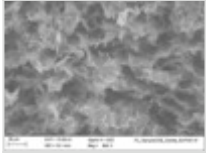
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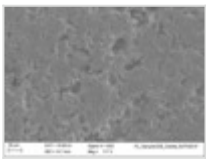
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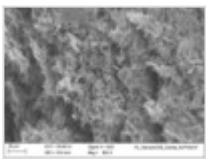
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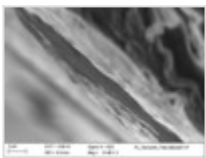
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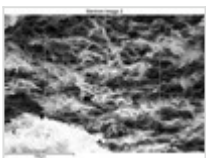
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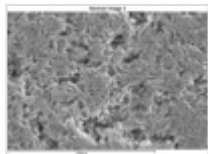
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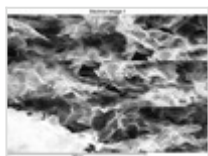
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Electron-Image-1.png

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72066977-389c-4e65-b8e7-787aa8b02e6a_17.dat

sha256: 9b02eec346dea5cede1cc66621b609ebfe35d86bb2459f707e8c0f2523be9dee

72066977-389c-4e65-b8e7-787aa8b02e6a_22.dat

sha256: 03cbf2d9f8ceea17cab499dc1e5ac57b5485c7a5166f92fa0c7c2276e9aa1754

a833026e-0cd0-426c-8edc-920c1b63d125_6.dat

sha256: e9dce5a3fdd1bc22bba071233af4b6a721c165c7cf0617028b74cb5ac8146ca9

a833026e-0cd0-426c-8edc-920c1b63d125_8.dat

sha256: a546fe6a5948b9755c35598467c50e7b095d82be3177b288f11467e5bcf69803

a833026e-0cd0-426c-8edc-920c1b63d125_9.dat

sha256: 09e71a30b04439477d318960ce9a59265d455d40f9f29880c6bdcb8dbe591bfd

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Tags: SEM EDX Sigma 18/08/2021 Synth Nanoplexus 400 2021

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a833026e-0cd0-426c-8edc-920c1b63d125_13.dat
sha256: 890d3f1387748af2c8240746ab41da2d203137e312341d4b13f15631fb76aeb9

a833026e-0cd0-426c-8edc-920c1b63d125_17.dat
sha256: 67ddbf33a7e57f94ad3ba86343fb4b748aa922ba096446178989419a6c8e8499

a833026e-0cd0-426c-8edc-920c1b63d125_22.dat
sha256: 47ce6c7c5e934f8ea0c65a9bd978824530406f1d6b0e43e98bcd3109afaef89e

abc2c5a1-9543-4f62-a150-0552f4adcbb8.dat
sha256: 9b27e0455bcef11c0af90e1c6bc82064ea074fea8a26cbf8a417762a8a098281

ac1cfe48-8459-4b33-9feb-56fb5534b1dclive.dat
sha256: 11e343a70733f068eba4739e7982bc6ca6a287383db647702507927a070b795f

ac1cfe48-8459-4b33-9feb-56fb5534b1dcreal.dat
sha256: b4b1d1389d4c5919f06f4dac3f25d934f303a54cbd677c80368af87d6de1e7ac

LineScan2.docx
sha256: 5395ca6a51bd20d1fa750c3975657d66f5a9b49546ec9b62bb6d73115770ac25

LineScan1.docx
sha256: 9f5513dc13cc1590e884097c3077c0613cb76cf37da915e815310d03dd902ec4

Line-Sum-Spectrum-1.txt
sha256: 9951568226078db24b0b4a8a3413caab1fd2dcb81bf3cedd2ecc6d2272068f2f

LineScan3.docx
sha256: 528427db1a3a0855078c2729f900b6169c7cf718dac2db2b4a17cd9e26d05248

Line-Sum-Spectrum-2.txt
sha256: 6ce8789dc9ef5cb8a35aeab965b53799261df0a25bb51065ef4b54771864120a

Line-Sum-Spectrum-3.txt
sha256: f9c399a044521e54d7883a6f296ab7e6133d921568e5df070310115865388924

5c78a3e7-4791-44fd-a7cc-751731377dd9live.dat
sha256: 11e343a70733f068eba4739e7982bc6ca6a287383db647702507927a070b795f

03d725a2-581b-4fae-805d-f718afe9db2freal.dat

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sha256: 45d86753a1e0b24f7206123b71ecf96e89dcd6ebc1c754f03c3c356cbdbac34b

5c78a3e7-4791-44fd-a7cc-751731377dd9real.dat

sha256: 2953b112131f33b8b0cc7907f40bc14965948d38a4eecd1c987b6f7ed939e67

5d6cfa62-8bfc-42b9-bbce-b6b7cedae34e_6.dat

sha256: 95cd376c0791eb1d7bbb066593033a4d78dc3548c5be131070ceb7668b5e7194

5d6cfa62-8bfc-42b9-bbce-b6b7cedae34e_8.dat

sha256: 16a7e648008b09db8745226227f4015b9c8a5b24ec31e79d7a481300f6d5c535

5d6cfa62-8bfc-42b9-bbce-b6b7cedae34e_9.dat

sha256: 8811d6ced138844af2866c990c226d7698dca78f81bbab519a23a18b5d2bd2c6

5d6cfa62-8bfc-42b9-bbce-b6b7cedae34e_13.dat

sha256: 93ff4eb7e22755342c232a1aec4ff46f3bb50d0719325232822154be8511f0bd

5d6cfa62-8bfc-42b9-bbce-b6b7cedae34e_17.dat

sha256: 5f47e7b44706b63f9a8d1d1adc5cc5aa00c8cff4f80df6ddec2aaacf48dab712

5d6cfa62-8bfc-42b9-bbce-b6b7cedae34e_22.dat

sha256: f358a500b0468f090426be49dced8dd7f64e2a53d313d4384f85bb9397ec8714

7aace10b-627c-48e4-9e6e-0ccc227a43ca.dat

sha256: 8aedf987517c91b14d3d27e448c5d4f88152c567432d7c39a39436b81ef59411

9bf627b5-9d93-46d2-91a8-1a7e8ece9bf7.dat

sha256: 4be100ce12c91c14ae37e67d6b6f887415a6e0ee9584ca8c5e26166b449239d0

40c0b392-d1a1-46f6-9d55-349a83c5a42elive.dat

sha256: 2ed6d1146d84156e1bf0a9a95fd1f68fb6c8d59f8a2c1440d6b27573b57faa47

40c0b392-d1a1-46f6-9d55-349a83c5a42ereal.dat

sha256: 7807fe7e406b7fe1f373c575502dd43bdb3c715ff647fd004d90d045ab10c1a7

53ebeece-8761-4710-bb74-94090f8652f4.dat

sha256: d349da4def316addea7cef8ff1fee10d78af3c34be6be411ec82809b62c1cc37

01318484-c6d3-4a44-b597-ce28879c08e8live.dat

sha256: 11e343a70733f068eba4739e7982bc6ca6a287383db647702507927a070b795f

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01318484-c6d3-4a44-b597-ce28879c08e8real.dat
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sha256: 0aa4d05661e5b229c6e5b92aa040785f891ec46d3ceb96cf08ac17a52f1a1cdf

72066977-389c-4e65-b8e7-787aa8b02e6a_6.dat
sha256: f7ce8cd4a61a160efc97ef2e96521fc8f7e3da27334d5231acd08ded6474365a

72066977-389c-4e65-b8e7-787aa8b02e6a_8.dat
sha256: 175fe537455a22d98fa59aceed44d52fd63cba0142b56915bcca88b04dfd6e99

72066977-389c-4e65-b8e7-787aa8b02e6a_9.dat
sha256: f8acac4030a703fb4e79371b39d642080153ec0635500927f60f94aa19bf190d

72066977-389c-4e65-b8e7-787aa8b02e6a_13.dat
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72066977-389c-4e65-b8e7-787aa8b02e6a_17.dat
sha256: 9b02eec346dea5cede1cc66621b609ebfe35d86bb2459f707e8c0f2523be9dee

72066977-389c-4e65-b8e7-787aa8b02e6a_22.dat
sha256: 03cbf2d9f8ceea17cab499dc1e5ac57b5485c7a5166f92fa0c7c2276e9aa1754

a833026e-0cd0-426c-8edc-920c1b63d125_6.dat
sha256: e9dce5a3fdd1bc22bba071233af4b6a721c165c7cf0617028b74cb5ac8146ca9

a833026e-0cd0-426c-8edc-920c1b63d125_8.dat
sha256: a546fe6a5948b9755c35598467c50e7b095d82be3177b288f11467e5bcf69803

a833026e-0cd0-426c-8edc-920c1b63d125_9.dat
sha256: 09e71a30b04439477d318960ce9a59265d455d40f9f29880c6bdcb8dbe591bfd

a833026e-0cd0-426c-8edc-920c1b63d125_13.dat
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a833026e-0cd0-426c-8edc-920c1b63d125_17.dat
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a833026e-0cd0-426c-8edc-920c1b63d125_22.dat

SEM - Zeiss Sigma VP FEG-SEM (SDD + EBSD) I

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Tags: SEM EDX Sigma 18/08/2021 Synth Nanoplexus 400 2021

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sha256: 47ce6c7c5e934f8ea0c65a9bd978824530406f1d6b0e43e98bcd3109afaef89e

ac1cfe48-8459-4b33-9feb-56fb5534b1dclive.dat

sha256: 11e343a70733f068eba4739e7982bc6ca6a287383db647702507927a070b795f

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sha256: 9b27e0455bcef11c0af90e1c6bc82064ea074fea8a26cbf8a417762a8a098281

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sha256: 11e343a70733f068eba4739e7982bc6ca6a287383db647702507927a070b795f

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sha256: 329460fd2c40b15ba1fea744680a27a275ddb106c7757fd89f4b623606e41700

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d5bccc09-8553-40a2-b7d9-28fc6fc9ebc1_17.dat

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d5bccc09-8553-40a2-b7d9-28fc6fc9ebc1_22.dat

sha256: 0f0670f9da0460d81a9ed35a905ae4459c955a8f5f48130bfee052710c1bc837

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d745f666-73b5-4108-b527-500b69b5baa9.dat

sha256: ee5c54e23521a2cfe81f11b7740a10a9e091b471d8d93c3af6ad86a773bc4e6d

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Tags: SEM EDX Sigma 18/08/2021 Synth Nanoplexus 400 2021

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03d725a2-581b-4fae-805d-f718afe9db2flive.dat
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5d6cfa62-8bfc-42b9-bbce-b6b7cedae34e_8.dat
sha256: 16a7e648008b09db8745226227f4015b9c8a5b24ec31e79d7a481300f6d5c535

5d6cfa62-8bfc-42b9-bbce-b6b7cedae34e_9.dat
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5d6cfa62-8bfc-42b9-bbce-b6b7cedae34e_13.dat
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7aace10b-627c-48e4-9e6e-0ccc227a43ca.dat
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sha256: 7807fe7e406b7fe1f373c575502dd43bdb3c715ff647fd004d90d045ab10c1a7

53ebeece-8761-4710-bb74-94090f8652f4.dat

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01318484-c6d3-4a44-b597-ce28879c08e8live.dat

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72066977-389c-4e65-b8e7-787aa8b02e6a_8.dat

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72066977-389c-4e65-b8e7-787aa8b02e6a_9.dat

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72066977-389c-4e65-b8e7-787aa8b02e6a_17.dat

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72066977-389c-4e65-b8e7-787aa8b02e6a_22.dat

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a833026e-0cd0-426c-8edc-920c1b63d125_6.dat

sha256: e9dce5a3fdd1bc22bba071233af4b6a721c165c7cf0617028b74cb5ac8146ca9

a833026e-0cd0-426c-8edc-920c1b63d125_8.dat

sha256: a546fe6a5948b9755c35598467c50e7b095d82be3177b288f11467e5bcf69803

a833026e-0cd0-426c-8edc-920c1b63d125_9.dat

sha256: 09e71a30b04439477d318960ce9a59265d455d40f9f29880c6bdcb8dbe591bfd

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a833026e-0cd0-426c-8edc-920c1b63d125_13.dat
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sha256: 67ddbf33a7e57f94ad3ba86343fb4b748aa922ba096446178989419a6c8e8499

a833026e-0cd0-426c-8edc-920c1b63d125_22.dat
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d5bccc09-8553-40a2-b7d9-28fc6fc9ebc1_8.dat
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d5bccc09-8553-40a2-b7d9-28fc6fc9ebc1_13.dat
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sha256: 0f0670f9da0460d81a9ed35a905ae4459c955a8f5f48130bfee052710c1bc837

d85f1487-0e92-4230-81f7-de462f7e9a35.dat

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FreezecastEDX.oip

sha256: b2a67992de9be8c35afe9ce34bb17703de3dff70bbf9665f1ed92bdd6330aac9



Unique eLabID: 20230314-a0849f43619a16ac23778bbb2e69f8df5364b4c6

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=94>