

Sublimating frozen CsCl solutions in ESEM: the number and size of salt particles are influenced by the ice sublimation temperature, salt concentration and freezing method

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Text S1. Calculating the particle density

The shape of the droplet was approximated by a spherical cap with a base diameter of 4 mm and a height of 1 mm. The maximum drop height was measured using an electron microscope, as a difference in the working distance between the plane of focus of the drop-top and the surface of the silicon pad (wafer). The droplet's base and volume were calculated to be 12.6 mm² and 6.81 mm³, respectively.

The surface densities are estimated based on the image analyses of the micrographs via the Mountains® software (Digital Surf). A few representative spots were analyzed for each salinity except for 0.5M CsCl; that calculation is not provided, as no small salt particles were detected in the highly concentrated sample.

Average volume densities of the salt particles (a number of particles generated from 1 mm³ of the original frozen sample after the ice sublimation) were calculated via multiplying the average surface densities by the area of the base (12.6 mm²), and subsequently dividing this total number of salt particles in a sample by the volume of the droplet (6.81 mm³).

		0.005 M CsCl, non-seeded	0.005 M CsCl, seeded	0.05 M CsCl, non-seeded	0.05 M CsCl, seeded
Surface density of salt particles / mm ⁻²	Spot #1	5550	7730	56900	62800
	Spot #2	1260	12000	21500	59500
	Spot #3		22900		43800
	Spot #4		8400		72400
	Spot #5		8640		
	Mean	3405 ± 3033	11934 ± 6350	39200 ± 25032	59625 ± 11885

Volume density of salt particles / mm ⁻³		6300 ± 5613	22081 ± 11748	72528 ± 46314	110319 ± 21989
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Table S1. The calculated surface density (mm⁻²) and volume density (mm⁻³) with sample standard deviation under different conditions.

Supplementary figures

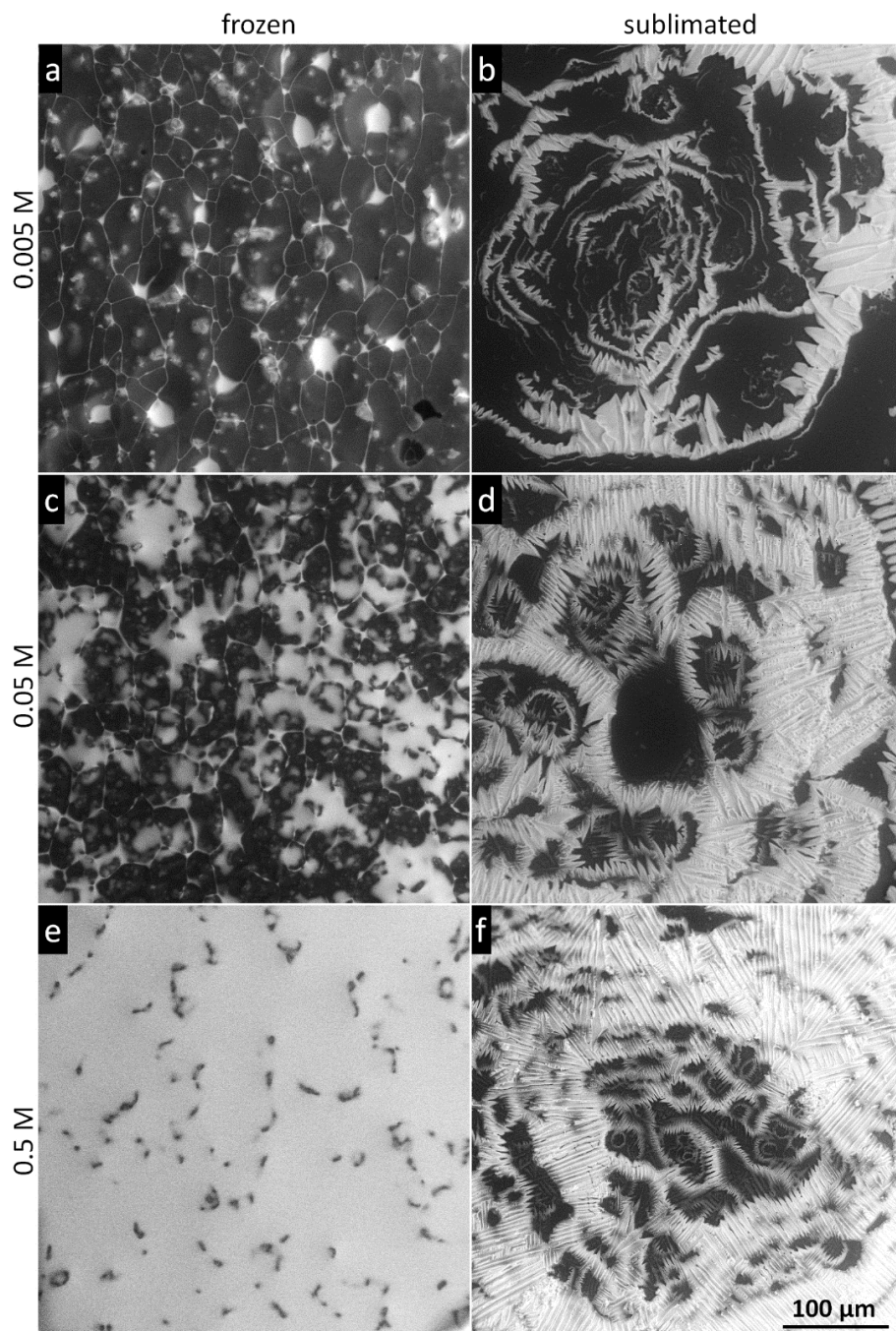


Figure S1: The structure of the frozen samples (before the sublimation) and the CsCl salt residua after the sublimation of the 0.005, 0.05, and 0.5 M non-seeded frozen samples at $-20\text{ }^{\circ}\text{C}$, i.e., above the T_{en} . In the frozen samples (panels a, c, and e), the white and dark areas represent liquid brine and the ice, respectively. In the sublimated samples (panels b, d, and f), the white and dark areas represent crystallized CsCl salt and the silicon pad, respectively.

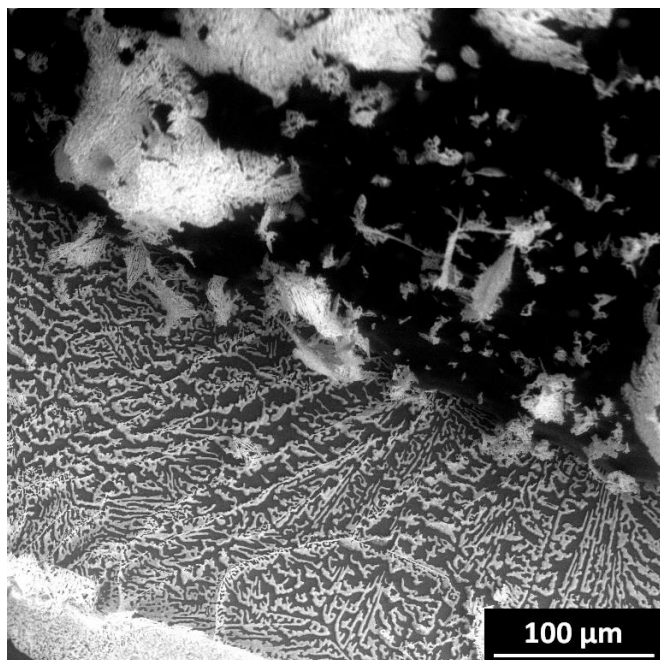


Figure S2: Partially sublimated 0.05 M seeded sample. Prior to sublimation, the frozen sample was cooled down to -25°C , then heated up to -20°C , then cooled down to -25°C again. When heated to -20°C , the brine at the surface became liquid and leaked to the edge of the sample where it solidified after further cooling. Left-down corner displays a broader salt rim of the drop caused by the liquefaction of the brine above its T_{eu} .

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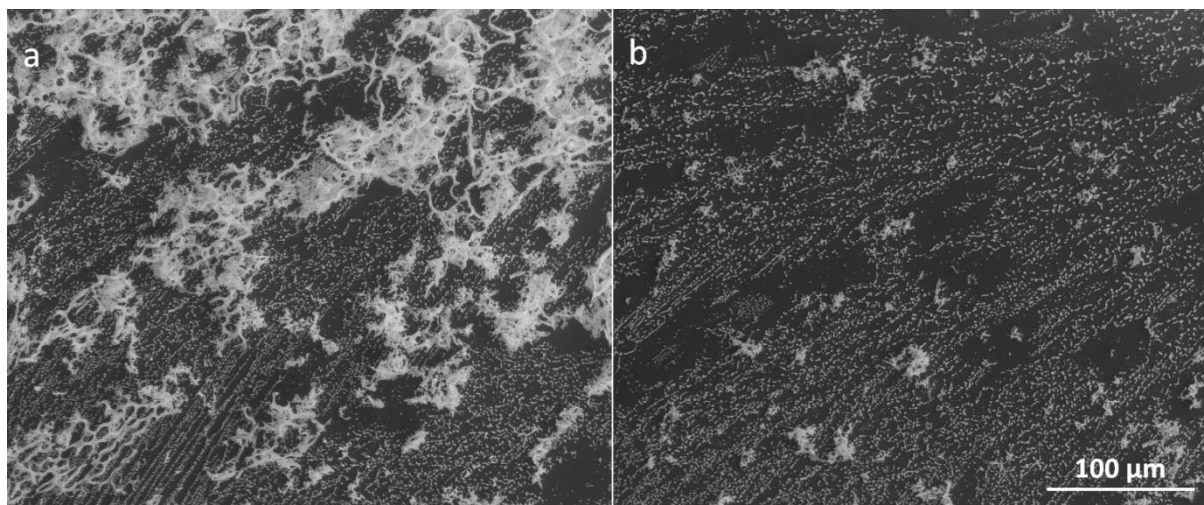


Figure S3: The salt residues in 0.05 M non-seeded sample just after the sublimation process at -25°C (a) and after venting the chamber, blowing the sample with air gun, and evacuation procedure (b). A same spot is imaged in both panels. The scale in panel b is valid for both the images.

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