Supplementary Information: Watching Mesoporous Metal Films Grow During Templated Electrodeposition with In Situ SAXS

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S1. Simulated Azimuthal angles for Fd3m symmetry where the (111) plane is aligned parallel to the substrate

The phase and lattice parameter of the sample are identified from the characteristic peaks shown in the 1D scattering profile. The d spacing, *d* and the lattice parameter, *a* are related to the Miller indices of the reflection, (*hkl*), by

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

The azimuthal angle, χ between the uniaxial axis of rotation (horizontal on the images) and a given predicted spot is approximately given by

$$\cos(\chi) = \frac{[h^r k^r l^r] \cdot [hkl]}{|[h^r k^r l^r]||[hkl]|}$$

where $[h^r k^r l^r]$ is the reflection being simulated and [*hkl*] is the uniaxial axis of rotation located parallel to the substrate normal ([111] for Q_{II}^{D} and [110] for Q_{II}^{G}). For each (hkl) reflection there are multiple {hkl} combinations that contribute to the scattering where |(hkl)|=|{hkl}| and for each of these {hkl} variation there are reflections with azimuthal angles ±χ.

S2. Wedge radial profile of nanostructured platinum and lipid template

Radial profiles within 25° wedges extending in the positive and negative X and Y directions from the centre of the SAXS image of a platinum nanostructure with the lipid template in place (shown in Fig. 4b) were taken in the xy and zy plane. The average signal from the wedges extending in the positive and negative of each direction are shown in Fig. S2. For comparison, the radial profile within wedges is also shown for a previously reported platinum nanostructure with the template removed[1]. It can be observed that the peak positions do not match indicating a non-cubic structure.

Wedge position and size were selected in order for the X and Y average to integrate over the on- and off-axis reflections respectively. According to predicted Azimuthal angles for a (111) orientation on-axis reflections will occur where (hk + kl + hl) = 3, whereas for off axis reflections (hk + kl + hl) = - 1. The 1/d spacing of the off-axis and on-axis reflections does not agree for the film with the template removed which suggests a rhombohedral distortion of the cubic lattice according to,

$$\frac{1}{d^2} = \frac{(h^2 + k^2 + l^2)sin^2\alpha + 2(hk + kl + hl)(cos^2\alpha - cos\,\alpha)}{a^2(1 - 3cos^2\alpha + 2cos^3\alpha)}$$

where the lattice parameter is found to be, a = 147 Å and the lattice angle, α = 87.8.

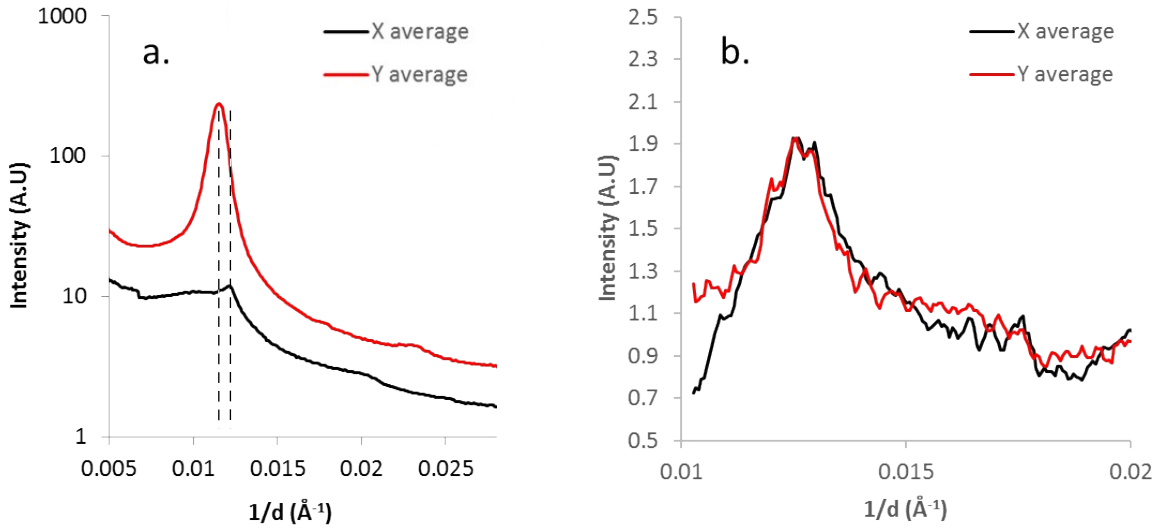

Figure S2. Radial profiles taken within 25˚ wedges extending out from the beam centre in the positive X and Y directions for platinum films with the template removed (a) and still in place (b). Dashed lines on (a) demonstrate the difference observed in peak positions.

S3. Disorder of samples deposited using different deposition potentials

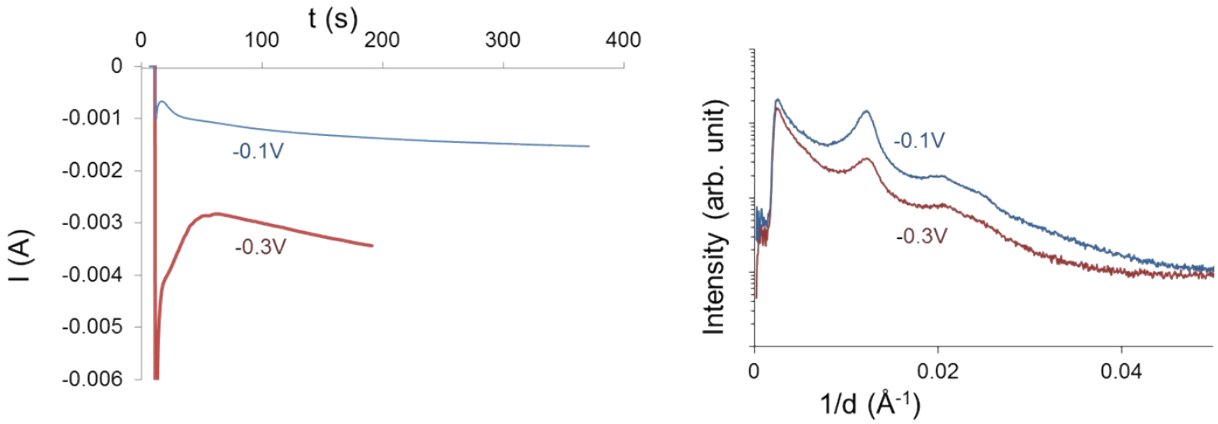

Figure S3. Left: deposition transients for mesoporous platinum deposited onto two archival gold DVDs of area 0.7 cm² at -0.1V and -0.3V vs Ag/AgCl. Average current density values during deposition were -1.8 mA/cm² and -4.3 mA/cm² respectively. Right: integrated transmission SAXS patterns from the same two samples.

References

(1) Richardson, S. J.; Burton, M. R.; Staniec, P. A.; Nandhakumar, I. S.; Terrill, N. J.; Elliott, J. M.; Squires, A. M. Aligned Platinum Nanowire Networks from Surface-Oriented Lipid Cubic Phase Templates. *Nanoscale* **2016**, *8*, 2850–2856.