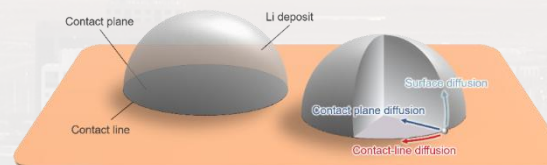
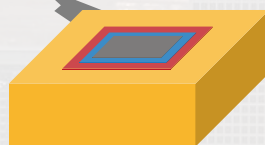
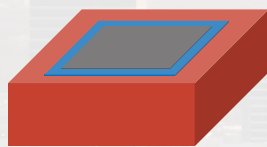
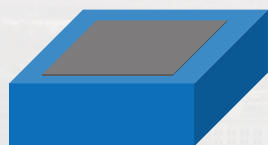


# Li deposition mechanisms: a perspective by combining imaging with theoretical analysis

**Tao Gao, PhD**

Assistant Professor  
Department of Chemical Engineering  
University of Utah



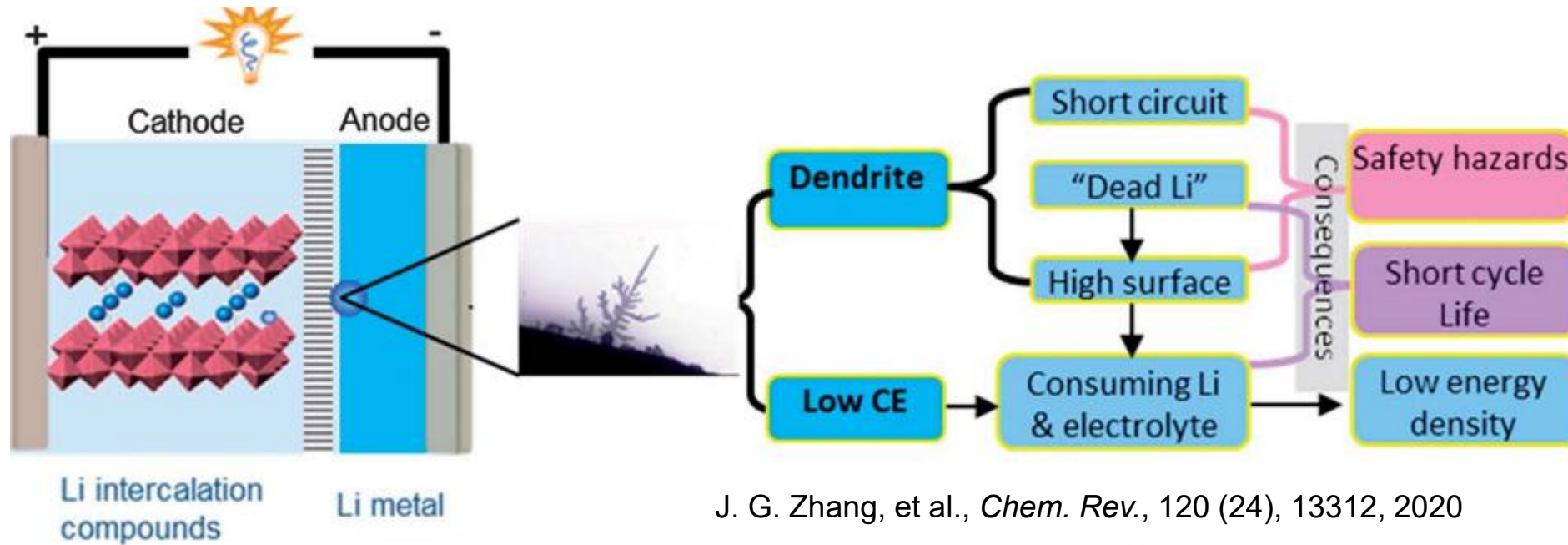
✉ [taogao@chemeng.utah.edu](mailto:taogao@chemeng.utah.edu)

🌐 [taogao-echem.net](http://taogao-echem.net)

🐦 [@TaoGao2020](https://twitter.com/TaoGao2020)

# Li metal anode: motivation and challenges

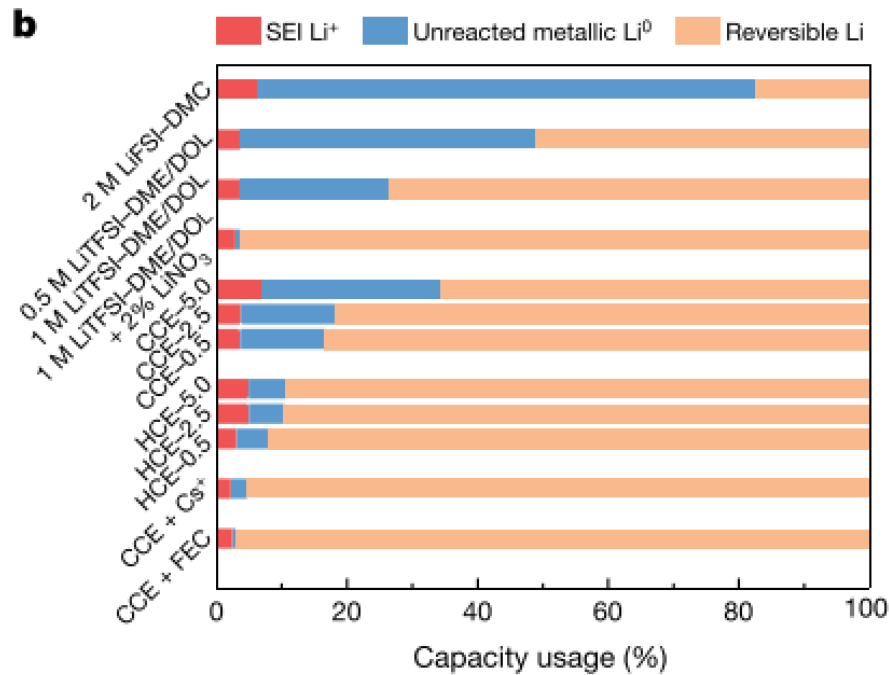
**Motivation:** High energy batteries, i.e., Li-S, Li-O<sub>2</sub>, Li-NMC



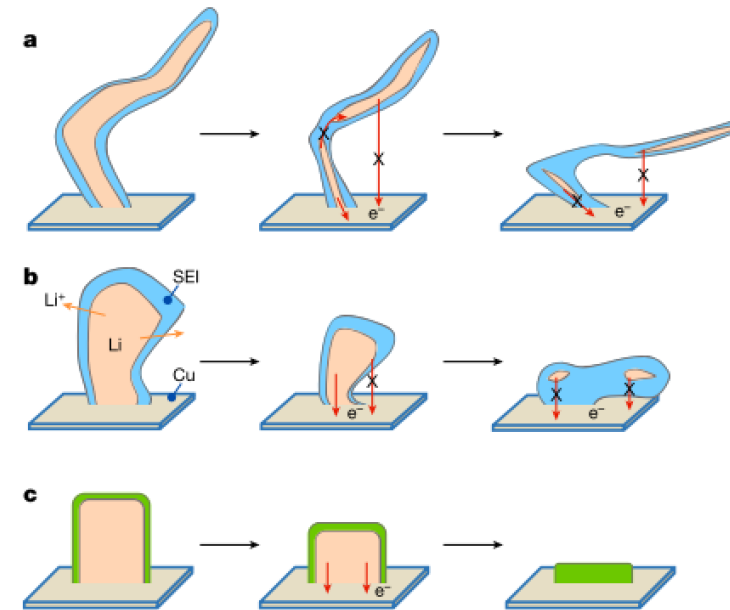
## Challenges:

- Dendrite formation
- Low columbic efficiency (CE)
  - SEI formation
  - Dead Li

# What prevents CE from reaching 100%?



Y. S. Meng et al, *Nature* **2019**, 572, 511.

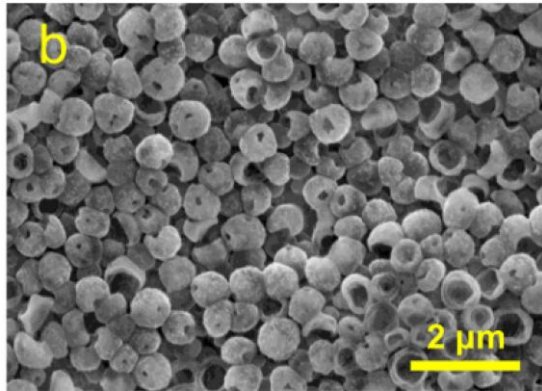


J. G. Zhang, et al., *Chem. Rev.*, 120 (24), 13312, 2020

- **Dead Li** is the major cause
- **Morphology matters**: skinny deposits with high surface area have more dead Li
- **Ideal** Li deposits:
  - Large granular size with low surface area
  - Uniform SEI to allow uniform dissolution

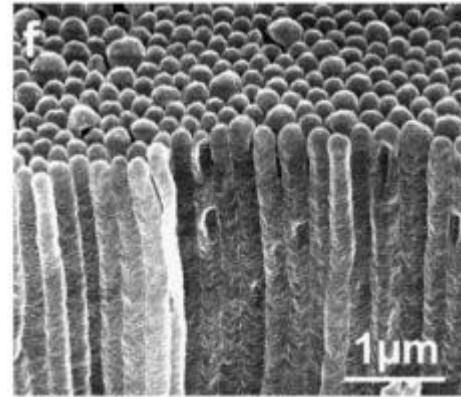
# Many different morphologies observed

(hemi-)sphere



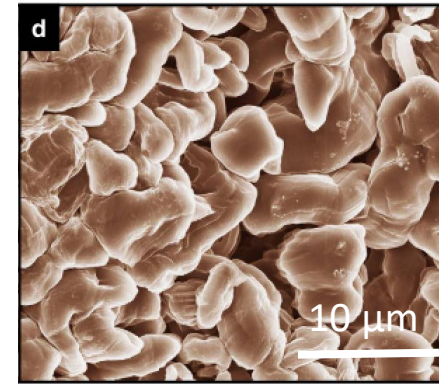
Y. Cui et al, *Nano Lett.*, 2017, 17(2), 1132

compact nanorods



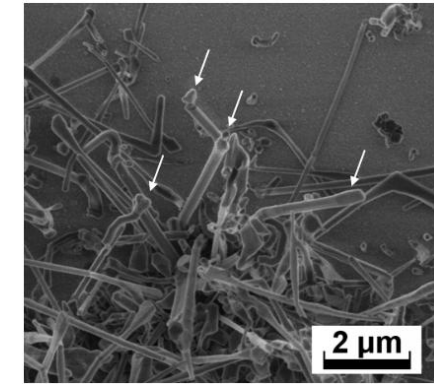
J. G. Zhang et al, *Nano Energy* 2015, 15, 135.

granular



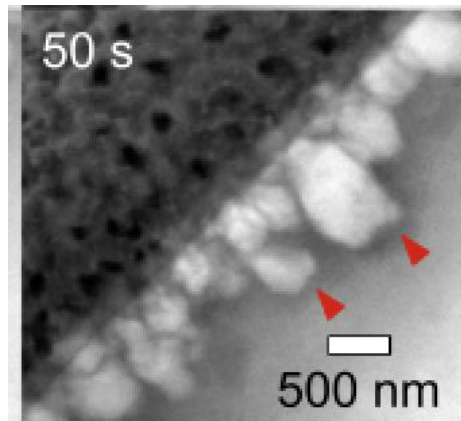
J. G. Zhang et al, *Nat. Commun.* 2015, 6

whisker



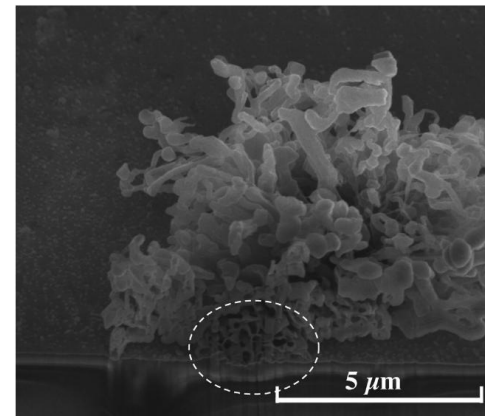
R. Monig, *J. Power Sources* 2014, 261, 112.

Eden-cluster



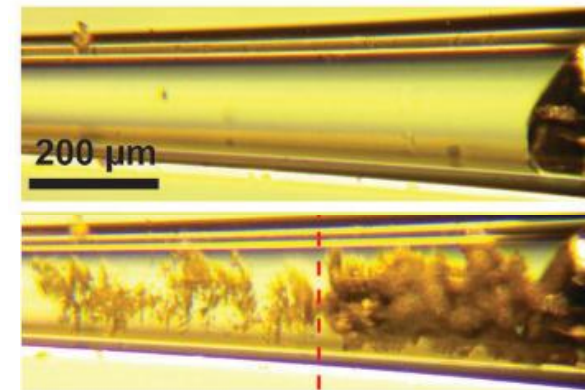
M. Z. Bazant, J. Li et al, *Nano Energy* 2017, 32, 271.

bushes



R. Monig et al, *Electrochim. Acta* 2014, 136, 529

tip-splitting dendrite

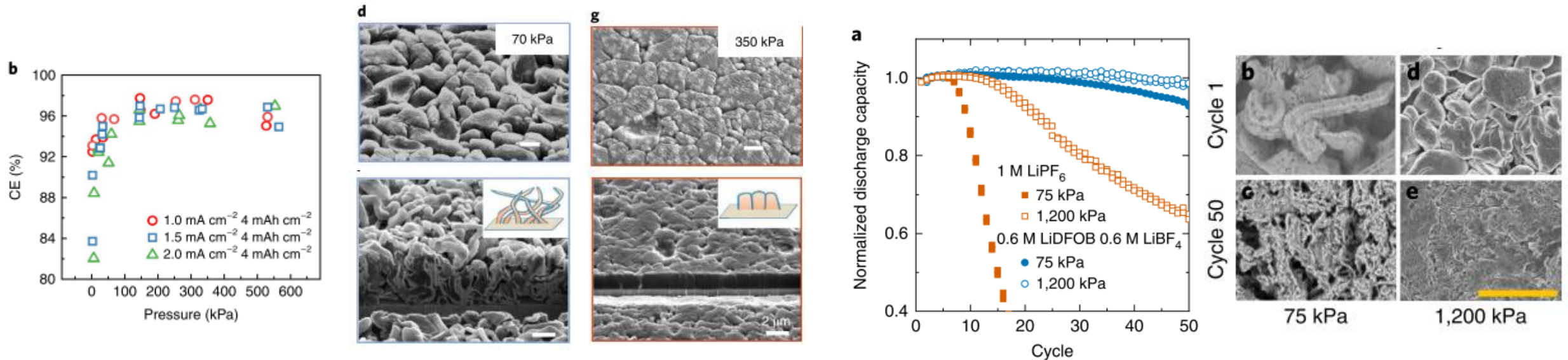


P. Bai, M.Z. Bazant et al, *Energy Environ. Sci.* 2016, 13.

# Fundamental Questions

- How do **different morphologies** form?
  - How do various **factors** regulate morphological evolution?
    - P, T, J, electrolyte, substrate...
- How does **morphology** correlate with **CE** and **safety**?

**in-situ** experiment can monitor the deposition in real-time

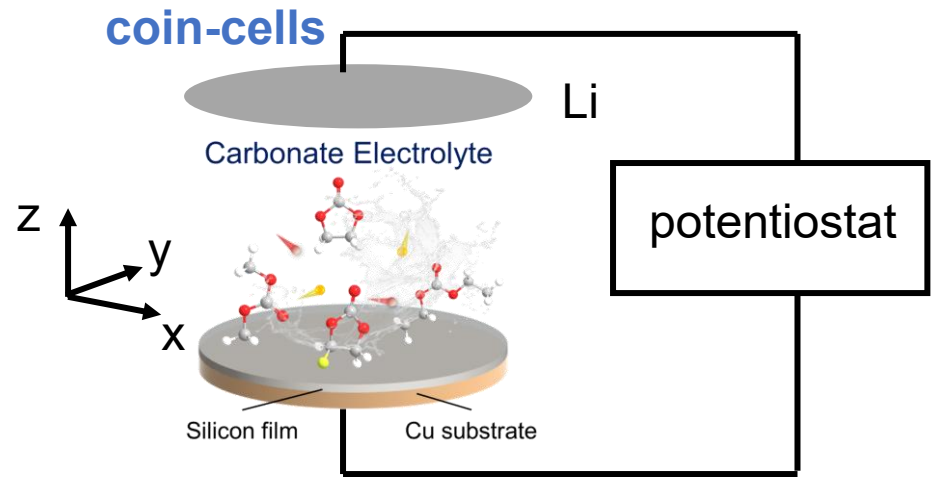


S. Y. Meng et al., Nature Energy, 2020,6,987

J. Dahn, et al, Nature Energy, 2019, 4(8), 683–689.

**ex-situ** study can better reproduce the actual operation condition

# Ex-situ SEM imaging + theoretical analysis

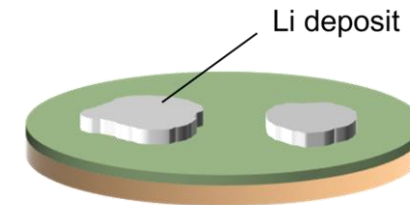


deposition at different  
**Current (I)**  
**Capacity (Q)**  
**Pressure (p)**  
**Temperature (T)**



disassemble  
 within 5 mins

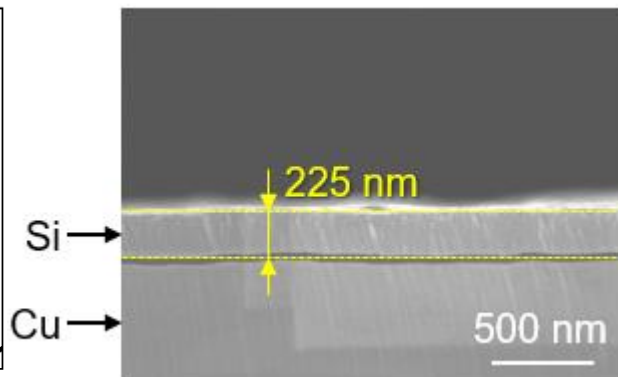
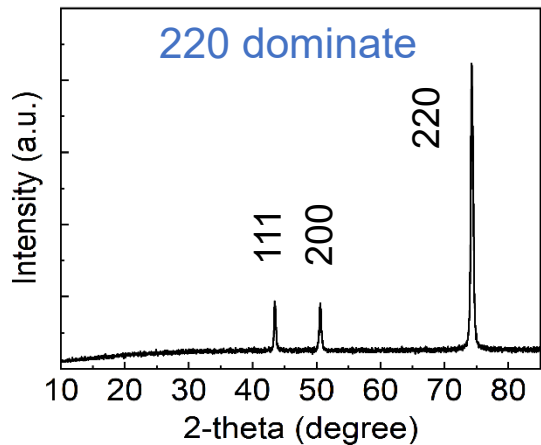
SEM imaging  
 +  
 theoretical analysis



**Electrolyte:** 1M  $\text{LiPF}_6$  in EC:DEC:DMC (1:1:1 vol) + 5wt% FEC

**Cu:** Commercial foil

**Si:** amorphous nano-film on Cu



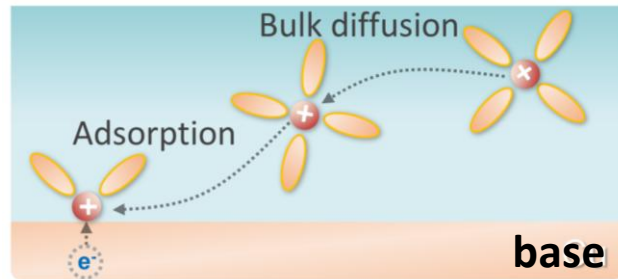
Scope:

- 1) how Li **nucleates** and **grows**?
- 2) how does the **morphology** affect **CE** and **safety**?

# Physico-chemical processes in Li deposition

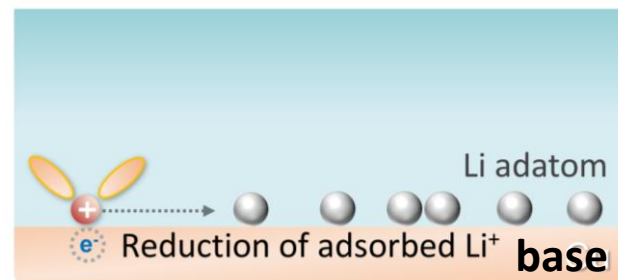
## Bulk diffusion in the electrolyte

long-range,  $10^2 \mu\text{m}$ ,  $D_{\text{elyte}}$



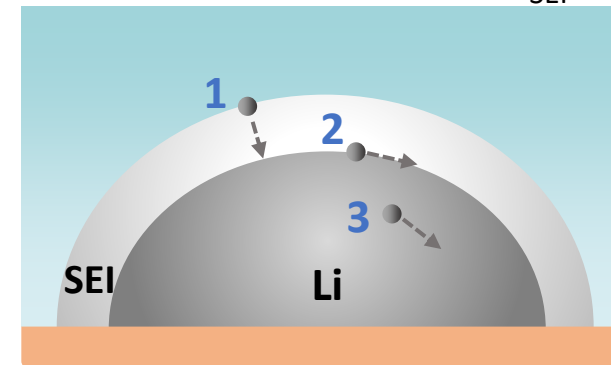
## Reaction

de-solvation, electron transfer,  $J_0$



## 1. transport through SEI

short-range, 10 nm,  $D_{\text{SEI}}$



## 2. Interface diffusion

short-range, nm,  $D_{\text{SEI-Li}}$

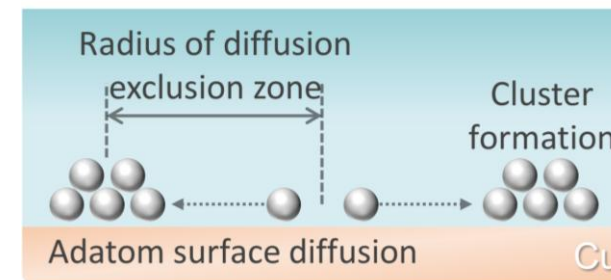
## 3. bulk diffusion in Li

short-range, nm,  $D_{\text{Li}}$

**growth**

## Surface diffusion

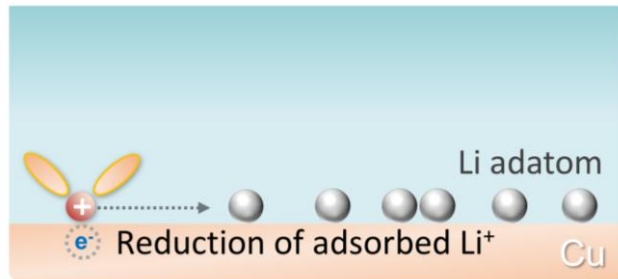
Short-range, nm,  $D_{\text{elyte-cu}}$



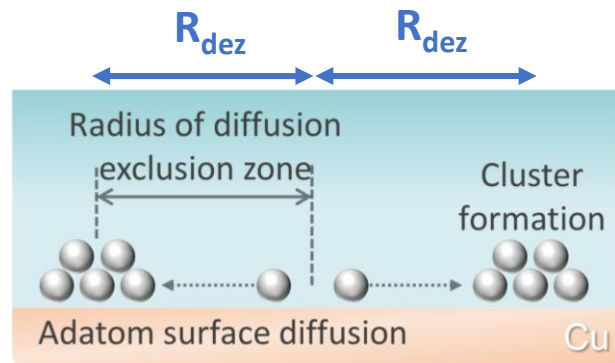
**nucleation**

# Nucleation: theory

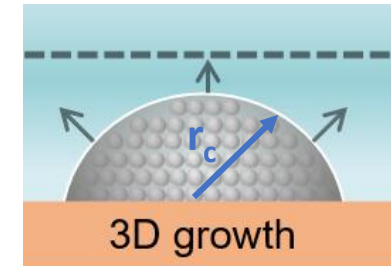
## Classical nucleation theory\*



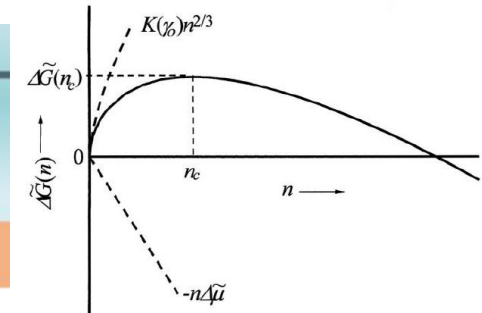
Reduction of  $\text{Li}^+$  to form adatom



- Adatom surface diffusion
- Cluster formation



Stable nuclei forms once passing  $r_c$



## Thermodynamics

$$\text{Li}^+ + e^- \rightarrow \text{Li}^0$$

$$r^3[(\mu_{\text{Li}^+} + \mu_{e^-}) - \mu_{\text{Li}^0}] = r^2\sigma + \text{strain}$$

driving force      barrier

Larger overpotential  
→ larger driving force  
→ more nuclei

$$N_{\text{crit}} = \frac{8BV_m^2\sigma^3}{27(zel\eta)^3}$$

## Mass transport

Higher  $D_{\text{elyte-cu}}$  → Large  $R_{\text{dez}}$  → less nuclei

$D_{\text{SEI-cu}}$  If considers SEI

\*Budevski, E. B., Staikov, G. T. and Lorenz, W. J. (1996) *Electrochemical phase formation and growth: an introduction to the initial stages of metal deposition*. John Wiley & Sons.

\*Gamburg, Y. D. and Zangari, G. (2011) *Theory and practice of metal electrodeposition*. Springer Science & Business Media.

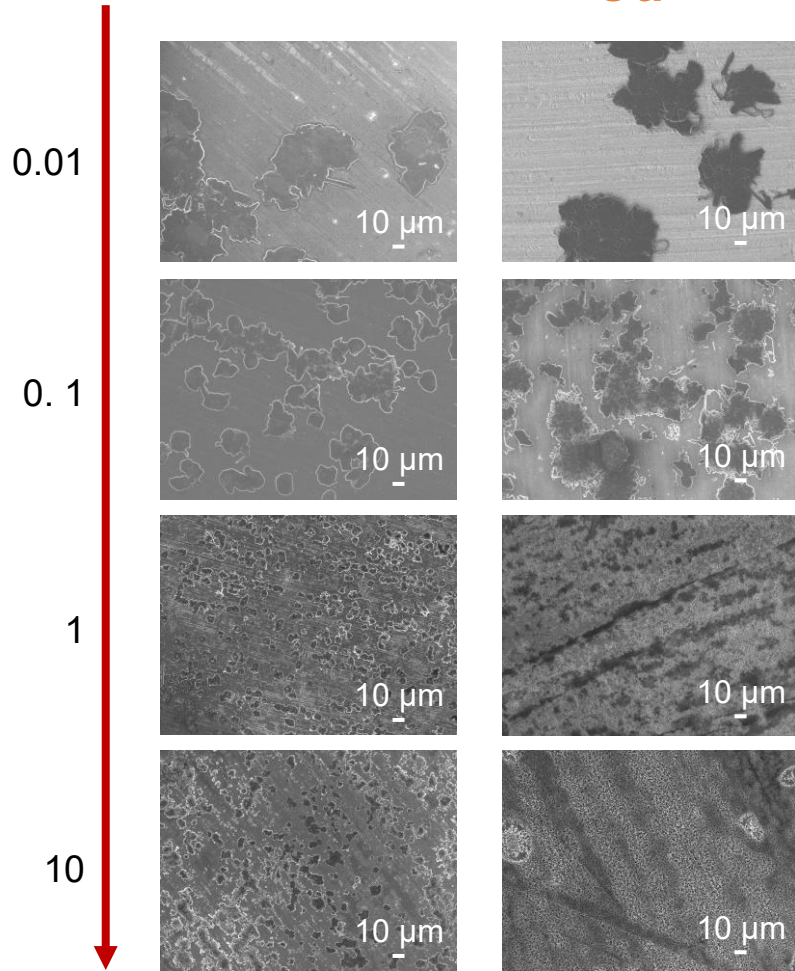
\*Milchev, A. (2002) *Electrocrystallization: fundamentals of nucleation and growth*. Springer Science & Business Media.

# Nucleation: dependence on current

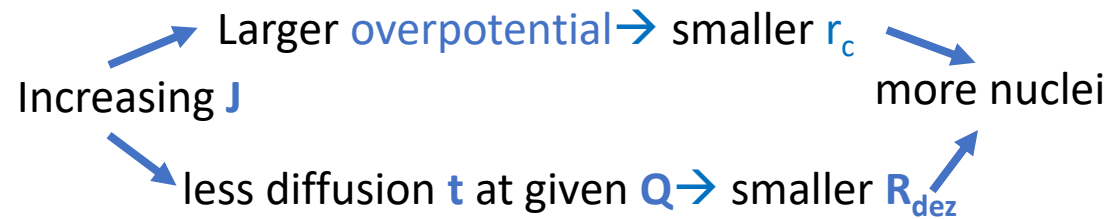
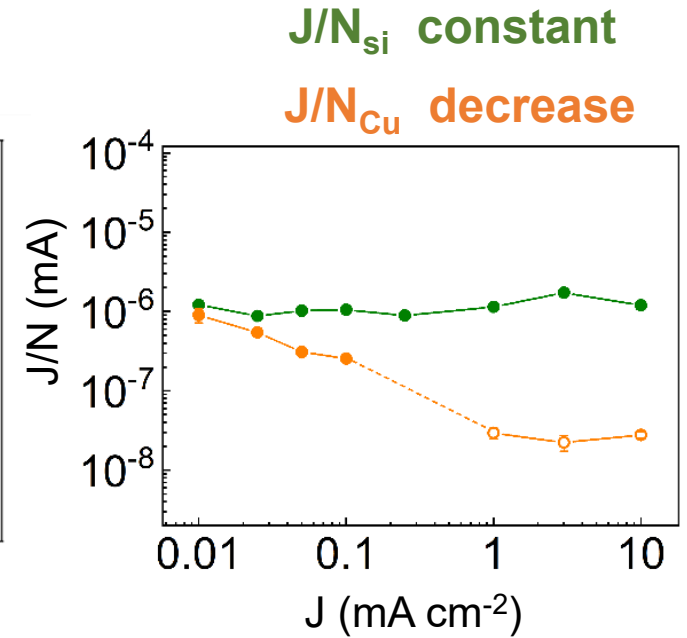
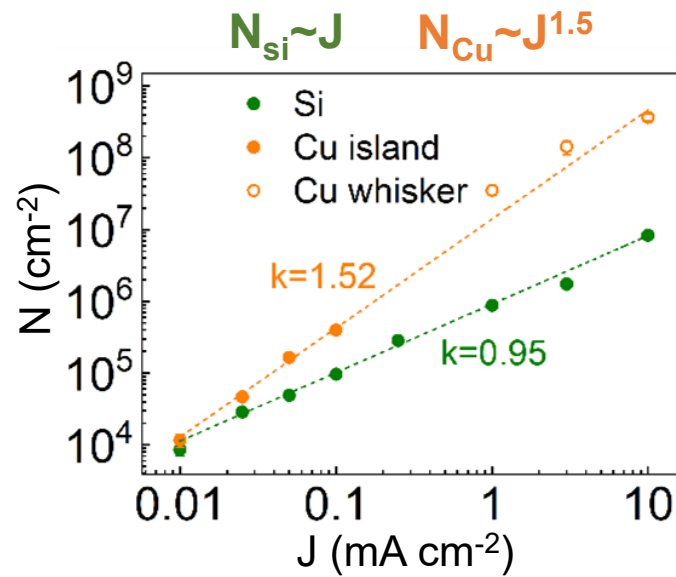
$Q=0.1 \text{ mAh/cm}^2$

Si

Cu

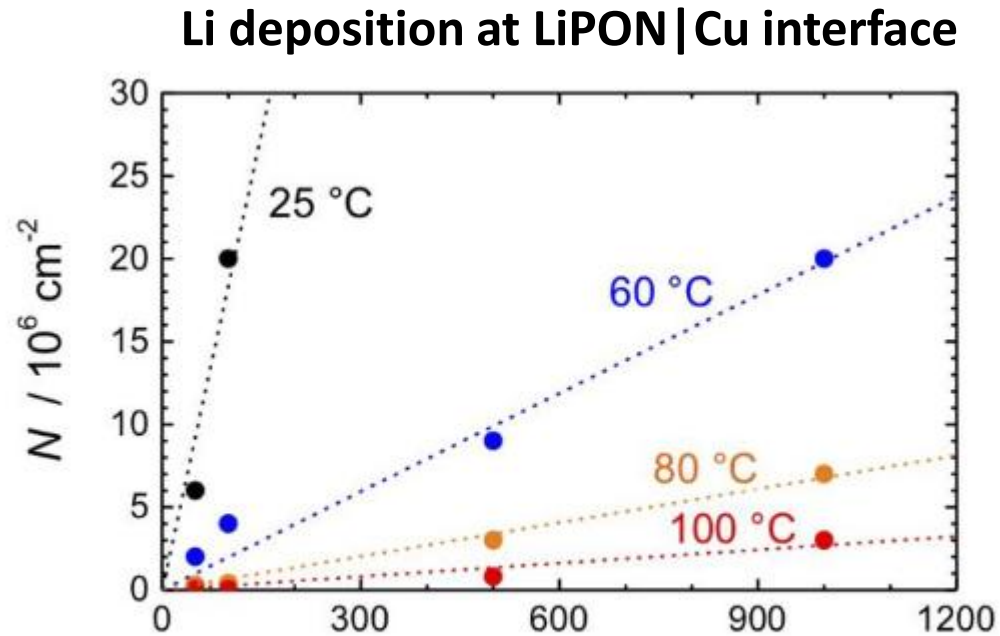


$J \text{ (mA cm}^{-2}\text{)}$



- **Si** responds to  $J$  by proportionally increasing  $N$ . Current per Li island is invariant at given  $T$
- for Si, nucleation is governed by surface diffusion:  $N \sim R_{\text{dez}}^{-2}$ ,  $R_{\text{dez}} \sim (Dt)^{0.5}$ ,  $t \sim J^{-1}$ , so  $N \sim D^{-1}J$
- For Cu, nucleation is not.

- Similar linear  $N \sim J$  correlation observed for Li deposition on li-wetting materials



**Liquid:**  $N=10^4 \text{ cm}^{-2}$  at  $J=0.025 \text{ mA cm}^{-2}$  and RT  
**Solid:**  $N=10^6 \text{ cm}^{-2}$  at similar condition

Surface diffusion in liquid electrolyte is easier than solid electrolyte

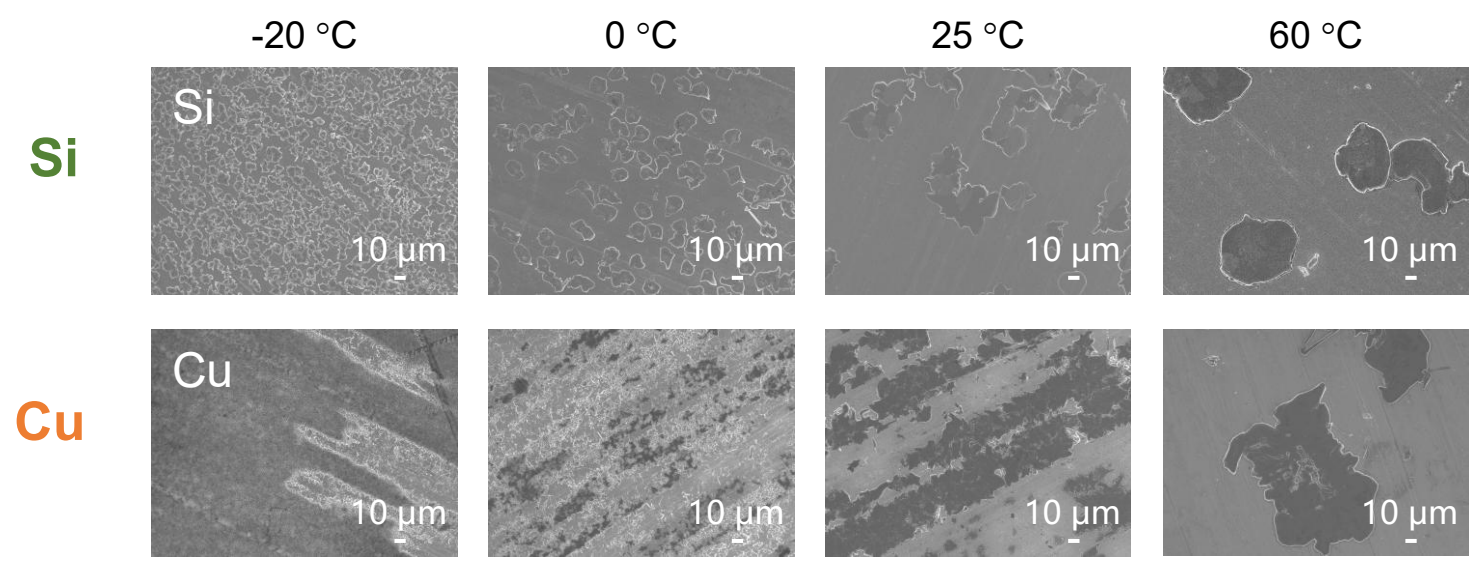
Current density /  $\mu\text{A cm}^{-2}$   
 Yamamoto, 2020, *ACS Applied Materials & Interfaces*, 207, 4894.

$$E=0.53 \text{ eV} > E_{s,cu} = 0.33 \text{ eV}$$

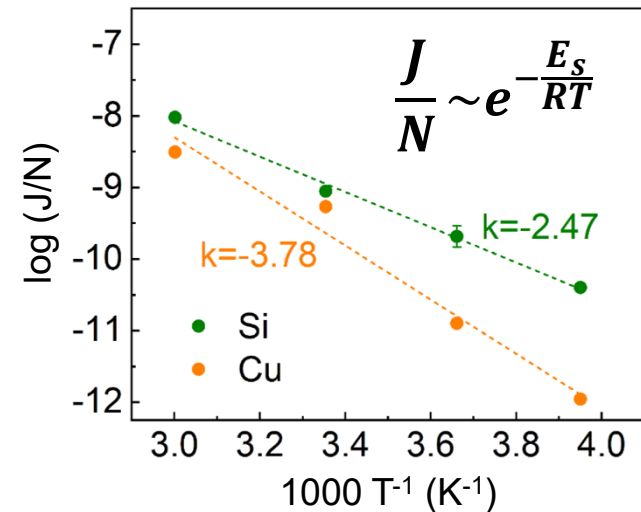


# Nucleation: dependence on temperature

Q=0.1 mAh/cm<sup>2</sup>, J=0.025 mA/cm<sup>2</sup>

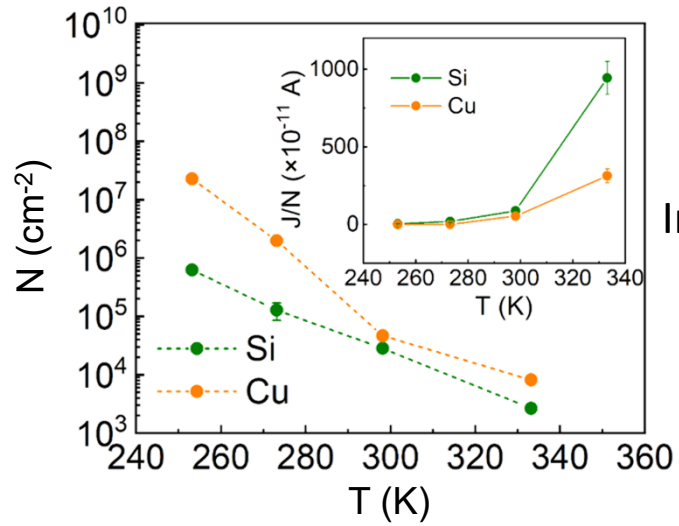


Arrhenius behavior



$E_{s,si} = 0.21 \text{ eV}$   
 $E_{s,cu} = 0.33 \text{ eV}$

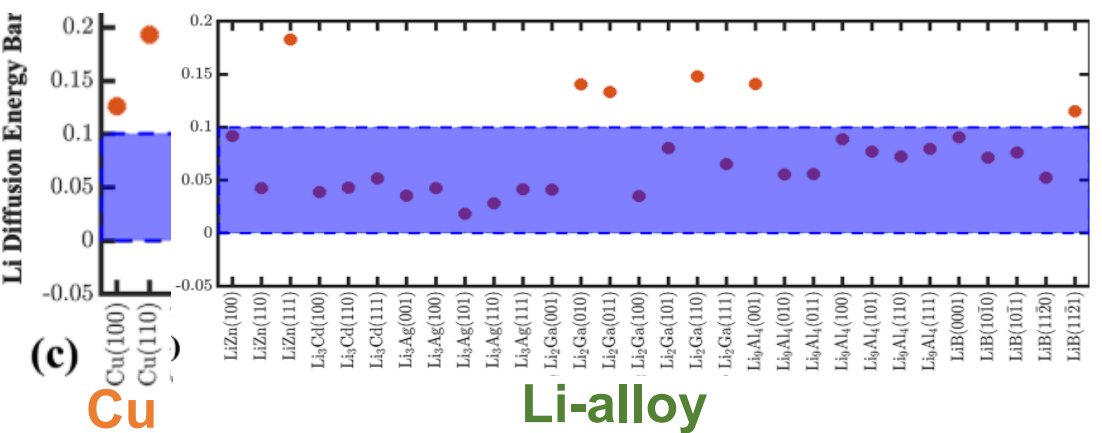
surface-diffusion-controlled



Increasing T

less overpotential → |

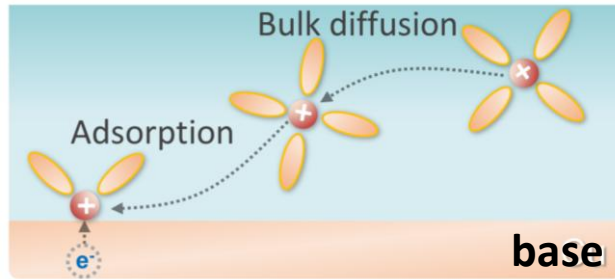
higher D<sub>elyte-cu</sub> → larger



# Which controls the growth of Li deposit?

## Bulk diffusion in the electrolyte

long-range,  $10^2 \mu\text{m}$ ,  $D_{\text{elyte}}$

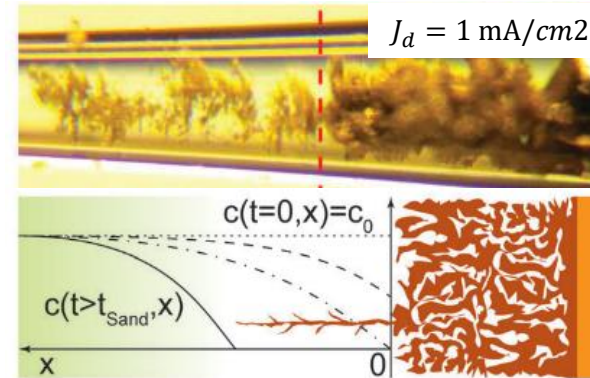


$$J_d = \frac{2cFD_{\text{elyte}}}{t_a L}$$

Brissot, et al. 1999, *Journal of Power Sources*

**unlikely the control step**

## electrolyte-diffusion-controlled



Bai, Bazant et al, *Energy Environ. Sci.* 2016

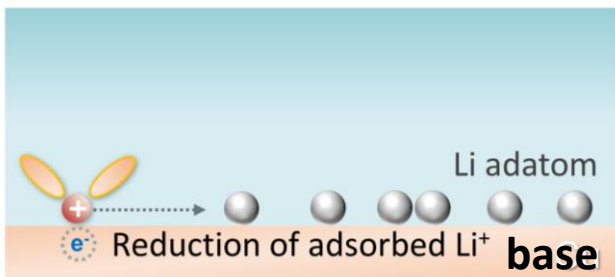
For Li metal batteries,  $L=25 \mu\text{m}$ , tortuosity=5

$$J_d = \frac{2cFD_{\text{eff}}}{t_a L} = 40 \text{ mA/cm}^2$$

For 5 mAh/cm<sup>2</sup> loading  
**40 mA/cm<sup>2</sup>  $\approx$  8C charging**

## Reaction

de-solvation, charge transfer,  $J_0$

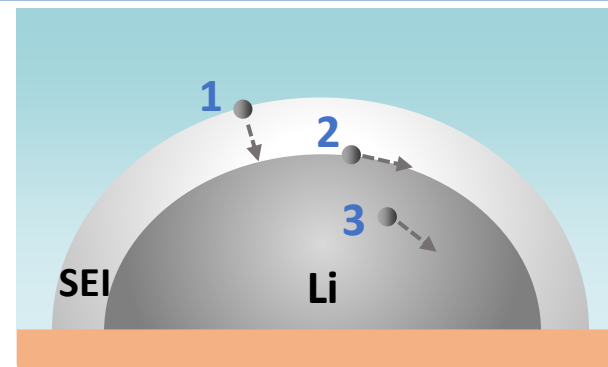


$$J_0 = \frac{RT}{FR_{ct}}$$

$$= 0.2 \text{ mA/cm}^2$$

Bard and Faulkner, 2001, *Electrochemical Methods Fundamentals and Applications*

**can be the control step**



**explicit boundary N/A**  
**unknown parameter or length scale**

**1. transport through SEI**  
short-range, nm,  $D_{\text{SEI}}$

**2. Interface diffusion**  
short-range, nm,  $D_{\text{SEI-Li}}$

**3. bulk diffusion in Li**  
short-range, nm,  $D_{\text{Li}}$

How Li deposit grows under **quasi-eq** ( $J \ll J_0 < J_d$ ) and **rxn-controlled** condition ( $J_0 < J < J_d$ )?

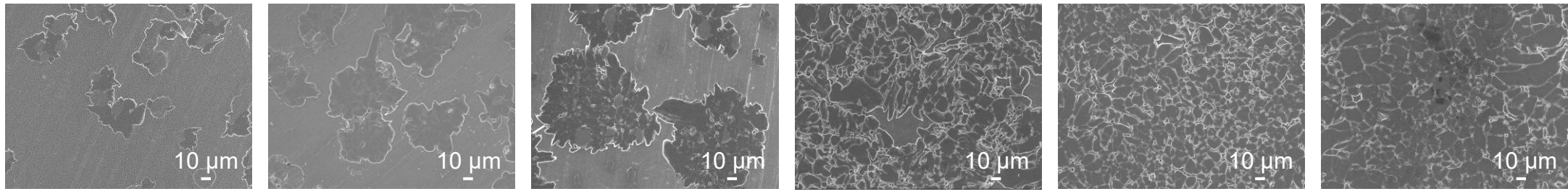


# Li deposition under quasi-equilibrium

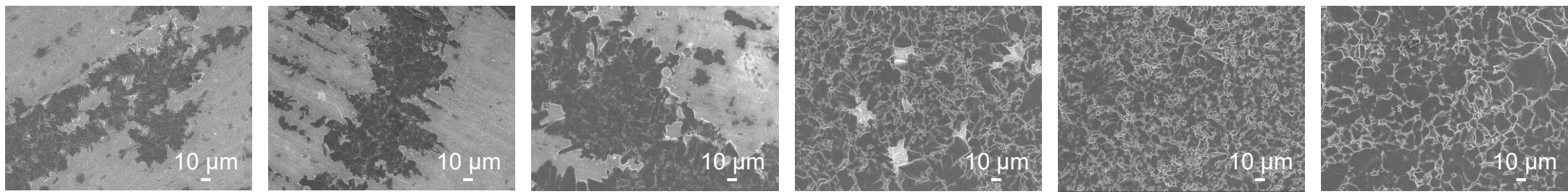
$J=0.025 \text{ mA/cm}^2$ ,  $J_d=40 \text{ mA/cm}^2$ ,  $J_0=0.2 \text{ mA/cm}^2$

0.1      0.2      0.4      0.8      1      4       $Q \text{ (mAh cm}^{-2}\text{)}$

Si



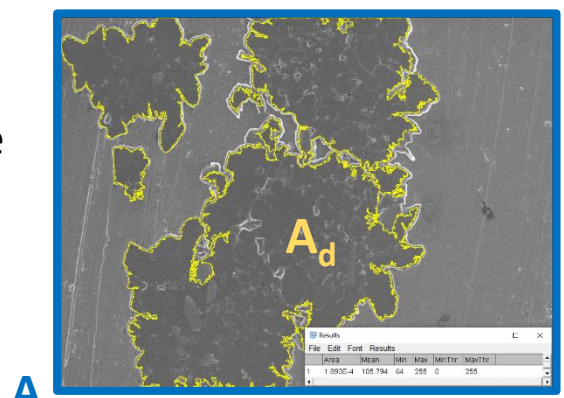
Cu



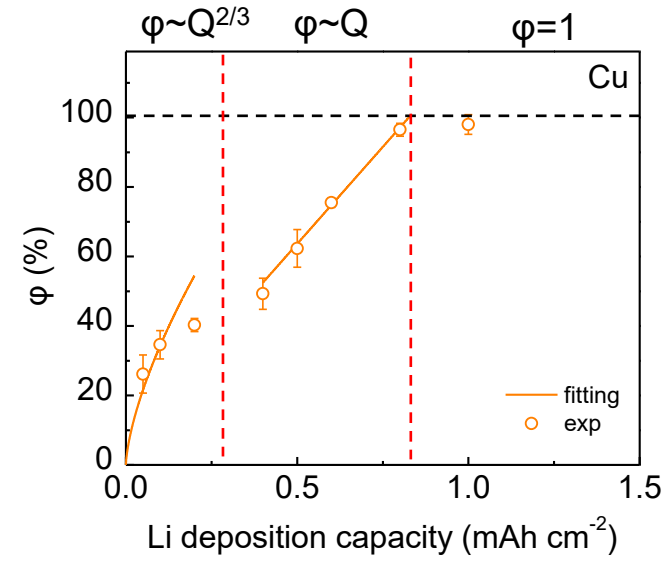
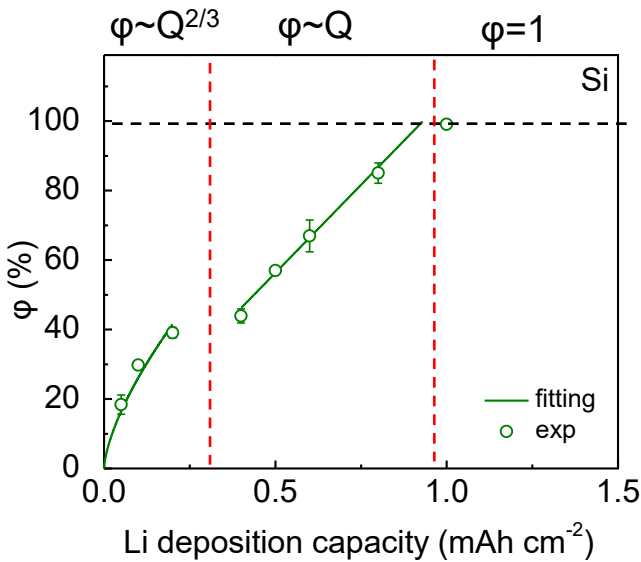
Computing in-plane area of deposits ( $A_d$ )

surface coverage

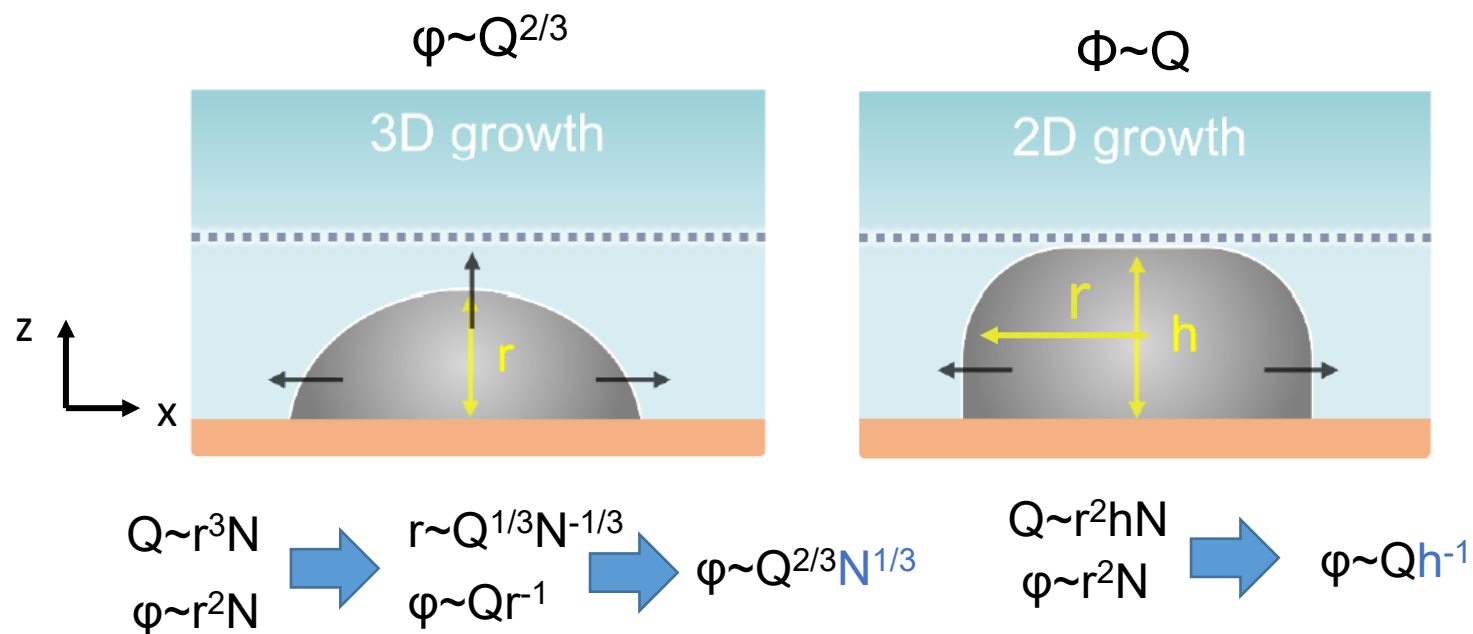
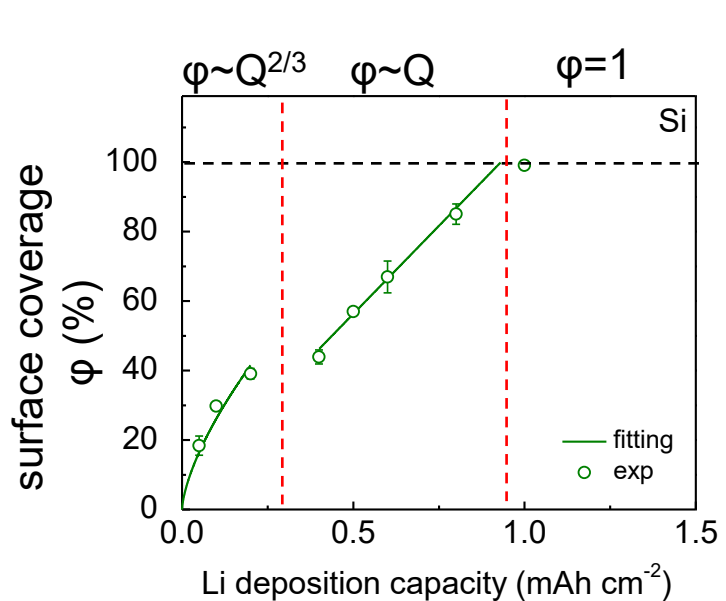
$$\phi = \frac{A_d}{A_s}$$



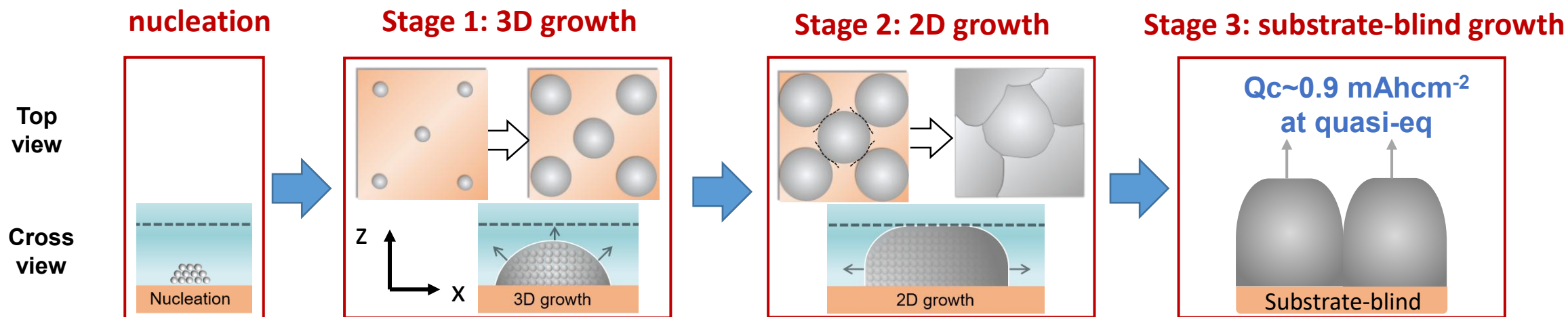
The area of the substrate ( $A_s$ )



# 3-stage growth for both Cu/Si under quasi-eq



$\phi$ : surface coverage,  $r$ : radius of the deposit,  $N$ : density of nuclei,  $h$ : height of the deposit



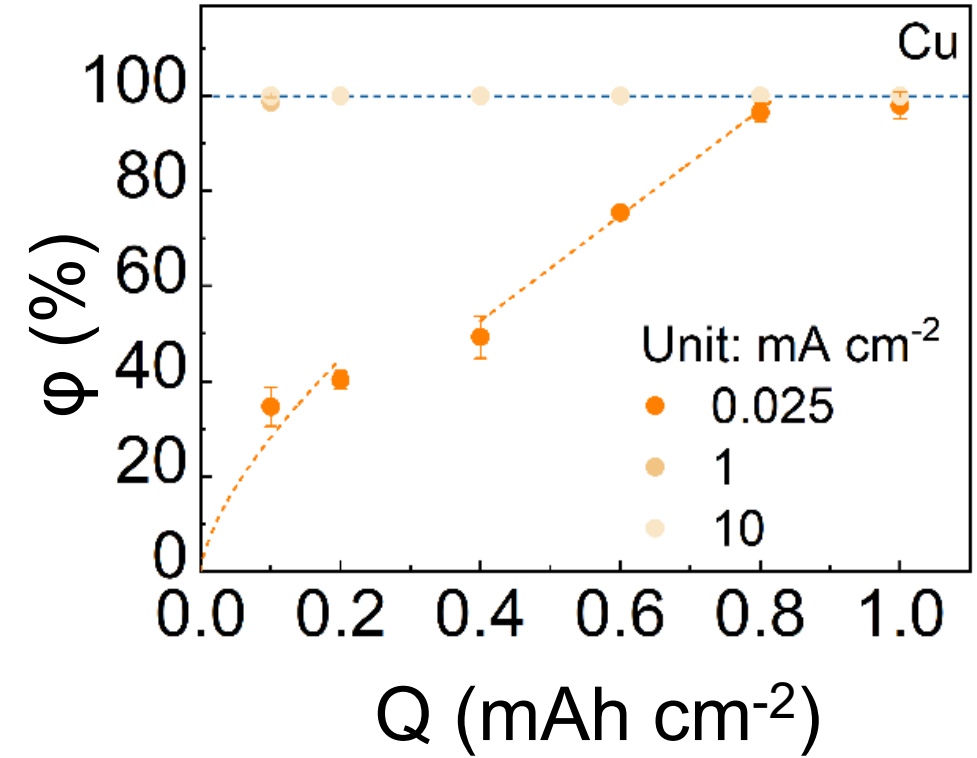
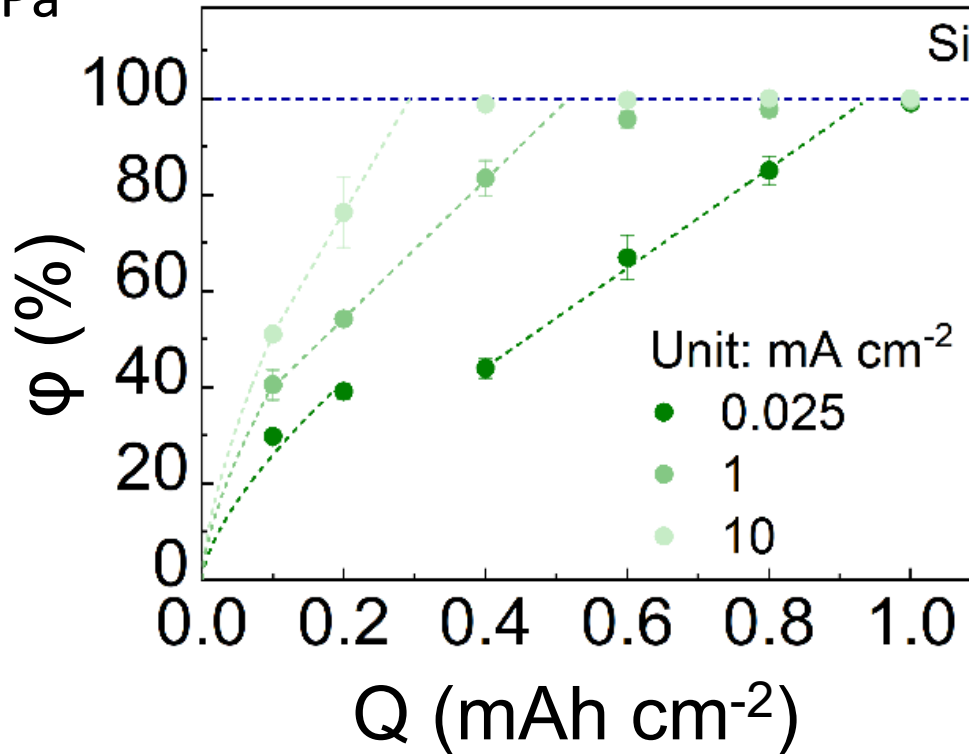
# 3-stage growth: current effect

Pressure=81.2 kPa

$J_d=40 \text{ mA/cm}^2$

$J_0=0.2 \text{ mA/cm}^2$

**Current:  
more nuclei**



**3-stage growth:**

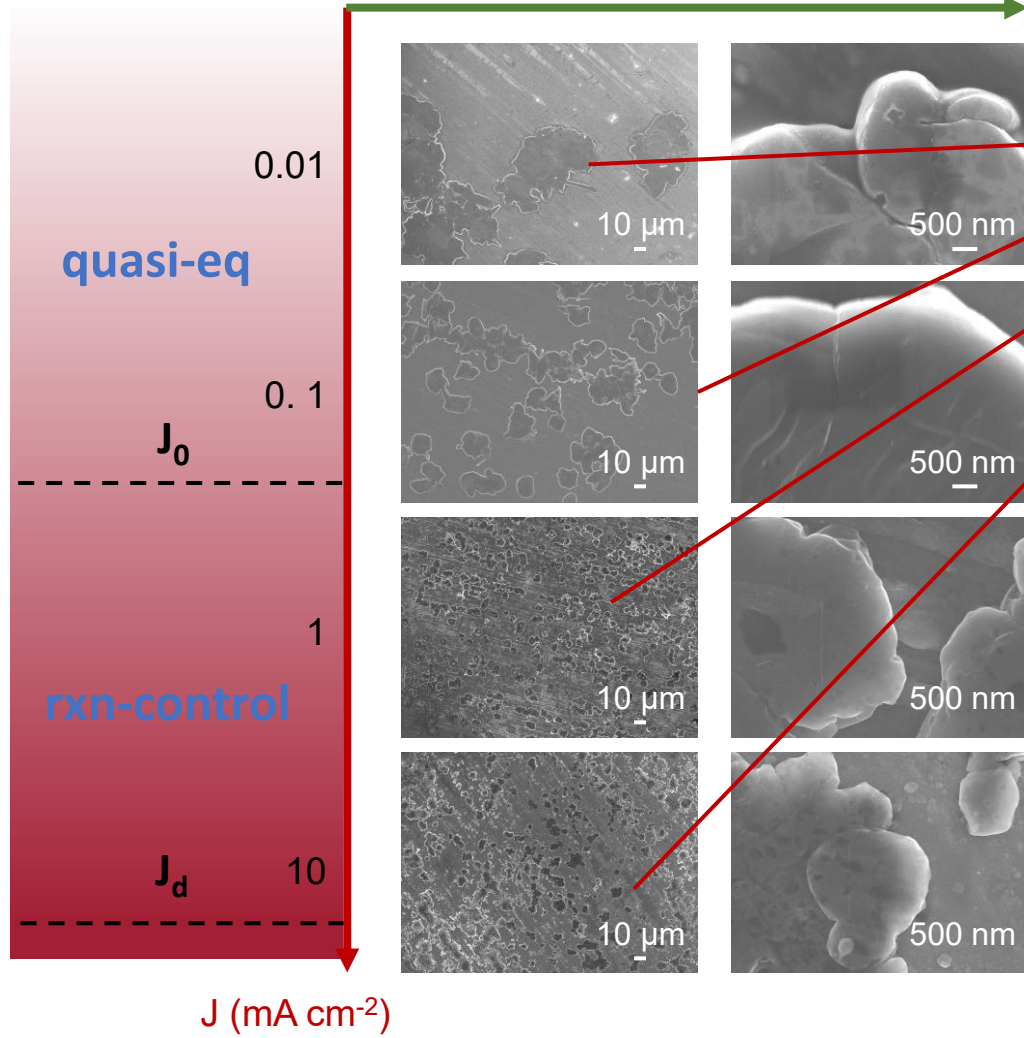
- Si at both **quasi-eq and reaction-controlled.**
- Cu **ONLY** at **quasi-eq**

# Why Cu differs from Si?

Q=0.1 mAh/cm<sup>2</sup>

Si

Zoom-in



Li deposits  
as **islands**

# Why Cu differs from Si?

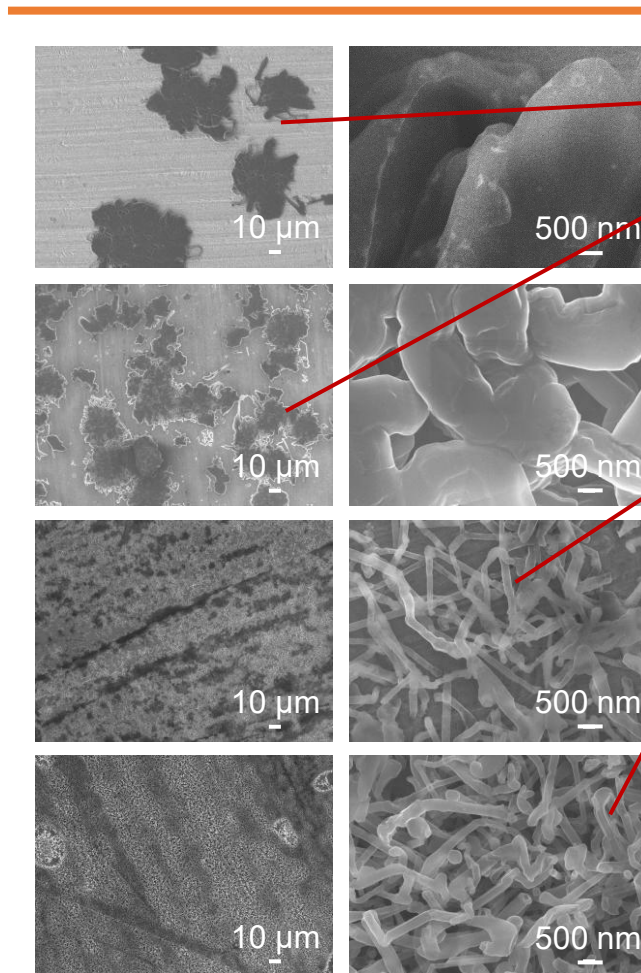
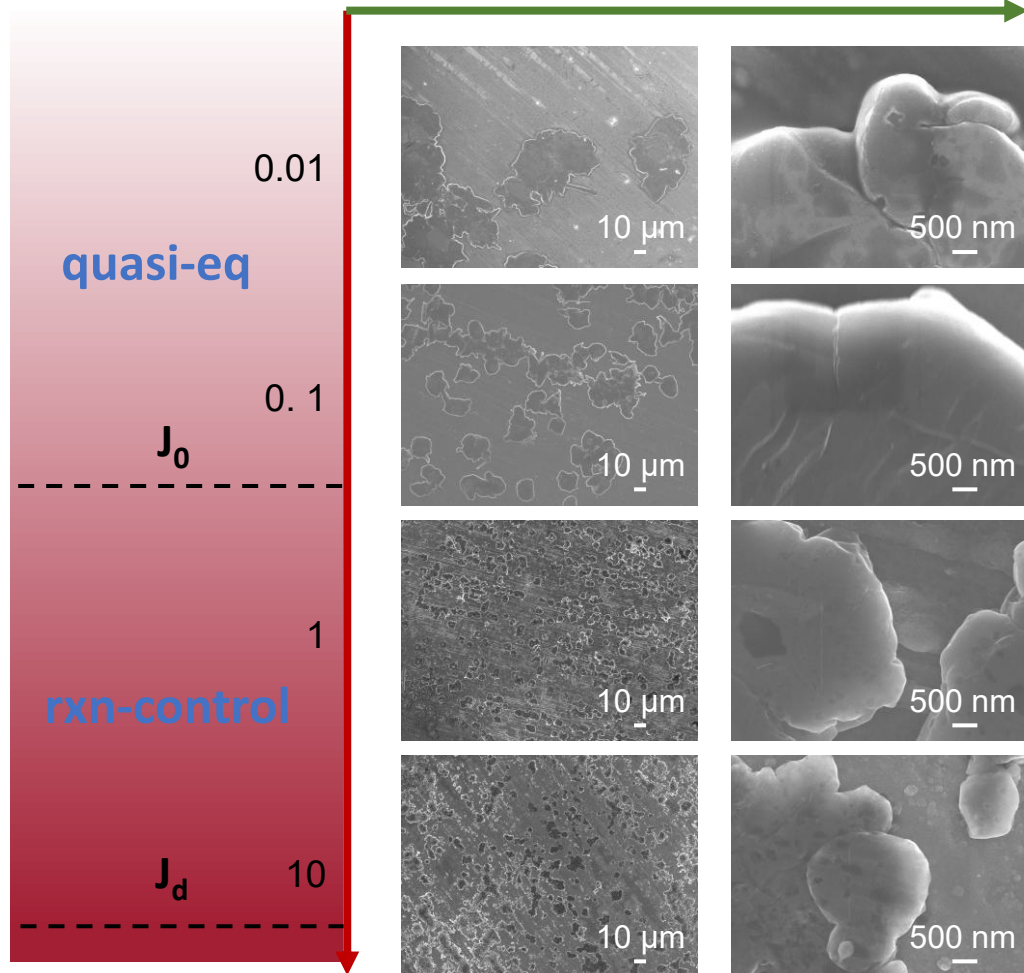
Q=0.1 mAh/cm<sup>2</sup>

Si

Zoom-in

Cu

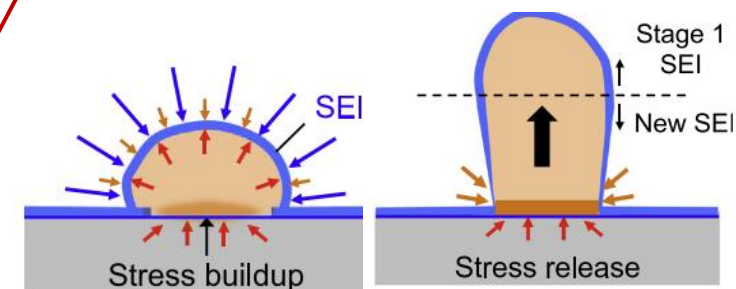
Zoom-in



Island with hair

Skinny needle-like deposit  
Invariant diameter in length direction

“Whisker”



Bazant, Li et al, *Nano Energy* 2017, 32, 271.

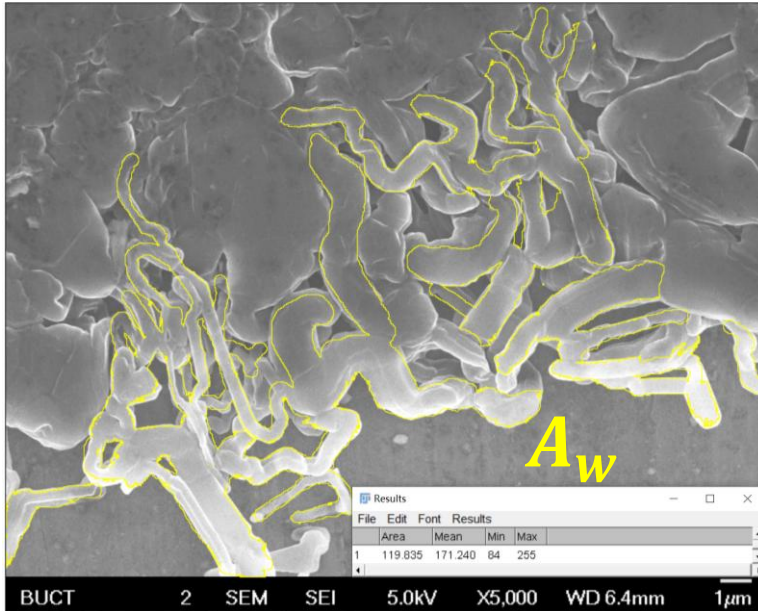
J (mA cm<sup>-2</sup>)

Li whisker tends to form on Cu at high current

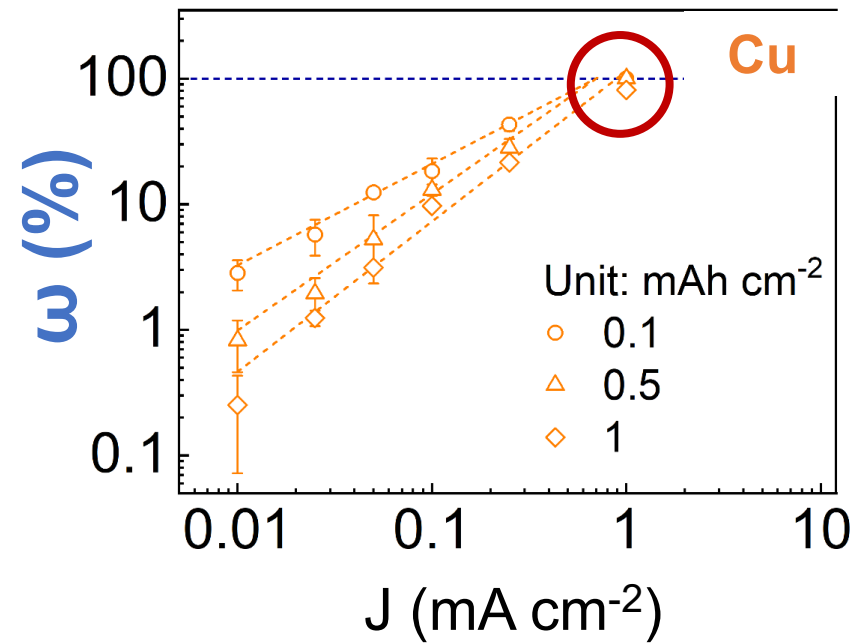
# How prone is Cu to form whiskers?

Whisker:

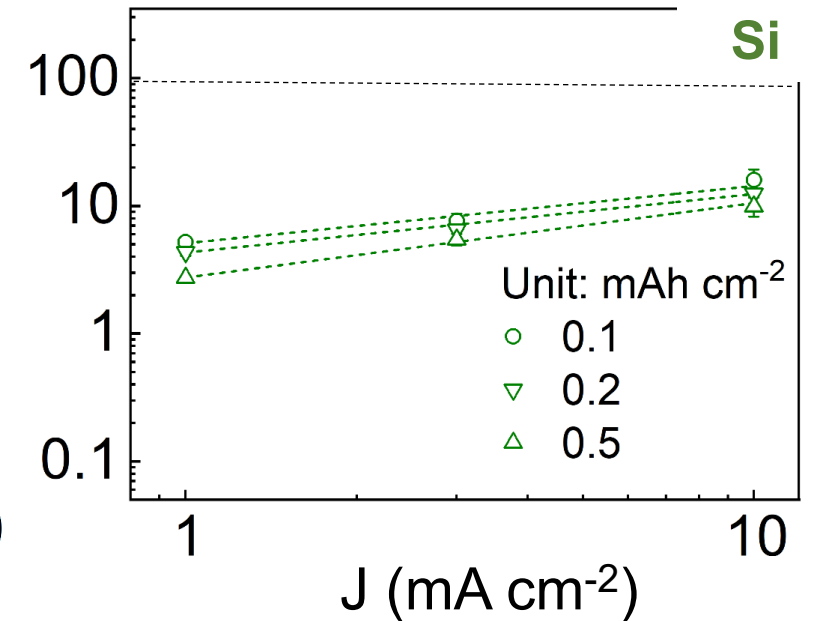
Li deposit with  $I_w/d_w \geq 5$



$J_{w,cu} = 0.79 \pm 0.1 \text{ mA/cm}^2$



$J_{w,si} \gg 10 \text{ mA/cm}^2$



Fraction of whisker

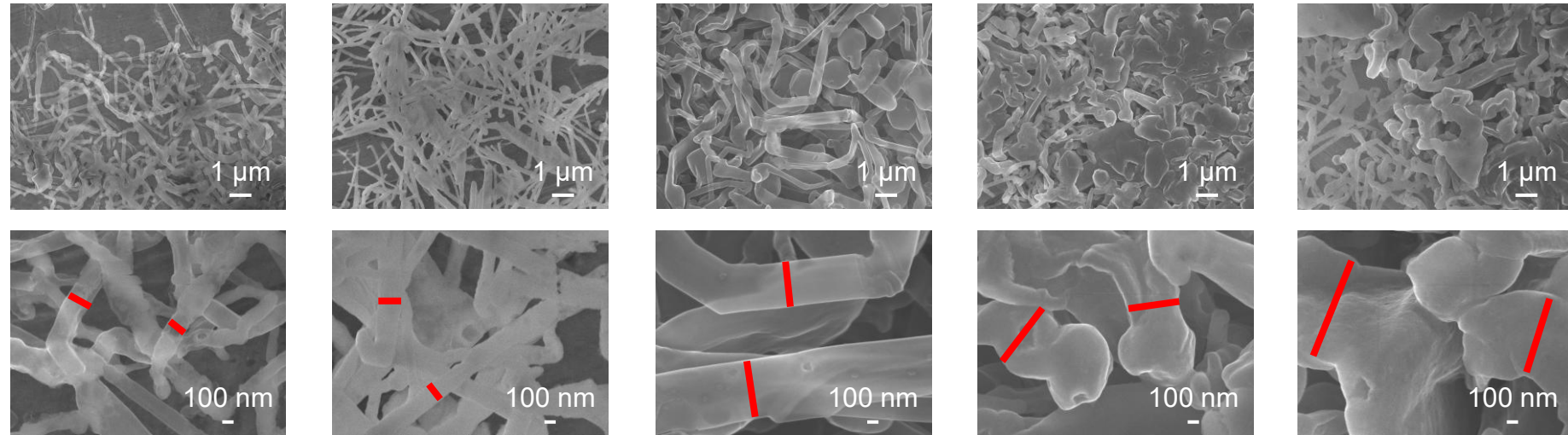
$$\omega = \frac{A_w}{A_d}$$

- $J_w$ : intrinsic property of electrolyte-substrate pair
- Can be used to measure how prone to form whisker

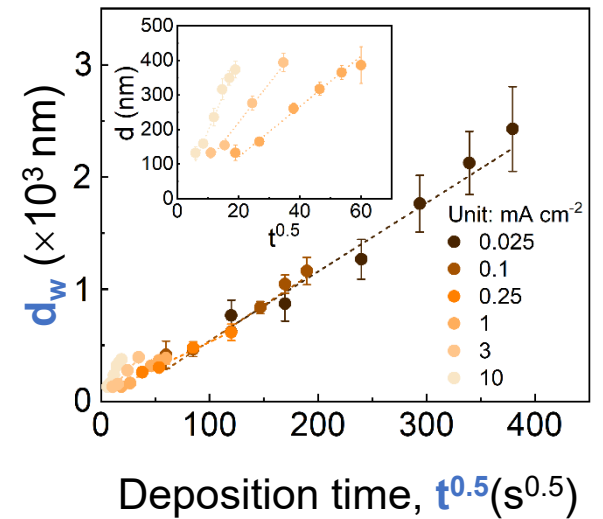
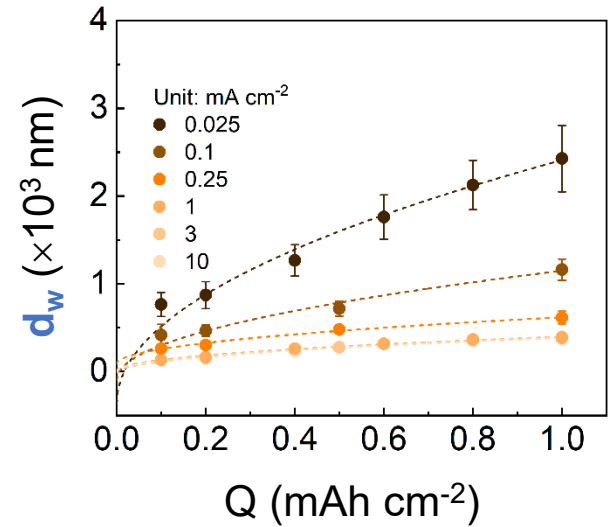
# What will happen to the formed whisker?

Cu

0.1                      0.2                      0.4                      0.6                      0.8                      Q (mAh cm<sup>-2</sup>)



J = 1 mA/cm<sup>2</sup>  
 > J<sub>w,cu</sub>

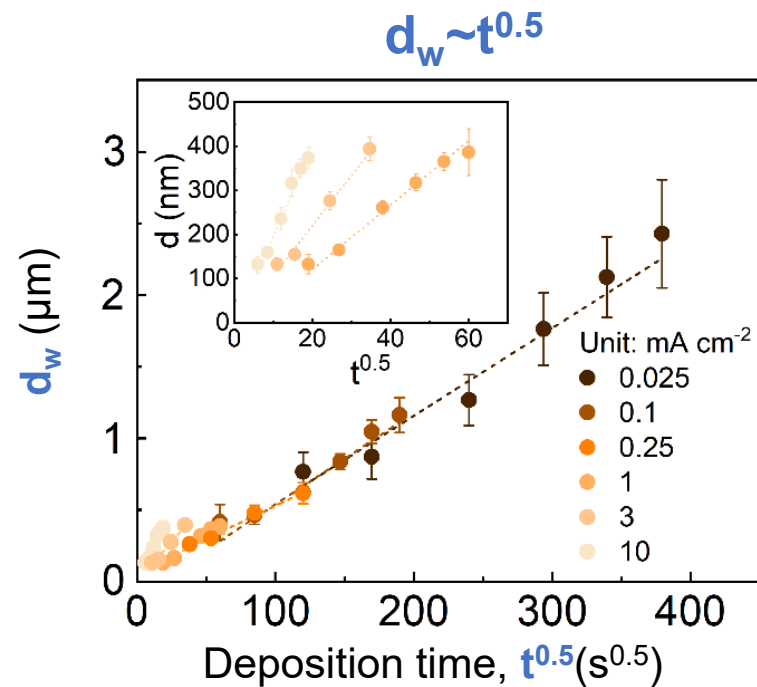


For  $J \leq 1 \text{ mA/cm}^2$ , all curves collapse on the same master curve

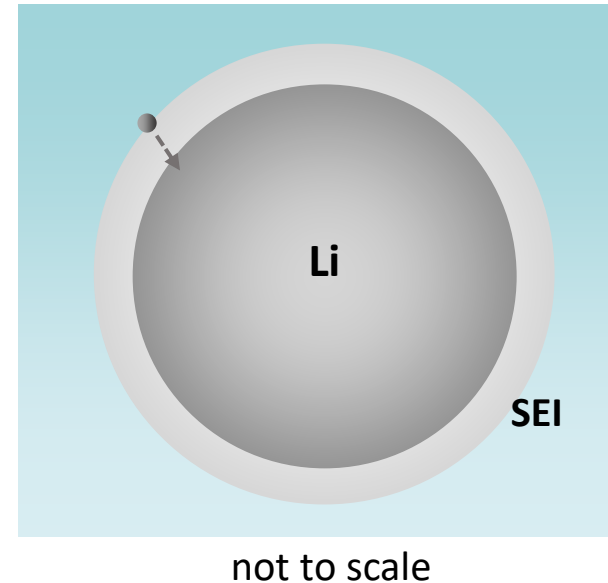
$$d_w \sim t^{0.5}$$

The growth of whisker in the radial direction is **diffusion-controlled**

# Diffusion of Li through SEI



Cross-section of whisker



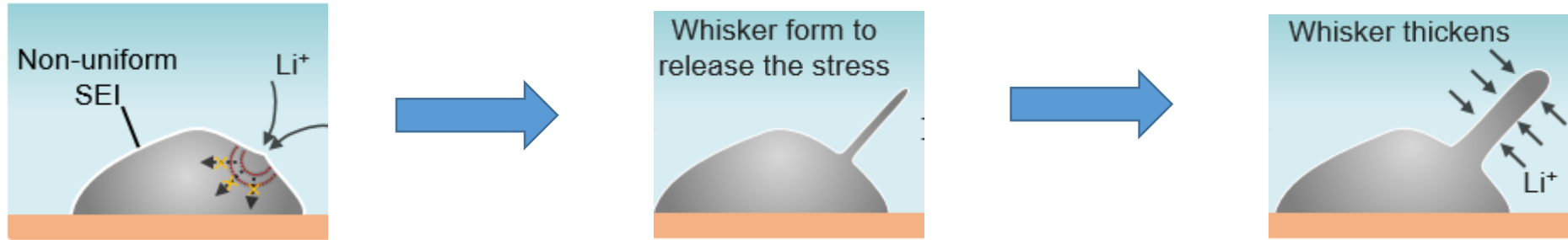
transport through SEI  
short-range, nm,  $D_{Li}^{SEI}$

Given SEI growth is limited by the diffusion of solvent molecule ( $D_{sol}^{SEI}$ )\*

$$d = k \frac{D_{Li^+}^{SEI}}{\sqrt{D_{sol}^{SEI}}} t^{0.5}$$

From the slope of the fitting,  $D_{Li}^{SEI}$  calculated to be  $\sim 10^{-12} \text{ cm}^2/\text{s}$

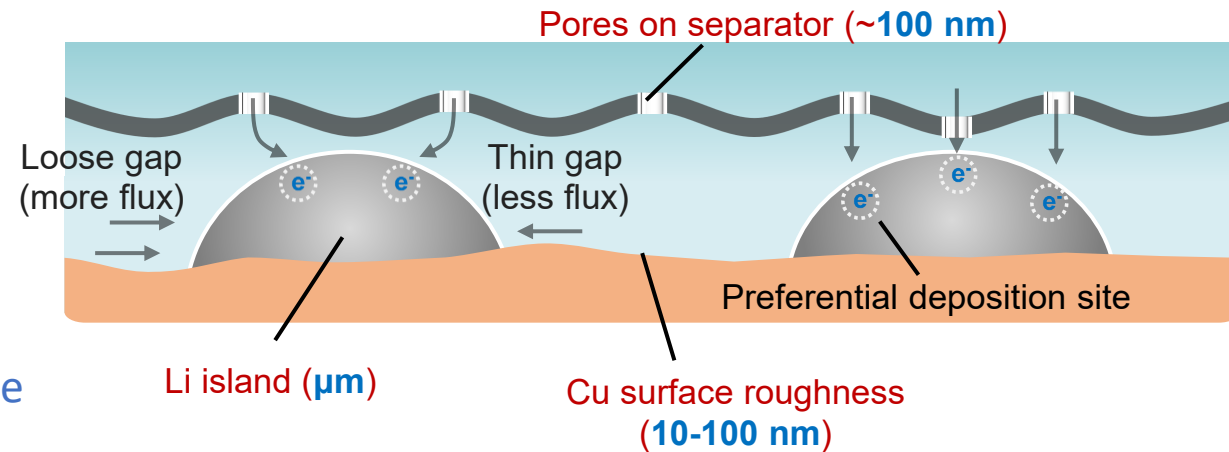
# Whisker: formation and growth



- Non-uniform deposition
- Stress accumulation
- Stress-release by cracking SEI
- Li shoots out by coble creep
- Whisker grows in radial direction (controlled by through-SEI diffusion)

## Why non-uniform deposition?

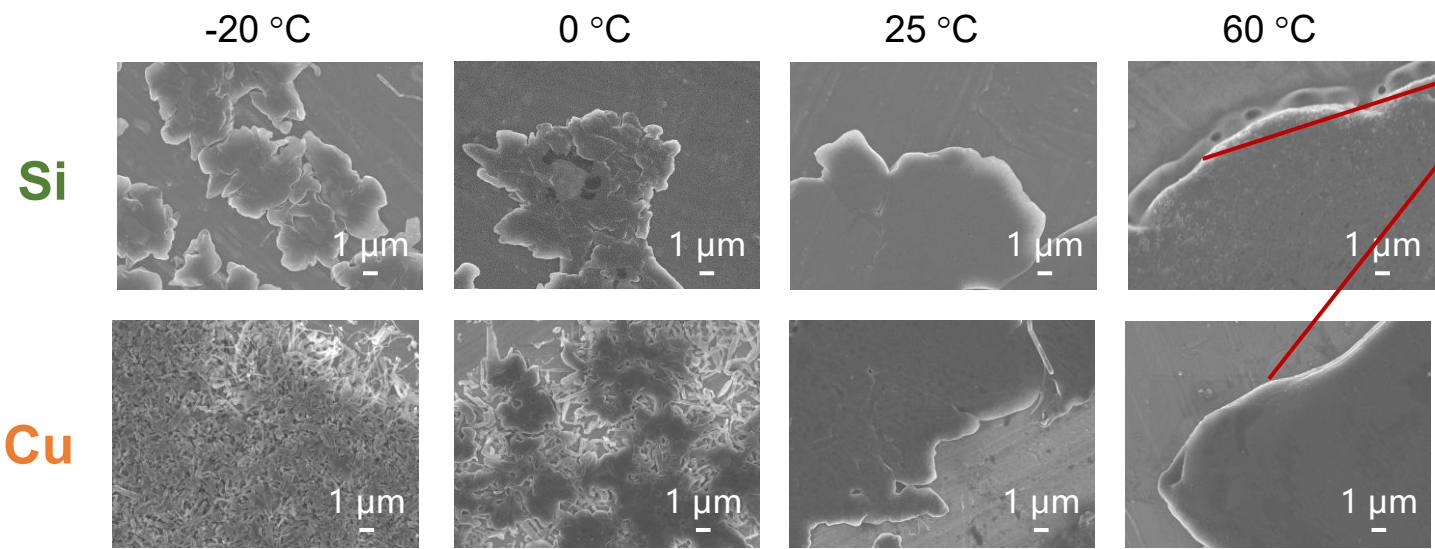
- Non-uniform SEI
  - Potential dependent decomposition of electrolyte
  - Surface defects/impurity on substrate
- Non-uniform local flux
  - Spatially heterogeneous separator/substrate interface



## Why Cu is more prone to whisker than Si?

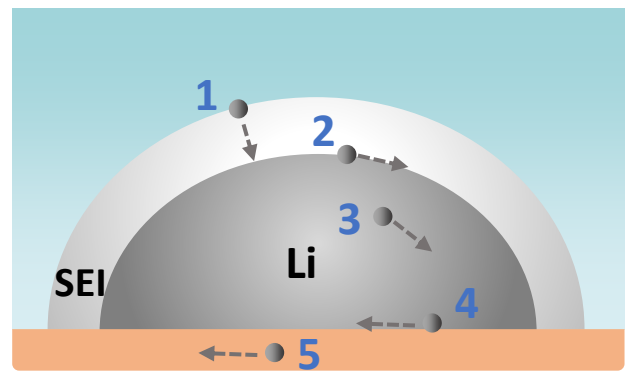
# Morphology of Li deposit at different T

Q=0.1 mAh/cm<sup>2</sup>, J=0.025 mA/cm<sup>2</sup> (quasi-eq condition).



The edges become **smoother** at **high T**

Thermodynamically,  
The deposit wants to **reduce  $P_d$**  to minimize **surface energy**



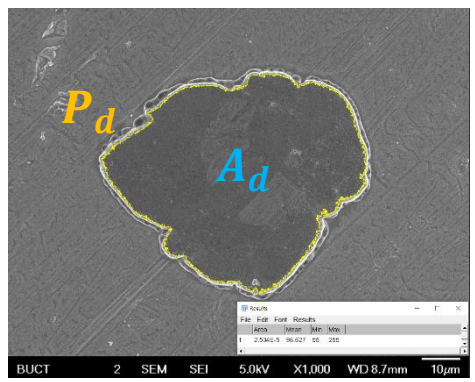
- 2. Interface diffusion ( $D_{SEI-Li}$ )
- 3. bulk diffusion in Li ( $D_{Li}$ )
- 4. Interface diffusion ( $D_{Li-substrate}$ )
- 5. bulk diffusion in substrate ( $D_{substrate}$ )

**Diffusion in  $LiSi_x$  alloy and along  $Li-LiSi_x$  interface reduces whisker tendency**

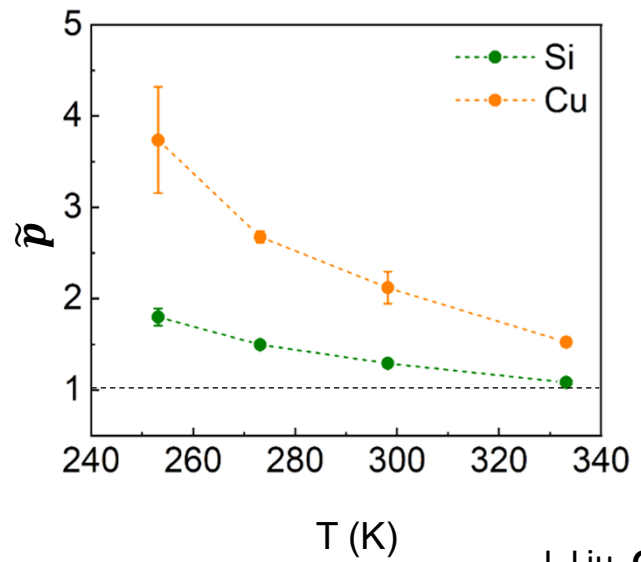
dimensionless perimeter

$$\tilde{p} = \frac{P_d}{\sqrt{4\pi A_d}}$$

=1 for circle



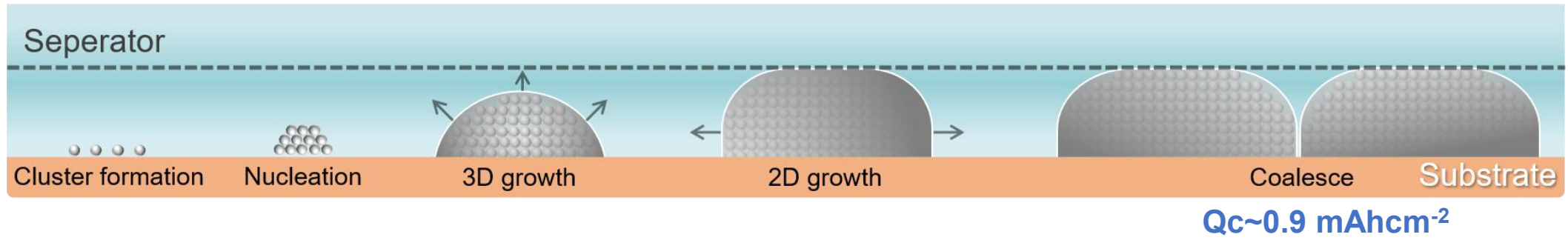
Li deforms faster when on Si



# Summary of Li deposition mechanism

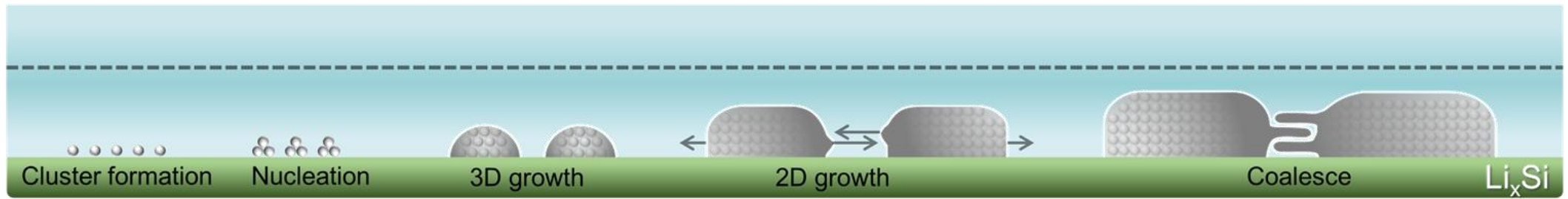
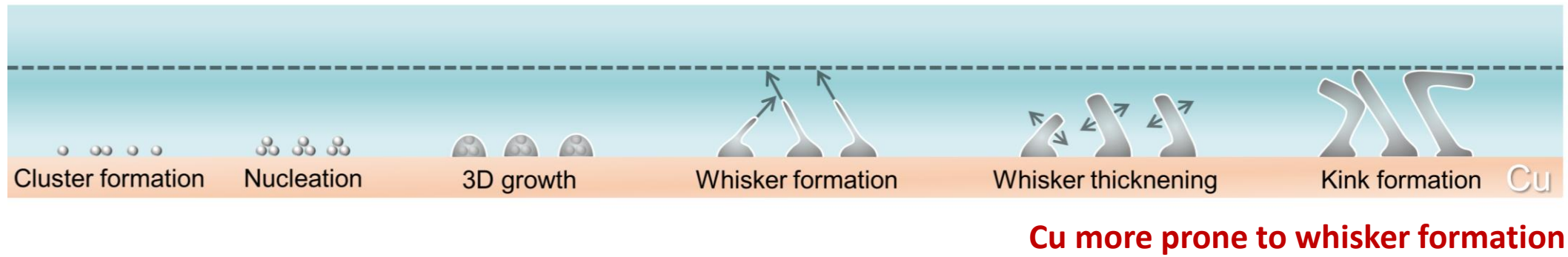
quasi-eq

$$J \ll J_0 \ll J_d$$



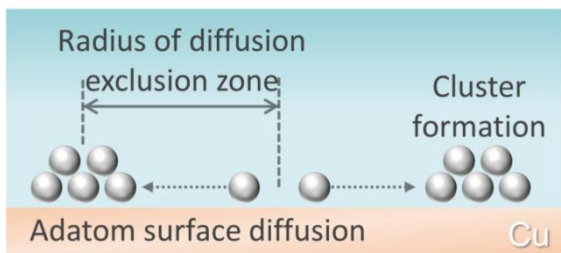
rxn-control

$$J_0 < J < J_d$$

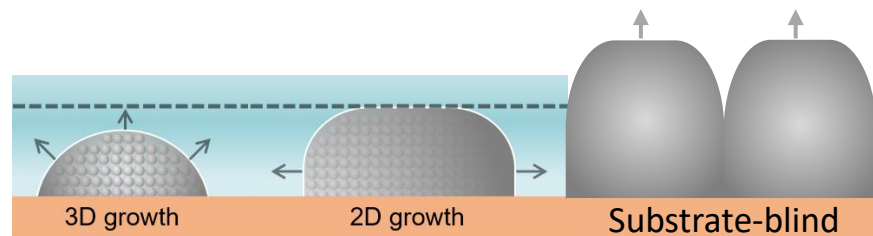


# More quantitatively...

## Nucleation



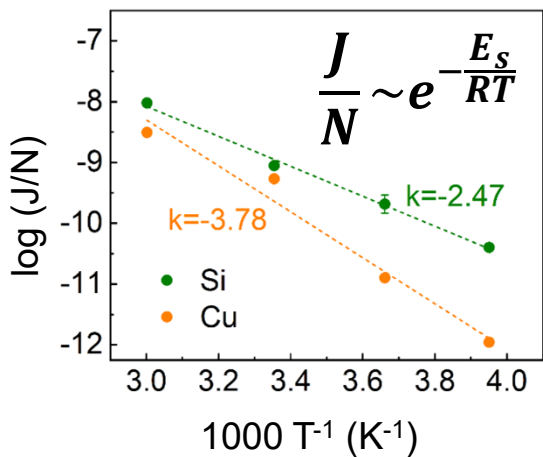
## Growth



## Whisker formation and growth



### Surface diffusion controlled

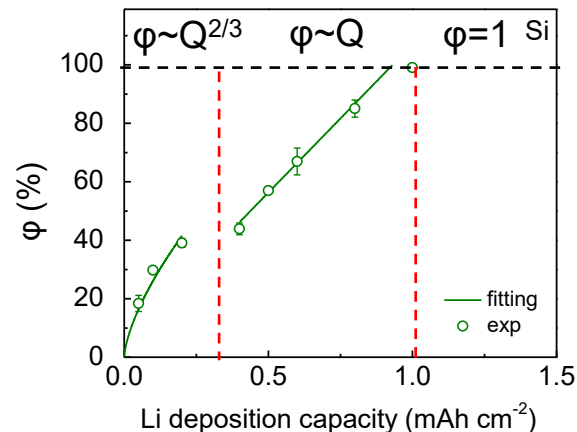


### surface diffusion

$E_{s,si} = 0.21 \text{ eV}$

$E_{s,cu} = 0.33 \text{ eV}$

### 3-stage

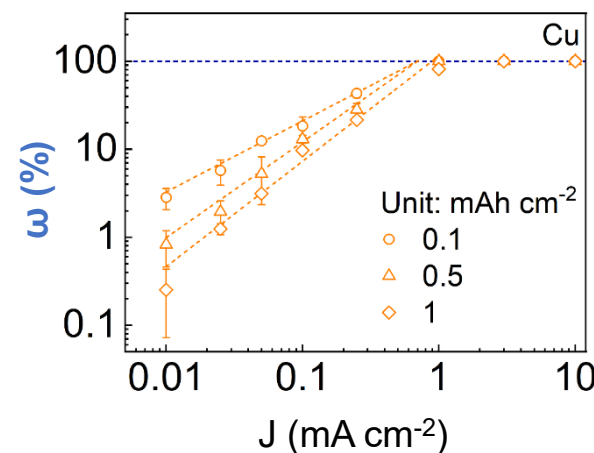


$Q_c \sim 0.9 \text{ mAh cm}^{-2}$  at quasi-eq

0.5 at J=1 mA/cm<sup>2</sup>

0.3 at J=10

### J<sub>w</sub>: the tendency to form whisker

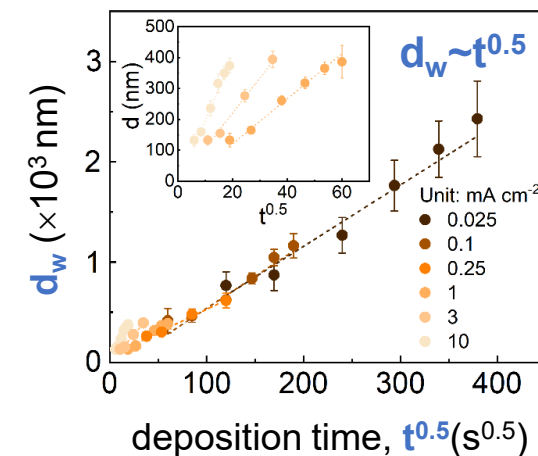


$J_{w,cu} = 0.79 \text{ mA/cm}^2$

$J_{w,si} \gg 10 \text{ mA/cm}^2$

Diffusion in LiSi<sub>x</sub>  
suppress whisker

### Through-SEI diffusion controlled



### transport through SEI

$D_{Li}^{SEI} : 10^{-12} \text{ cm}^2/\text{s}$



# Acknowledgement



Collaborator:  
Jing Niu (BCTU)  
Feng Wang (BCTU)

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🐦 [@TaoGao2020](https://twitter.com/TaoGao2020)



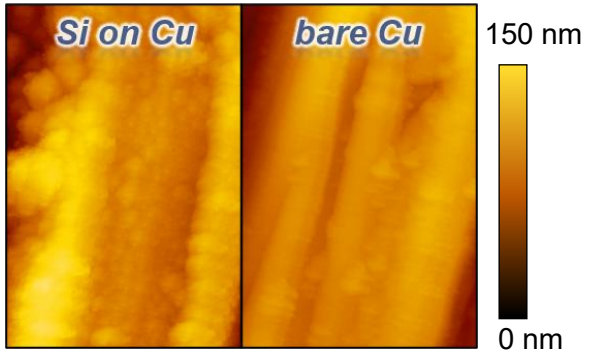
# The challenge of improving CE

Year	Composition	Substrate	CE	Current density (mA/cm <sup>2</sup> )	Capacity (mAh/cm <sup>2</sup> )
2015	4M LiFSi-DME	Cu	99.1%	0.2	1
2018	10M LiFSi-DMC	Cu	99.2%	0.2	1
2018	1.2M LiFSi/DMC-BTFE	Cu	99.3%	0.5	5
2018	1 M LiPF <sub>6</sub> in FEC/FEMC/HFE	Cu	99.2%	0.1	1
2019	1M LiPF <sub>6</sub> carbonate	Ammonia treated carbon film		0.8	
2019	LiFSi-1.2DME-3TTE	Cu	99.3%	0.5	1
2019	1 M LiFSi/DME-TFEO	Cu	97.74%	0.5	5
2020	1M LiFSi/FDMB	Cu	99.3%	0.5	1
2020	LiPF <sub>6</sub> /EC-DEC-LiNO <sub>3</sub> -Sn(OTf) <sub>2</sub>	Cu	98.4%	1	1
2021	1M LiFSi-DMTMSA	Cu	99%	0.5	1

- **Electrolyte design** has increased CE up to **99.3%** at  $J \leq 1 \text{ mA/cm}^2$
- **80% capacity** at **1000th cycle**, CE **>99.98%**
- **Fundamental knowledge** required to guide the design of electrolyte and substrate

J.-G. Zhang, et al. *Nat. Commun.* **2015**, 6, 6362  
 J. G. Zhang, W. Xu, et al. *Joule* **2019**, 3, 1662.  
 J. G. Zhang, et al. *Nat. Energy* **2019**, 4, 796.  
 Y. Cui, Z. Bao, et al. *Nat. Energy* **2020**, 5, 526.  
 Y. Lu, et al. *Adv. Mater.* **2020**, 32, 1.

J. Li, et al. *Nat. Energy* **2021**, 6.  
 K. Xu, C. Wang, et al. *Nat. Nanotechnol.* **2018**, 13, 1000.  
 C. Wang, et al. *Chem* **2018**, 4, 174  
 J. G. Zhang, et al. *Adv. Mater.* **2018**, 30, 1.  
 J. Liu, et al. *Nat. Nanotechnol.* **2019**, 14, 594

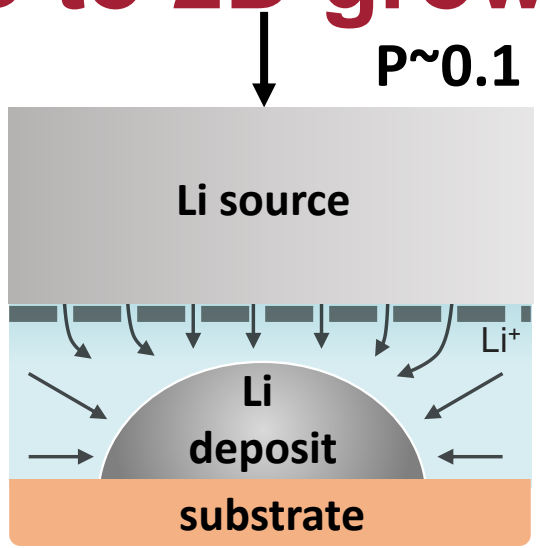


Roughness 10-100 nm



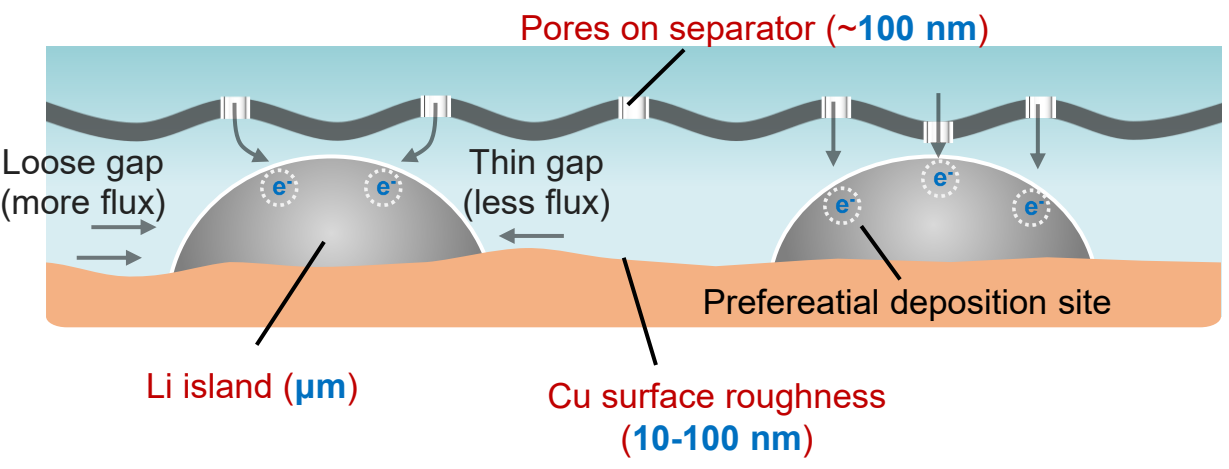
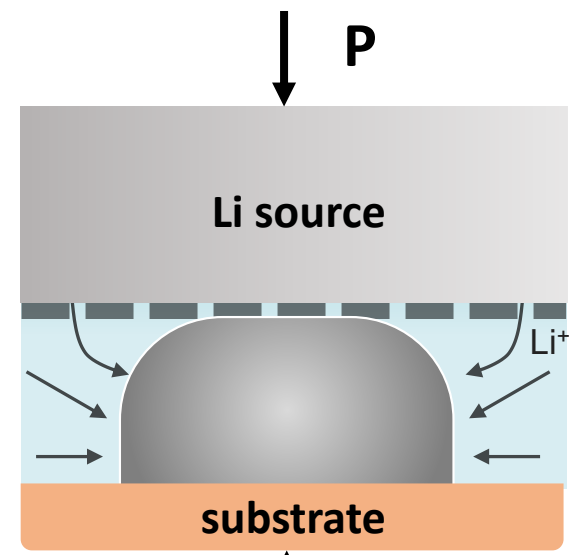
# Why 3D to 2D growth?

3D growth



Possibly due to constrained z-direction transport

2D growth

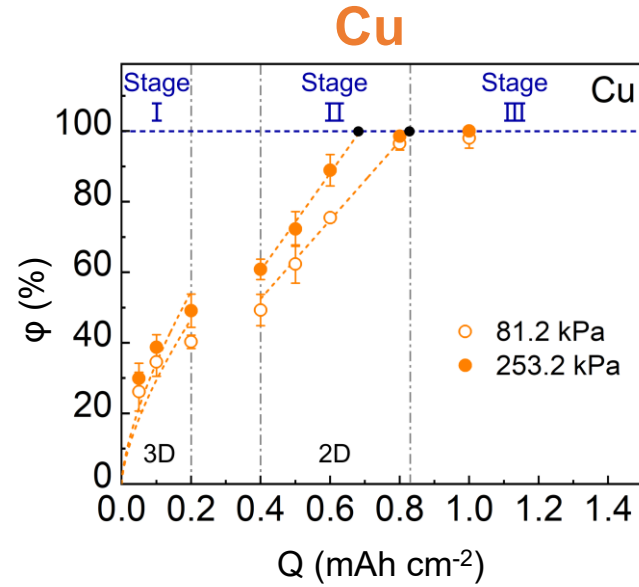
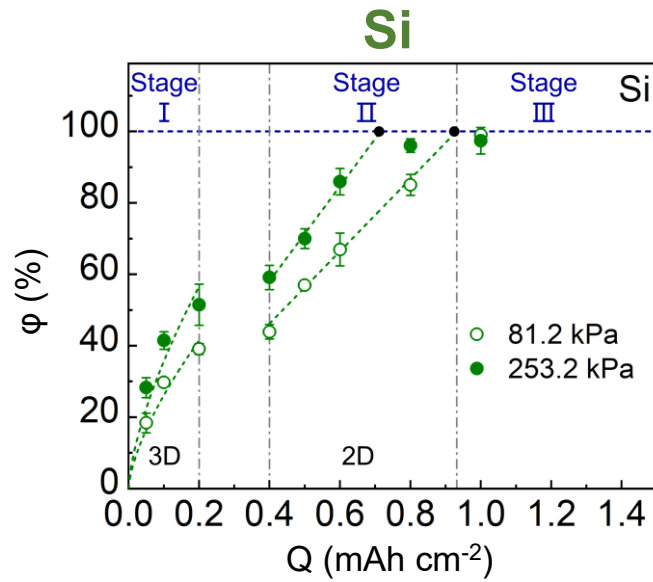


- The deposition occurs under quasi-equilibrium condition ( $J \ll J_0 < J_d$ ), so the effect of Li transport in the electrolyte and reaction heterogeneity are negligible
- due to the surface roughness of the substrate and the separator, there is some small voids between the separator and the substrate despite of the pressure. These voids should be where nucleation occurs due to smaller nucleation barrier (no stain energy from the separator)
- Once the Li island grows to certain height, the stain from the separator makes the z-direction growth thermodynamically difficult. Growing into the nano-pores of the separator is also thermodynamically unfavorable due to the large surface energy<sub>8</sub>

# 3-stage growth: pressure effect

pressure effect

$J=0.025 \text{ mA/cm}^2$



pressure: more nuclei, dense

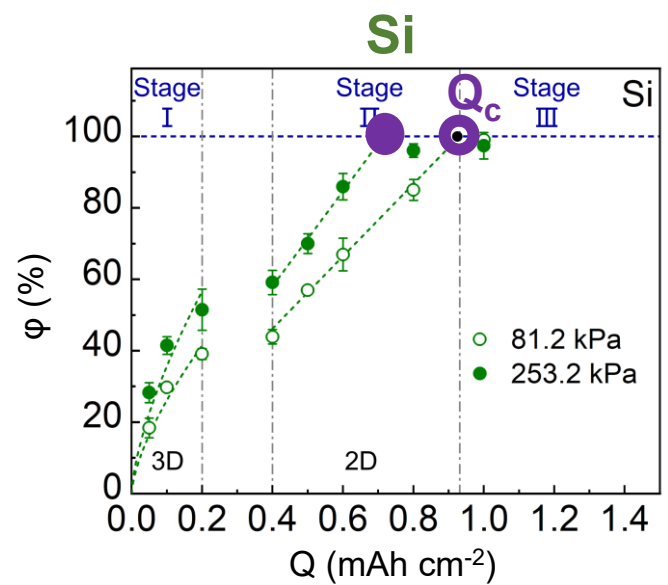
3-stage growth: Si at quasi-eq and reaction-controlled. Cu ONLY at quasi-eq



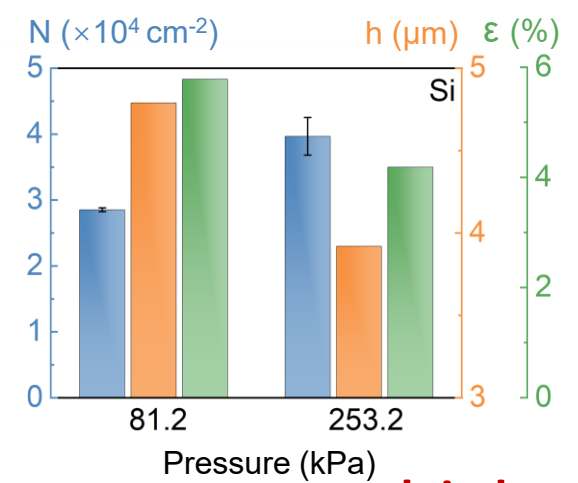
# 3-stage growth: pressure and current effect

pressure effect

$J=0.025 \text{ mA/cm}^2$

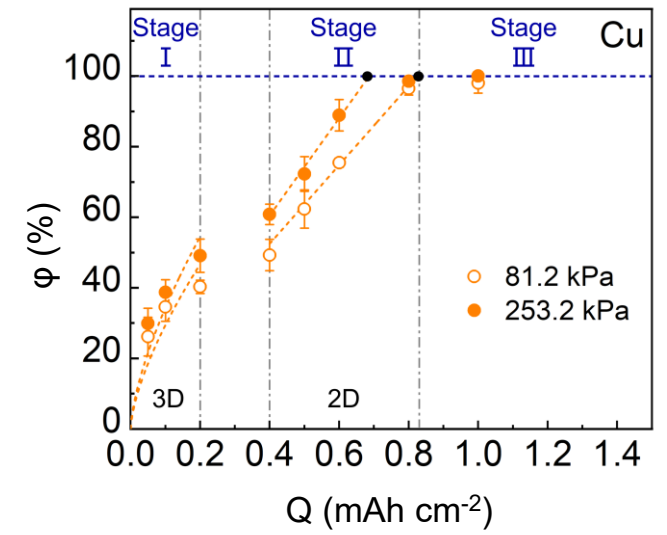


$\phi \sim Q^{2/3} N^{1/3}$      $\phi \sim Q h^{-1}$      $\epsilon \sim h Q_c^{-1}$



pressure: more nuclei, dense

Cu

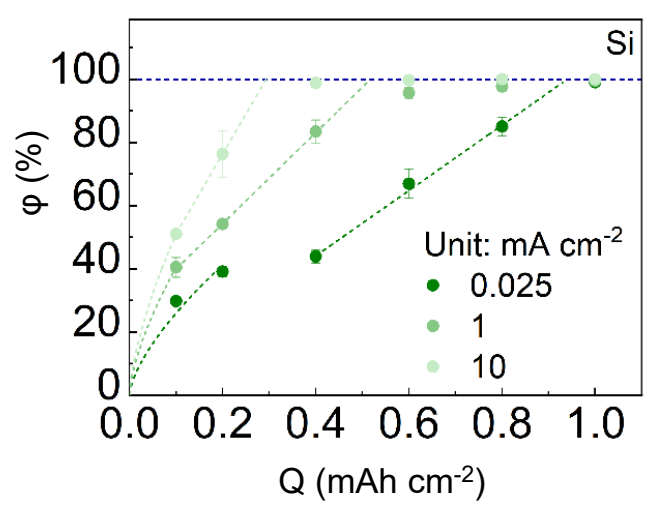


current effect

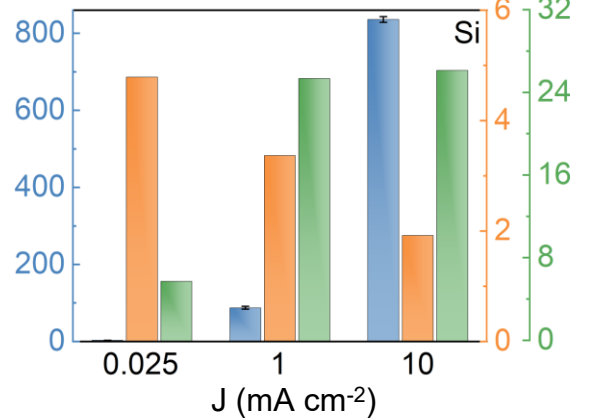
$p=81.2 \text{ kPa}$

$J_d=40 \text{ mA/cm}^2$

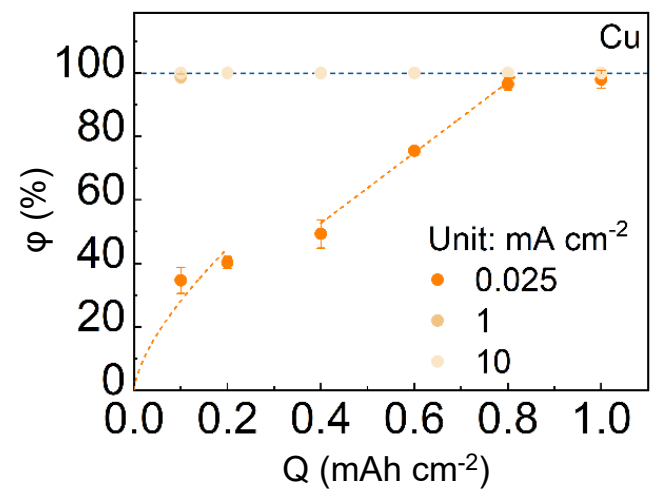
$J_0=0.2 \text{ mA/cm}^2$



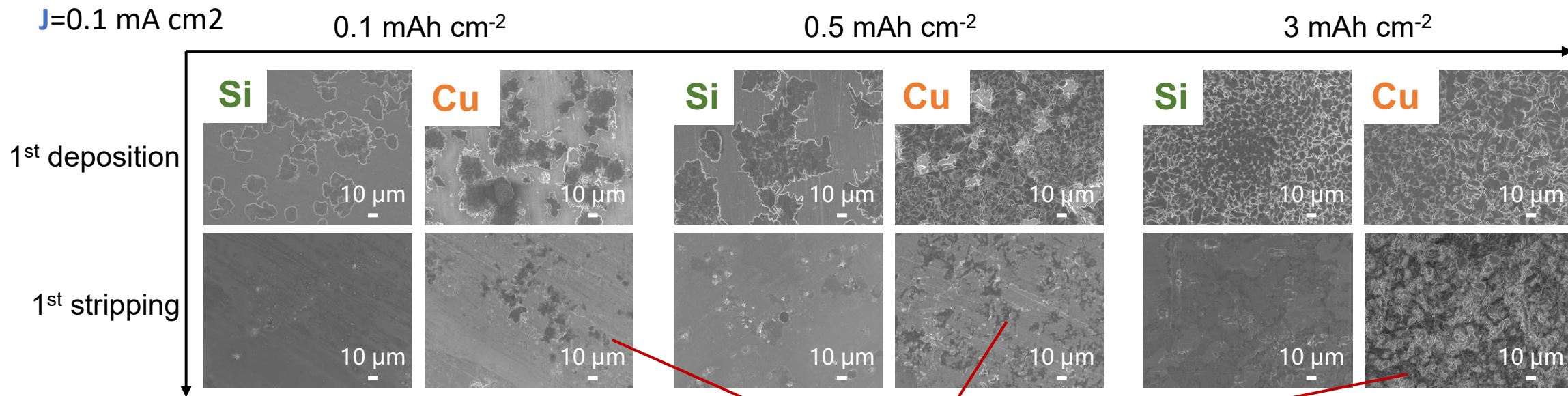
$N$  ( $\times 10^4 \text{ cm}^{-2}$ )     $h$  ( $\mu\text{m}$ )     $\epsilon$  (%)



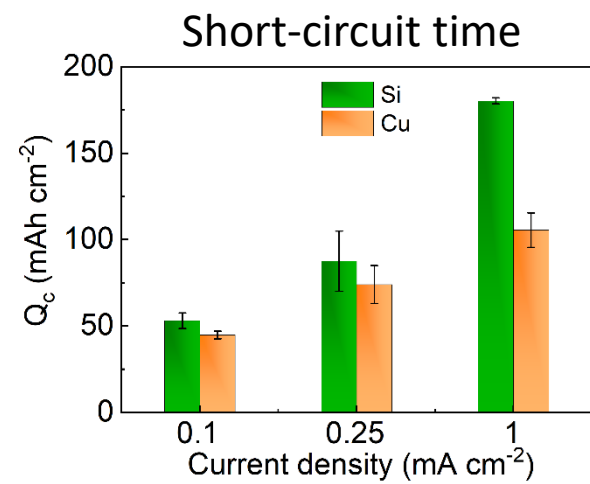
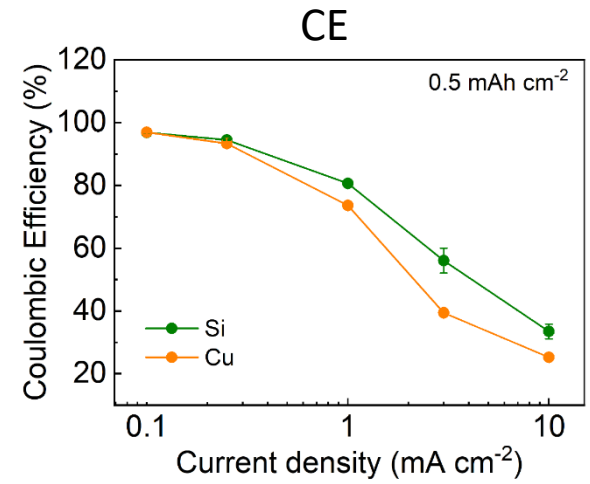
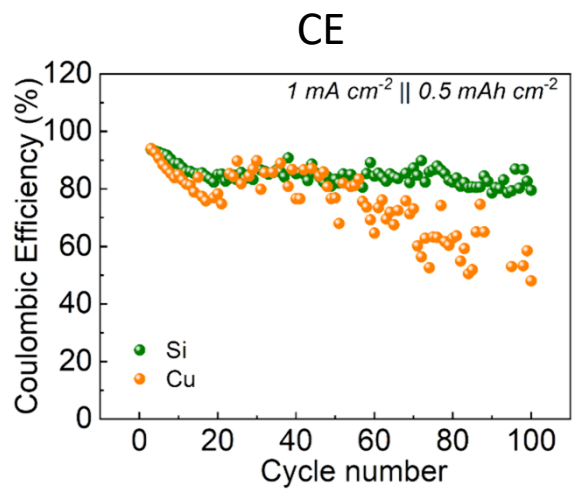
current: more nuclei, loose



# Reversibility of Li deposition/stripping

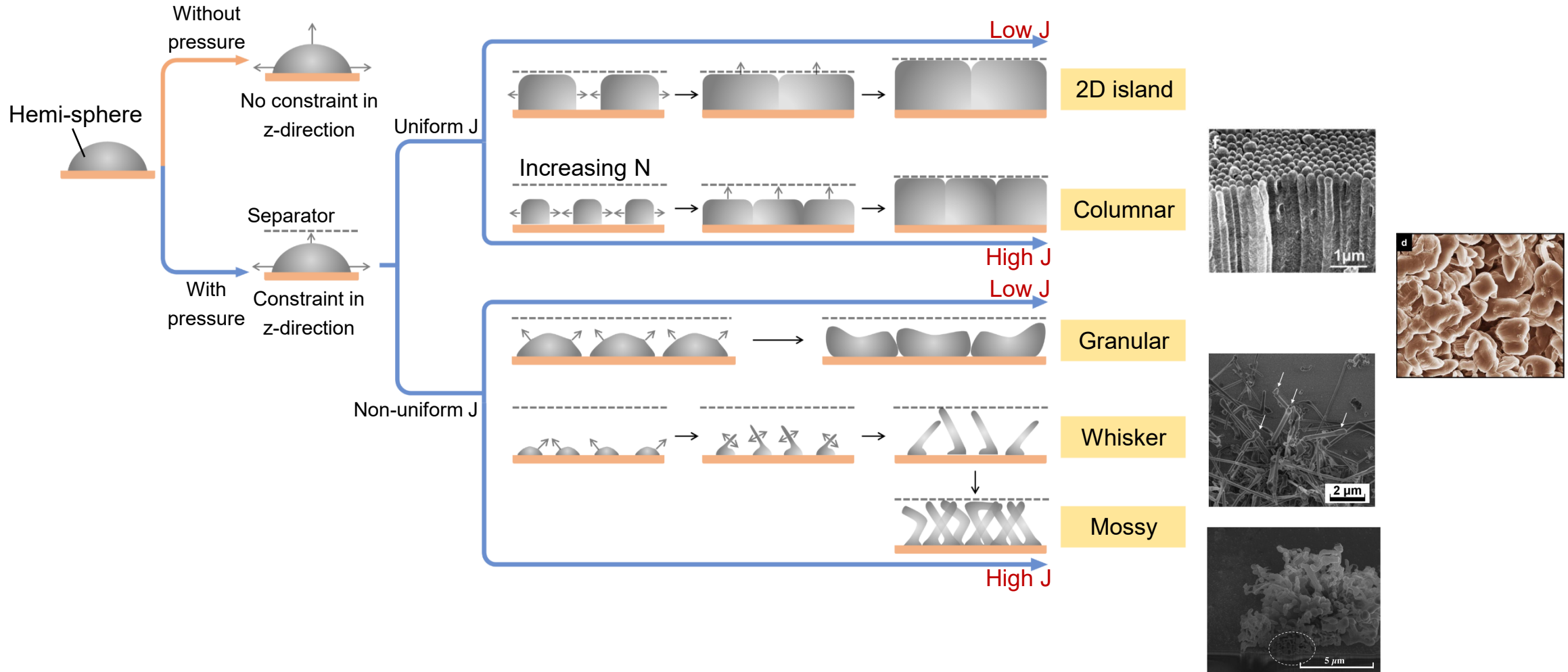


**Dead Li**



# How different morphologies form?

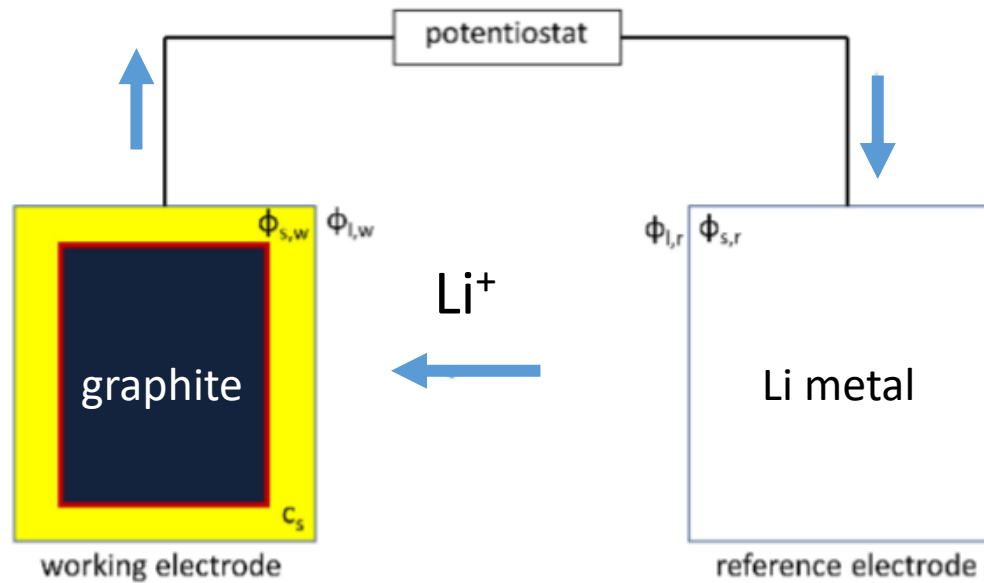
Reaction-controlled growth  $J_0=0.2 \ll J \ll J_d=40 \text{ mA/cm}^2$





Back up slides

# Fundamental insights into $V_{\text{graphite}}$



Negative (lithiation)

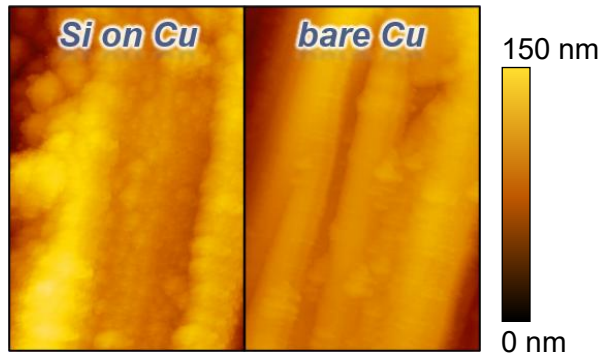
$$V \approx \frac{\mu_s}{e} + \eta_w < 0$$

zero when saturated

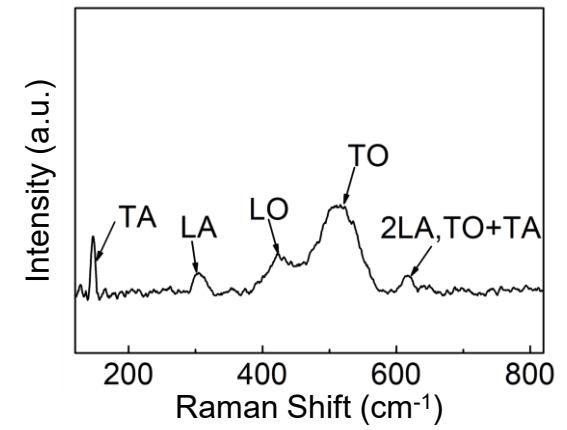
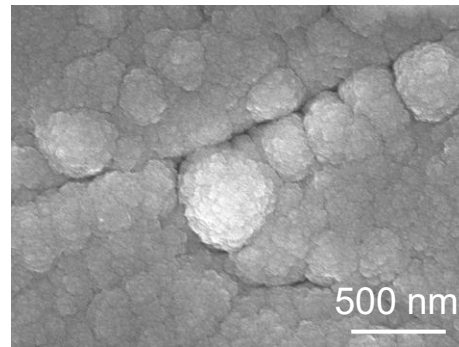
$$V = \phi_w - \phi_r = \Delta\phi_w - \Delta\phi_r + (\phi_{l,w} - \phi_{l,r}) = -\frac{\cancel{\mu_s} - \cancel{\mu_{\text{Li}}}}{e} + \frac{\cancel{\mu_{\text{Li}^+,w}} - \cancel{\mu_{\text{Li}^+,r}}}{e} + (\eta_w - \cancel{\eta_r}) + (\phi_{l,w} - \phi_{l,r})$$

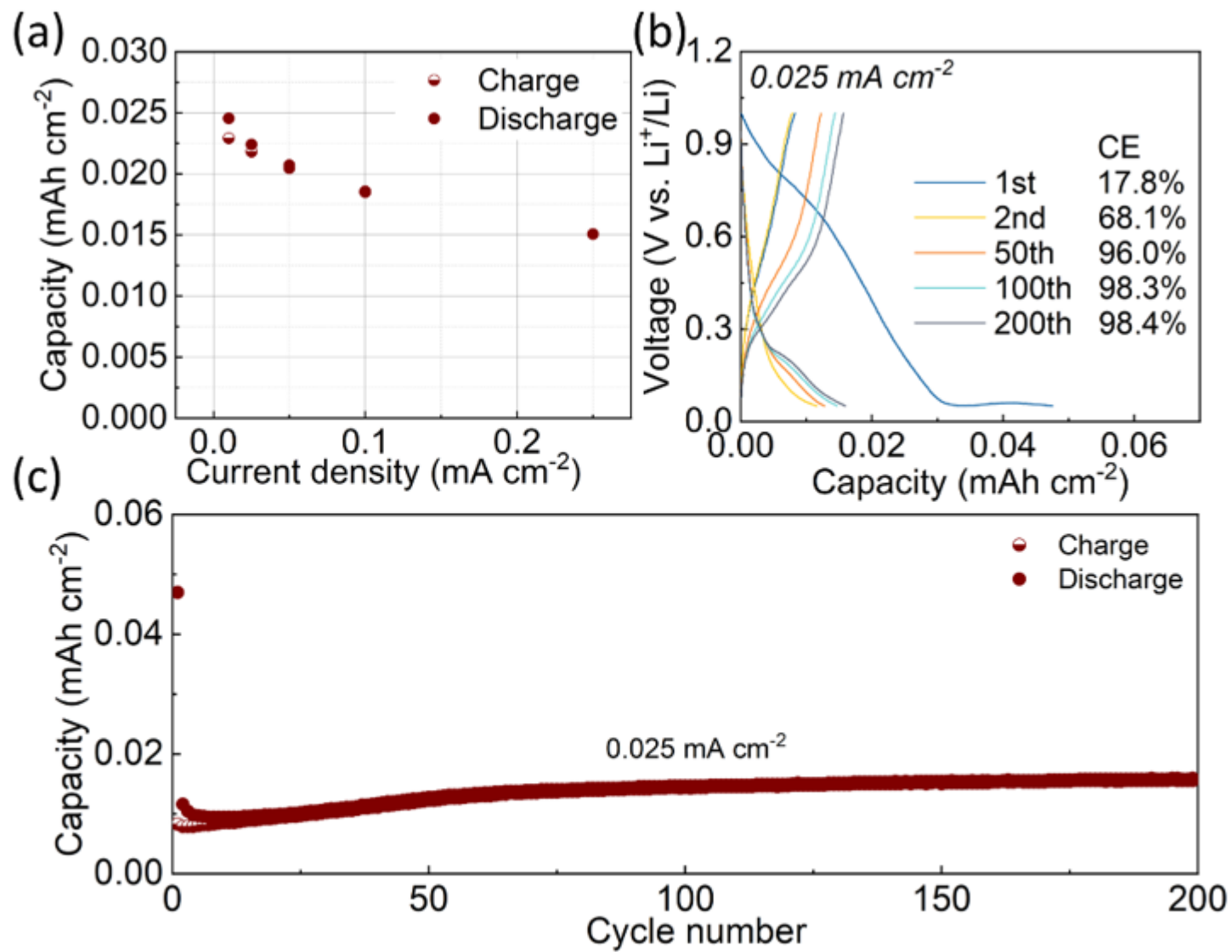
reference
Fast Li/Li<sup>+</sup> reaction at RE

No conc. gradient
High conductivity of electrolyte



Roughness 10-100 nm





Supporting Figure S2. The electrochemical properties of the silicon film.



3D growth:

$$Q = \frac{2/3 \pi r^3 \rho C_s}{A}, r \sim Q^{1/3}$$

$$S = \frac{\pi r^2 N}{A}$$

$$\frac{S}{Q} = \frac{3N}{2\rho C_s r}$$

$$S = \frac{3N}{2\rho C_s r} Q$$

$$S = aQ^{2/3}$$

$$a = \frac{3\sqrt[3]{3}}{2\sqrt[3]{2}\sqrt[3]{\pi}\sqrt[3]{\rho^2}\sqrt[3]{C_s^2}} N$$

2D growth:

$$Q = \frac{S_{\text{dep}} r_c \rho C_s}{A}$$

$$S = \frac{S_{\text{dep}}}{A}$$

$$\frac{S}{Q} = \frac{1}{\rho C_s r_c}$$

$$S = \frac{1}{\rho C_s r_c} Q$$

$$S = kQ$$

$$k = \frac{1}{\rho C_s r_c}$$

Si-Single spacer:

$$3D:a = 121.28 \pm 7.26$$

$$N = 2.7 \pm 0.1 \times 10^4 \text{ (cm}^{-2}\text{)}$$

$$2D:k = 101.35$$

$$r_c = 4.79 \text{ (}\mu\text{m)}$$

Cu-Single spacer:

$$a = 136.5 \pm 17.15$$

$$N = 3.0 \pm 0.4 \times 10^4 \text{ (cm}^{-2}\text{)}$$

$$2D:k = 101.35$$

$$r_c = 4.34 \text{ (}\mu\text{m)}$$

$$\rho = 0.534 \text{ g cm}^{-3}$$

$$C_s = 3860 \text{ mAh g}^{-1}$$

Si-Double spacer:

$$3D:a = 165.53 \pm 14.57$$

$$N = 3.7 \pm 0.3 \times 10^4 \text{ (cm}^{-2}\text{)}$$

$$2D:k = 123.51$$

$$r_c = 3.92 \text{ (}\mu\text{m)}$$

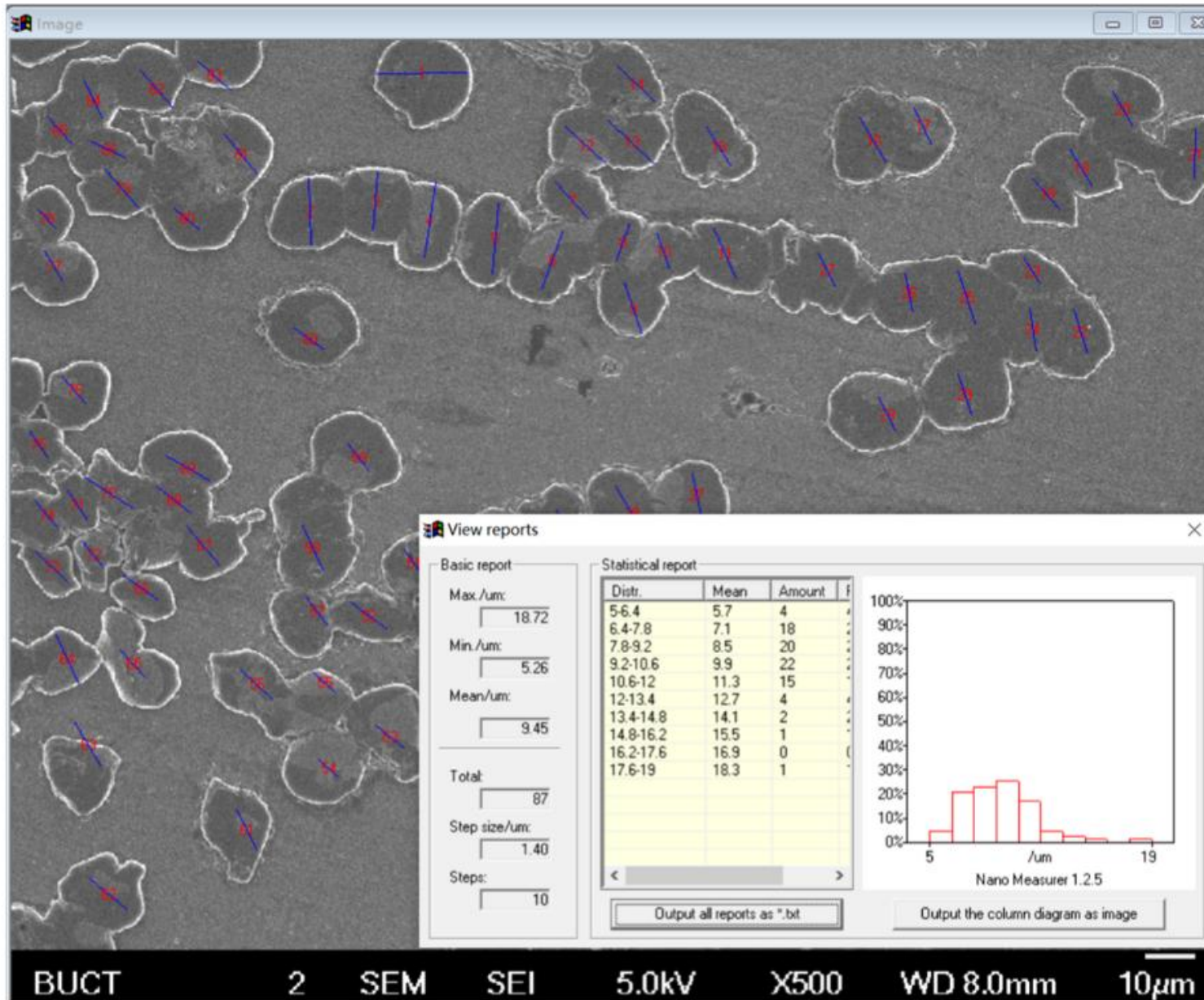
Cu-Double spacer:

$$a = 159.27 \pm 15.75$$

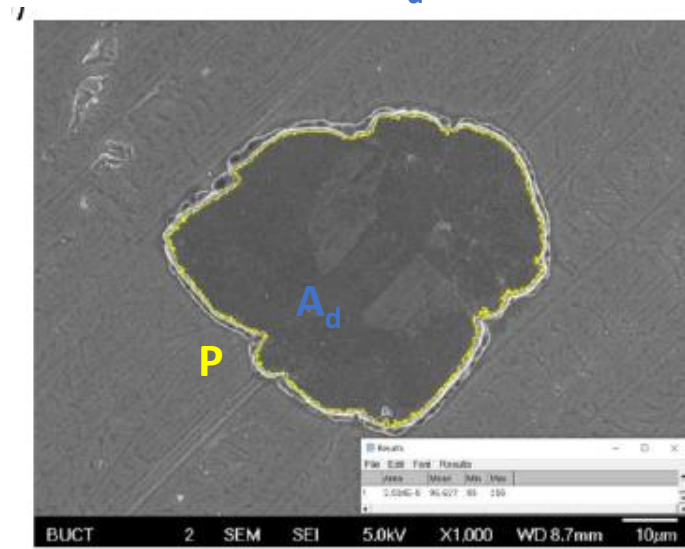
$$N = 3.55 \pm 0.35 \times 10^4 \text{ (cm}^{-2}\text{)}$$

$$2D:k = 138.08$$

$$r_c = 3.51 \text{ (}\mu\text{m)}$$



compute  $P$  and  $A_d$  with ImageJ



Cu

Magnification  $\rightarrow$

Current density ( $\text{mA cm}^{-2}$ )

0.01

0.025

0.05

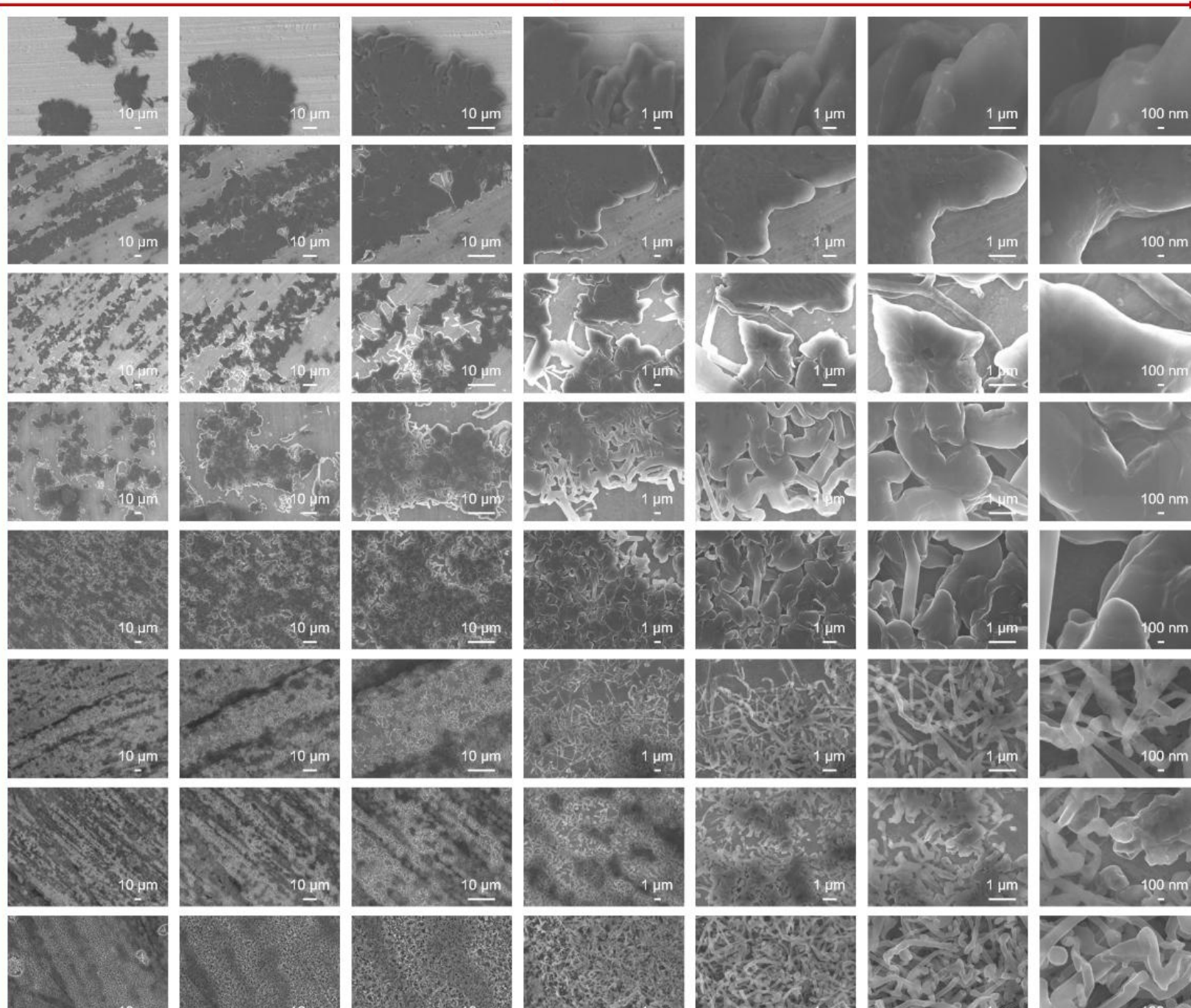
0.1

0.25

1

3

10





# Capacity(mAh cm<sup>-2</sup>)

Si

Initial

0.1 mAh cm<sup>-2</sup>

0.2 mAh cm<sup>-2</sup>

0.4 mAh cm<sup>-2</sup>

0.8 mAh cm<sup>-2</sup>

1 mAh cm<sup>-2</sup>

Test Number

