## **Supplementary information**

# Li+ Crosstalk-driven Calendar Aging in Si/C Composite Anodes

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The supporting information includes:

Methods (experimental and simulations), detailed electrochemical profiles, notes and tables.

#### Experimental

Active materials preparation:  $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$  (NCM811, 3.5 µm of D50) and graphite (15.3 µm of D50) were purchased from Canrd. Si (99.99%, 10 µm of D50) was purchased from Xinnai Metals. The Si@C (7.2 µm of D50) was prepared by Gotion Tech. using chemical vapor deposition (CVD).

Electrode preparation and cells assembly: For slurry preparation, graphite: super P (SP, Tianjin EVS): polyvinylidene fluoride (PVDF, Canrd) was 96: 2: 2, and NCM811: SP: PVDF was 94: 3: 3 in weight, respectively. Each set of mixing materials were dissolved in N-Methyl-2-pyrrolidone (NMP, Sinopharm) in weight ratio of 1:2. For Si/C composite anodes, the AMs: polyacrylic acid (PAA, Aladdin, average molecular weight ~450,000): SBR (MTI): CNT (Jiacai Tech., diameter of 10-30 nm, length of 10-20  $\mu$ m): SP = 92.5: 4.4: 1.1: 0.1: 1.9 in weight. The Si content in this paper refers to the weight ratio of Si to AMs. The Si-containing mixing materials were solved in deionized water in weight ratio of 1:2. By controlling the coating thickness, the areal capacity was controlled to ~1.60 mAh cm<sup>-2</sup> for NCM811 cathode and ~1.88 mAh cm<sup>-2</sup> for graphite anode and Si/C composite anode. Cells were assembled in glove box (Etelux, Lab 2000,  $H_2O < 0.1$  ppm,  $O_2 < 10$  ppm), using a polypropylene (PP, Celgard 2500) separator and 80 µL electrolyte containing 1 M LiPF<sub>6</sub> in ethylene carbonate/ ethyl methyl carbonate/ diethyl carbonate (EC/EMC/DEC = 3:4:2 in volume) plus 10 wt% fluoroethylene carbonate (FEC) purchased from Canrd. In the three-electrode assembly, electrodes with diameter of 12 mm and 160  $\mu$ L electrolyte were used, and Li wire (China Energy Lithium) was used as a reference electrode.

*Electrochemical test:* Electrochemical tests were performed using a Neware system (CT-4008Tn). Before aging tests, all assembled cells were cycled with 0.1 mA (~0.06 C) constant current-constant voltage (CC-CV) charging to 4.2 V and 0.05 mA, and 0.1 mA CC discharging to 2.5 V for twice. The last discharge capacity was identified as calibrated capacity, and cells with calibrated capacity of ~1.8 mAh were selected. For calendar aging test, cells were 0.1 C CC-CV charged to 4.2 V and 0.05 C at first, then cells were stored at RT for hours. At the end, cells were 0.1 C discharged to 2.5 V. For repeating storage test, cells were subjected to the single aging test with the storage time limited to 24 h for 10 times. EIS test was conducted using an electrochemical workstation (Admiral SquidstatTM Prime) with a frequency range of  $10^{-1}$ - $10^{6}$  Hz and an applied signal amplitude of 5 mV. For GITT test, cells were charged at 0.1C (0.18 mA) with a 30-minute pulse and a 2-hour rest period to 4.3 V (Figure S14).

*Characterization:* After cell disassembly, anodes were rinsed with dimethyl carbonate (DMC, Sigma) and dried out for further characterizations. Optical images were captured using a camera (2F01M, Revealer). SEM images were acquired under an accelerating voltage of 15 kV (Phenom Scientific). SEM-EDS was conducted using a Genimi SEM 500 under an accelerating voltage of 3 kV. TEM-EDS images were acquired using a JEM-2100F at 200 kV. XPS (Thermo Fisher K-Alpha<sup>+</sup>) with Al Kα radiation was used to determine the chemical component of the anode surface and cross

section. An area of  $2 \times 2 \text{ mm}^2$  was etched using Ar<sup>+</sup> with 1 keV and 10 mA. Samples were transferred through a homemade air-tight device and exposed to air for less than 1 min.

#### Simulations

The 1D models established here were based on the pseudo two-dimensional (P2D) model developed by Newman and Doyle,<sup>1</sup> incorporating porous electrode theory and concentrated solution theory, and the corresponding governing equations are listed in Table S3.

*Si/C hybrid anode model construction*: The addition of Si is realized by incorporating an additional electrode into the anode.<sup>2</sup> To keep the porosity between the graphite anode and the Si/C composite anodes consistent, the Si content is controlled by the volume ratio f, denoted as the ratio of Si to active materials (AMs).  $\varepsilon_a$  represents the volume fraction of AMs in the anode, and the volume fractions of graphite ( $\varepsilon_c$ ) and Si ( $\varepsilon_{Si}$ ) are as follows:

$$\varepsilon_c = \varepsilon_a(1 - f) \tag{1}$$

$$\varepsilon_{Si} = \varepsilon_a f \tag{2}$$

The electrode capacity is converted according to the following equation:

$$Q = FL\varepsilon\Delta c_{s,\max} \tag{3}$$

where F, L,  $\varepsilon$ ,  $\Delta$ , and  $c_{s,max}$  represent Faraday constant (96485 C mol<sup>-1</sup>), electrode thickness, volume fraction of electrode active material, operating window of the electrode, and maximum Li<sup>+</sup> concentration, respectively. For the Si/C composite anodes with different Si contents, the anode thickness is expressed as:

$$L_{a} = \frac{Q_{a}}{F(\varepsilon_{C}\Delta_{C}c_{C,s,\max} + \varepsilon_{Si}\Delta_{Si}c_{Si,s,\max})}$$
(4)

where footnotes C and Si denote the corresponding physical parameters of graphite and Si, respectively.

*Calendar aging sector:* The calendar aging loss was proved to be dominated by the side reaction occurred on the anode.<sup>3,4</sup> Thus, only side reaction on anode was considered. The reaction current density on Si/C composite anodes  $(j_a)$  follows:

$$j_a = j_C + j_{Si} \tag{5}$$

$$j_{C/Si} = j_{lith.} + j_{SEI} \tag{6}$$

where the current density of AMs ( $j_{C/Si}$ , graphite or Si) equals the sum of (de)lithiation process ( $j_{lith}$ ) and side reaction ( $j_{SEI}$ ).

$$j_{lith.} = i_0 \left[ \exp\left(\frac{\alpha F \eta_{lith.}}{RT}\right) - \exp\left(\frac{-(1-\alpha)F \eta_{lith.}}{RT}\right) \right]$$
(7)

$$\eta_{lith.} = \Phi_1 - \Phi_2 - U_{Si/C} - j_{C/Si} R_{SEI}$$
(8)

$$j_{SEI} = i_{SEI,0} \left[ -\exp(\frac{(1-\alpha)F\eta_{SEI}}{RT}) \right]$$
(7)

$$\eta_{SEI} = \Phi_1 - \Phi_2 - U_{SEI} - j_{C/Si} R_{SEI}$$
(8)

where  $i_0$ ,  $\eta$ , U,  $R_{SEI}$  refer to the exchange current density, over potential, equilibrium potential and SEI impedance, respectively. And the footprint "lith./SEI" represents valuable (de)lithiation or side reaction, respectively. The side reaction is irreversible, so the  $j_{SEI}$  is described by the cathodic Tafel expression (Equation 7).<sup>5</sup>

The  $U_{SEI}$  was set to 0.4 V as reported.<sup>5–7</sup> And the  $R_{SEI}$  is changing with the SEI growth and expressed as:

$$R_{SEI} = \delta_{SEI} / K_{SEI}$$
<sup>(9)</sup>

$$d\delta_{SEI} / dt = -v_{SEI} j_{SEI}, \delta_{SEI,0} = 1 \,\mathrm{nm}$$
(10)

where  $\delta_{SEI}$  and  $K_{SEI}$  are thickness and ionic conductivity of SEI, and  $v_{SEI}$  is the moar volume of SEI.

The 2D models were established obeying the same physical equations. For the model geometry design, the parameters were adopted from those in 1D models. The primary distinction lies in configuring the electrolyte region as a conductive binder,<sup>8</sup> with  $j_{C/Si}$  occurring solely on the 1D boundary at the particle surface. A dilute species transport module is introduced, with solid-phase Li<sup>+</sup> diffusion occurring within the 2D computational domain inside the particles. All relative parameters are summarized in Table S4.



Figure S1. The voltage profiles of full cell, anode and cathode for cells with 10Si anodes.



Figure S2. The color changes of anodes (a) before ("pristine" mentioned in the text) and (b) after storage ("aged" mentioned in the text).



Figure S3. (a) Elemental maps of pristine (fully charged) graphite anode, 5Si anode, 10Si anode, and 15Si anode. (b) Element content of different anodes. (c) The F 1s spectra of pristine anodes.



**Figure S4.** (a) The voltage profile of full cells with 10Si anode from the 2D model. (b) Model verification compared with experimental data.



**Figure S5.** The SOC profiles of graphite particles. (a) SOC changes during ~0.1 C charging. (b) An enlarge image of (a). (c) SOC decrease during storage and (d) the enlarge image.



Figure S6. The potential distribution at (a) 0 h, (b) 1.6 h, and (c) 24 h.



**Figure S7.** The redistribution of  $c_{Li^+}$  in the Si particle. (a-c) The  $c_{Li^+}$  distribution. (d-f) The current density distribution, *I* in electrolyte. (g-i) The  $c_{Li^+}$  distribution in electrolyte.



**Figure S8.** The profiles of (a) average *I* and (b)  $c_{Li^+}$  in electrolyte during 24 h storage.



**Figure S9.** The side reaction for anodes with different Si content during 24 h storage. The (a)  $I_{SEI}$ , (b)  $Q_{SEI}$ , (c)  $\delta_{SEI}$  profiles of graphite. The (d)  $I_{SEI}$ , (e)  $Q_{SEI}$ , (f)  $\delta_{SEI}$  profiles of Si.



**Figure S10.** The Li<sup>+</sup> crosstalk between graphite and Si particles in improved 10Si anode during calendar aging. The (a)  $c_{Li^+}$  distribution and Li<sup>+</sup> movement, (b) current field and (c) concentration field in electrolyte at different storage time.



**Figure S11.** The change of Li<sup>+</sup> transfer between graphite and Si particles. The (a)  $c_{Li^+}$  distribution and Li<sup>+</sup> movement, (b) current field, (c) concentration field in electrolyte and (d) particle potential at different storage time.



Figure S12. The SOC changes of (a) graphite and (b) Si particles.



**Figure S13.** The formation voltage-capacity curves of cells with 15Si and 15Si@C anodes at 0.1 mA (~0.05 C).

Note S1:  $D_{Li^+}$  calculation based on EIS.

For the low-frequency region, the relationship between impedance (Z') and frequency ( $\omega$ ) follows:

$$Z' = R_{\Omega} + R_{ct} + A_w \omega^{-0.5} \tag{11}$$

where  $A_w$  is Warburg coefficient. And the Li<sup>+</sup> diffusion coefficient can be calculated as:<sup>9</sup>

$$D_{Li^{+}} = 0.5 \left(\frac{RT}{n^{2}F^{2}ACA_{w}}\right)^{2}, \omega \gg 2D_{Li^{+}}/L^{2}$$
(12)

where *R*, *T*, *n*, *F*, *A*, *C* represent the universal gas constant (8.314 J K<sup>-1</sup> mol<sup>-1</sup>), temperature (298 K), the electron transfer number (n=1), the Faraday constant (96485 C mol<sup>-1</sup>), electrode area (0.01 m<sup>-2</sup>) and Li<sup>+</sup> concentration (1×10<sup>3</sup> mol m<sup>-3</sup>).



Figure S14. Schematic of Li diffusion rate in GITT test.

Note S2:  $D_{Li^+}$  calculation based on GITT.

The  $D_{Li}^+$  was calculated based on:<sup>10,11</sup>

$$D_{Li^{+}} = \frac{4}{9\pi} \cdot \left(\frac{\Delta V_s}{\Delta V_t}\right)^2 \cdot \frac{r_p^2}{t_p}$$
(1)

where  $\Delta V_s$ ,  $\Delta V_t$ ,  $r_p$ ,  $t_p$  represents the steady-state open circuit voltage increment after the pulse, transient voltage increase after the pulse, the active particle radius, and the pulse duration, respectively. The  $r_p$  here was 10 µm for both anodes, and  $t_p$  was 2 h.



Figure S15. Voltage profiles of cells after more than 300 h storage.

<b>Table S1.</b> Elemental content table for fully charged (Pristine) anodes and aged anodes
(after 72 h of storage) in wt%.

	С	0	F	Si	Р
Pristine state					
Gr	63.03	11.81	23.04	0.47	1.64
5Si	63.65	9.89	22.42	2.96	1.08
10Si	65.94	15.94	11.22	6.6	0.3
15Si	68.5	8.74	10.44	11.77	0.55
After 72 h	of storage				
Gr	57.98	35.62	5.25	0.01	1.14
5Si	62.84	30.88	2.71	3.28	0.28
10Si	54.23	40.57	0.38	4.82	0
15Si	56.3	33.88	2.73	6.97	0.11

Cells	$R_{arOmega}\left(\Omega ight)$	$R_{SEI}\left( \Omega ight)$	$R_{ct}\left(\Omega ight)$
	1.831	11.42	22.91
15Si	2.491	16.26	19.42
	1.513	13.69	21.02
	1.56	13.29	32.63
15Si@C	1.692	15.11	28.83
	2.075	14.45	30.4

Table S2. Impedance values.

Process	Governing equations	Boundary and initial conditions	
Porous electrod	le regions (m = NCM811, C, or Si)		
Mass balance	$\frac{\partial c_{1,m}}{\partial t} = \frac{1}{r^2} \frac{\partial}{\partial r} \left( r^2 D_m \frac{\partial c_m}{\partial r} \right)$	$-D_{1,m} \frac{\partial c_m}{\partial r}\Big _{r=R_i} = j_m, \frac{\partial c_{1,m}}{\partial r}\Big _{x=0} = 0,$ $c_{1,m}\Big _{t=0} = c_{1,m}^0$	
Kinetics	$\eta_{NCM811}^{ct} = \Phi_1 - \Phi_2 - U_{NCM811}$ $j_{NCM811} = i_0 [\exp(\frac{\alpha F \eta_{NCM811}^{ct}}{RT}) - \exp(\frac{(1-\alpha)F \eta_{NCM811}^{ct}}{RT}]$	·)	
Charge balance	$\begin{split} i_1 &= -\sigma_m^{\text{eff}} \frac{\partial \Phi_1}{\partial x}, a_m j_m = \frac{1}{F} \frac{\partial i_2}{\partial x}, \\ i_1 &= I \end{split}$	$i_1\Big _{x=0} = I, i_1\Big _{x=\delta_n} = 0,$ $i_1\Big _{x=\delta_n+\delta_{sep}} = 0, i_1\Big _{x=L} = I$	
Electrolyte regi	ons		
		$D_{2,n}^{\text{eff}} \frac{\partial c_2}{\partial x}\Big _{x=0} = 0,$	
		$D^{\text{eff}} \frac{\partial c_2}{\partial c_2} = -D^{\text{eff}} \frac{\partial c_2}{\partial c_2}$	

Mass ba

alance 
$$\mathcal{E}_m \frac{\partial c_2}{\partial t} = \frac{\partial}{\partial x} \left( D_{2,m}^{\text{eff}} \frac{\partial c_2}{\partial x} \right) + a_m i_m \left( 1 - t_+ \right)$$

$$\begin{split} D_{2,n} \frac{\partial c_{2,n}}{\partial x}\Big|_{x=0} &= 0, \\ D_{2,n} \frac{\partial c_{2}}{\partial x}\Big|_{x=\delta_{a}} &= D_{2,sep}^{\text{eff}} \frac{\partial c_{2}}{\partial x}\Big|_{x=\delta_{a}}, \\ D_{2,p} \frac{\partial c_{2}}{\partial x}\Big|_{x=\delta_{n}+\delta_{sep}} &= D_{2,sep}^{\text{eff}} \frac{\partial c_{2}}{\partial x}\Big|_{x=\delta_{n}+\delta_{sep}}, \\ D_{2,p} \frac{\partial c_{2}}{\partial x}\Big|_{x=L} &= 0 \end{split}$$

$$i_{2} = -k_{m}^{\text{eff}} \frac{\partial \Phi_{2}}{\partial x} + \frac{2k_{m}^{\text{eff}}RT}{F} (1 - t_{+})(1 + \frac{dl \ln f_{+}}{dl \ln c_{2}}) \frac{\partial \ln c_{2}}{\partial x} \quad i_{2}|_{x=0} = 0, i_{2}|_{x=\delta_{n}} = I,$$

$$i_{2}|_{x=\delta_{n}+\delta_{sep}} = I, i_{2}|_{x=L} = 0$$

Charge balance

# where footnotes 1, and 2 refer to solid and liquid phase parameters, respectively.

Boundary and initial conditions

Parameters	Symbol	Value	Unit	Ref.
Radius of NCM811	R <sub>NCM</sub>	2	m <sup>-6</sup>	Measured
Radius of graphite	$R_C$	5	m <sup>-6</sup>	Measured
Radius of Si	$R_{Si}$	4	m <sup>-6</sup>	Measured
Transfer coefficient ( $\alpha_m$ )	$\alpha_a, \alpha_c$	0.5		5
Transfer coefficient of side reaction	α	0.5		5
Li <sup>+</sup> concentration in electrolyte	Ce	1000	mol m <sup>-3</sup>	Measured
Maximum Li <sup>+</sup> concentration of Si	$C_{Si,s,max}$	278000	mol m <sup>-3</sup>	12
Maximum Li <sup>+</sup> concentration of graphite	$C_{C,s,max}$	31507	mol m <sup>-3</sup>	12
Maximum Li <sup>+</sup> concentration of NCM811	$C_{NCM811,s,\max}$	50060	mol m <sup>-3</sup>	13
Diffusivity of NCM811	$D_{s, NCM}$	10-13	$m^2 s^{-1}$	14
Diffusivity of graphite	$D_{s, C}$	3.9×10 <sup>-14</sup>	$m^2 s^{-1}$	14
Diffusivity of Si	$D_{s, Si}$	10-15	$m^2 s^{-1}$	15
Exchange current density of NCM811	i <sub>0, NCM</sub>	10	A m <sup>-2</sup>	Estimated
Exchange current density of graphite	i <sub>0, C</sub>	0.1	A m <sup>-2</sup>	Estimated
Exchange current density of Si	$i_{0, Si}$	0.5	A m <sup>-2</sup>	Estimated
Exchange current density of side reaction for graphite	İq, sel, c	2×10 <sup>-6</sup>	A m <sup>-2</sup>	5,16
Exchange current density of side reaction for Si	İ <sub>0, SEI, Si</sub>	1.5×10 <sup>-4</sup>	A m <sup>-2</sup>	Larger than $i_{0, SEI, C}$ <sup>17</sup>
Thickness of graphite anode	$L_a$	81	m <sup>-6</sup>	Fitted
Thickness of separator	$L_s$	20	m <sup>-6</sup>	Measured
Thickness of cathode	$L_c$	70	m <sup>-6</sup>	Measured
Ratio of Si in AMs	f	0		
Anode porosity	$\mathcal{E}_{a}$	0.47		Estimated
Volume fraction of Si	$\mathcal{E}_{Si}$	$\mathcal{E}_a f$		
Volume fraction of graphite	$\mathcal{E}_{C}$	$\mathcal{E}_a(1-f)$		

Table S4. Key parameters in the models.

Cathode porosity	$\mathcal{E}_c$	0.43		Fitted
Working window of Si	$\Delta_{Si}$	1		Estimated
Working window of graphite	$\Delta_{C}$	1		Estimated
Working window of NCM811	$\Delta_{_{NCM}}$	0.72		Estimated
Initial SOC of NCM811	SOC <sub>NCM811</sub> ,	0.95		Fitted
Initial SOC of Si	SOC <sub>Si0</sub>	0.004		Fitted
Initial SOC of NCM811	$SOC_{C,0}$	0.018		Fitted
Density of the SEI on Si	$\rho_{SEI, Si}$	1.60×10 <sup>4</sup>	kg m <sup>-3</sup>	Estimated
Density of the SEI on graphite	$ ho_{SEI, C}$	1.69×10 <sup>3</sup>	kg m <sup>-3</sup>	18
Molar mass of the SEI on Si	$M_{SEI, Si}$	0.08	kg mol <sup>-1</sup>	Estimated
Molar mass of the SEI on graphite	M <sub>SEI, C</sub>	0.162	kg mol <sup>-1</sup>	18
Ionic conductivity of the SEI on Si	K <sub>SEI, Si</sub>	1×10-5	S m <sup>-1</sup>	Estimated
Ionic conductivity of the SEI on graphite	K <sub>SEI, C</sub>	5×10-5	S m <sup>-1</sup>	Estimated
Equilibrium potential of side reactions	U <sub>SEI</sub>	0.4	V	5–7

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