Supplementary Materials for

Extremely fast-charging lithium ion battery enabled by dual-gradient structure design

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Published 27 April 2022, Sci. Adv. 8, eabm6624 (2022)
DOI: 10.1126/sciadv.abm6624

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Supplementary Materials

Reconstruction of the 3D structure of electrodes.

The laboratory-based X-ray tomography technique (Xradia Versa XRM-500 system, Carl ZeissX-ray Microscopy Inc.) was adopted to collect the reconstructed 3D tomography data. The electrode sample for the X-ray tomography was prepared by cutting a small piece from the thick electrode. The cone beam system was operated with voltage of 50 keV. Transmitted X-ray traveled through the sample with the geometric magnified projection to the detector and then converted to the visible light by a scintillator. The absorption contrast in imaging was used for the internal structure study in terms of the density difference of particles and pores. 1600 projections with a 360° rotation were taken for one 3D volume with a pixel size of 0.5094 µm. All of the datasets were carried out a correction of beam hardening and then reconstructed with the FBP algorithm. The reconstruction of collected 3D X-ray tomography data was done by the software VGstudio MAX 2.2. From the reconstructed structures (fig. S6), we can see that the void space randomly and evenly distributed in the random sample whereas the void space congregates near the top surface in the dual-gradient sample. To identify the porosity, the gray-level images of cross-sections obtained by X-ray tomography were converted into binary images via image processing. A multi-level image threshold method was employed to distinguish the void space (gray scale intensity. 0) from other solid materials (gray scale intensity. 255). The porosity of the electrode was calculated based on the reconstructed 3D structure by dividing the volume of the void space to the total volume of the whole electrode. The overall porosity of the random sample and dual gradient sample were calculated as 34.2% and 33.4%, respectively. To identify the distribution of the porosity along vertical direction of the electrode, the 3D structure is evenly divided into five parts. Then, the porosity of each part is calculated along the vertical direction and plotted out as the porosity versus the thickness.

Electrochemical simulation.

We employed a particle-level theoretical model (29-31) to simulate the charging process in an electrode with a constant overall average porosity and total volume. Compared to the pseudo-two-dimensions (P2D) Newman model(32,33), the particle-level model enable us to directly investigate how the spatial distributions of porosity and different size particle affect the lithium ion battery performance. For simulation, we mainly focus on concentration polarization in the electrolyte and particle utilization. Thus, it is assumed that the electrode consists of particles with different sizes and the pore fully filled by electrolyte, neglecting neither the additives nor the formation of solid electrolyte interface (SEI) on the particle surface. Note that the layered structure of graphite particle can substantially affect the diffusion process, leading to lithium diffusion along the layered plane much larger than that across the layered plane. Thus, it is essential to take the anisotropic diffusion behavior into account. In this model, the electrode consists of n graphite particles. A domain parameter $\Psi_p$ ($p = 1, 2, \ldots n$) is introduced to characterize the orientation and positon of the $p^{th}$ particle. Here $\Psi_p$ equals 1 inside the $p^{th}$ particle and 0 outside the particle, varying smoothly from 0 to 1 on the particle surface. Fick’s law is used to describe the lithium diffusion in the $p^{th}$ particle.

$$\Psi_p \frac{\partial c_i}{\partial t} = \nabla \cdot (\Psi_p D_x \nabla c_i) + \left| \Psi_p J_p \right|,$$

(1)
where \( c_s \) represents the lithium concentration per unit volume in solid particles and \( t \) is time. \( J_p \) is the flux inserted into the particle due to electrochemical reaction on the surface during charging process. \( D_s \) is the anisotropic diffusion tensor in the global coordinate system, which is related to the diffusion tensor in the local coordinate system by

\[
D_s = Q \begin{bmatrix} 0 & 0 & 0 \\ 0 & D_s & 0 \\ 0 & 0 & D_s \end{bmatrix} Q^T,
\]

(2)

\[
Q = \begin{bmatrix} \cos \theta_x \cos \theta_y - \sin \theta_x \sin \theta_y \\ -\cos \theta_x \sin \theta_y - \sin \theta_x \cos \theta_y \\ \sin \theta_x \sin \theta_y \end{bmatrix} \begin{bmatrix} \sin \theta_z \cos \theta_x + \cos \theta_z \sin \theta_x & \sin \theta_x \sin \theta_z & \sin \theta_x \sin \theta_z \\ -\sin \theta_x \sin \theta_z & -\cos \theta_x \cos \theta_z + \sin \theta_x \cos \theta_z & \cos \theta_x \sin \theta_z \\ \cos \theta_z \sin \theta_y - \cos \theta_z \sin \theta_y & \sin \theta_z \sin \theta_y + \cos \theta_z \cos \theta_y & \cos \theta_z \sin \theta_y \cos \theta_y \end{bmatrix} .
\]

(3)

where are \( \theta_x, \theta_y \) and \( \theta_z \) Euler angles. The concentration evolution in the electrolyte is governed by the following equation

\[
\frac{\partial c_l}{\partial t} = \nabla \cdot (D_l \nabla c_l) - \nabla \cdot \left( \frac{i_t^0}{F z_+} \right),
\]

(4)

where \( c_l \) represents the Li\(^+\) ion concentration in the electrolyte, \( F \) is the Faraday constant. \( D_l, i_t^0 \) and \( z_+ \) represent the diffusion coefficient of the electrolyte, the transfer number of lithium ion and the charge of the lithium ion, respectively. \( i \) is the current density vector that satisfies the following equation due to the charge conversation

\[
\nabla \cdot i = 0.
\]

(5)

The intercalation flux within the interface between the particle and electrolyte is described by the Butler-Volmer equation

\[
J_p = k_0 \left( c_i \right)^{\alpha} \left( c_s,_{\text{max}} - c_s \right)^{1-\alpha} \left[ \exp \left( \frac{\alpha F \eta}{RT} \right) - \exp \left( -\frac{(1-\alpha) F \eta}{RT} \right) \right],
\]

(6)

where \( k_0 \) is the intercalation rate constant, \( \alpha \) is the symmetry factor, \( \eta \) is the overpotential that is defined by \( \eta = \phi_s - \phi_l - U^\Theta \). \( U^\Theta \) is the open circuit potential. \( \phi_s \) and \( \phi_l \) are the electrostatic potential of the particle and electrolyte, respectively. To identify the electrostatic potential of the electrolyte \( \phi_l \), the following equation is used

\[
\nabla \left[ \kappa \nabla \phi_l + \frac{k t_+^0}{F z_+} \left( \frac{\partial}{\partial c_i} \right) \nabla c_i \right] = 0.
\]

(7)
where $\kappa$ is the ionic conductivity, $\mu$ is the chemical potential defined by $\mu = \mu_0 + RT \ln(f_c)$, where $\mu_0$, $R$, $T$ and $f$ are the reference chemical potential, the ideal gas constant, temperature and the activity coefficient, respectively. In our simulation, we concentrate on the galvanostatic charging process, where a constant current density ($I_{app}$) is applied to the electrode. For simplicity, we assume that electrostatic potential $\phi_s$ is identical in different particles due to the high electron conductivity and the potential of the counterpart electrode (Li metal) maintains at 0 V. Thus, the electrostatic potential $\phi$ is determined by $I_{app} = -i_0[\exp(\alpha F(\phi_s - U^\theta)/RT) - \exp(-(1-\alpha)F(\phi_s - U^\theta)/RT)]$, where $i_0$ can be considered as the exchange current density at the Li-electrolyte interface.

For boundary conditions, the constant lithium ion flux ($J_{app} = I_{app}/F$) is applied at the top of the anode and zero current density at the bottom and two lateral sides of the electrode. Here, a virtual frame surrounding the whole electrode is introduced. It is characterized by another domain parameter $\Psi_w$, which equals 1 inside the side walls and 0 in the electrode, varying smoothly between them. Using these domain parameters, we can flexibly deal with the complex morphology by adopting the smoothed boundary method to combine the above conversation equations (Eq. (1), (4), (5)), interface reaction (Eq. (6)) together with boundary conditions (35,36). The governing equations are written as follows

$$\sum_{p=1}^{n} \psi_p \frac{\partial c_i}{\partial t} = \nabla \cdot \left( \sum_{p=1}^{n} \psi_p D_i \nabla c_i \right) + \sum_{p=1}^{n} \nabla \psi_p |J_p|, \quad (8)$$

$$\left(1 - \sum_{p=1}^{n} \psi_p - \psi_w\right) \frac{\partial c_i}{\partial t} = \nabla \cdot \left(\left(1 - \sum_{p=1}^{n} \psi_p - \psi_w\right)D_i \nabla c_i \right) - \sum_{p=1}^{n} \nabla \psi_p |J_p| + \nabla \psi_w |J_{app}|, \quad (9)$$

$$\nabla \cdot \left(1 - \sum_{p=1}^{n} \psi_p - \psi_w\right) \kappa \nabla \phi = F \sum_{p=1}^{n} \nabla \psi_p |J_p| - F \nabla \psi_w |J_{app}|$$

$$- \nabla \cdot \left(1 - \sum_{p=1}^{n} \psi_p - \psi_w\right) \frac{\kappa t^0}{F z_c} \left(\frac{\partial \mu}{\partial c_i}\right) \nabla c_i, \quad (10)$$

We proposed four types of electrode structure, i.e. the random type, the single-gradient type and two dual-gradient type (ST-dual-gradient and BT-dual-gradient), as shown in Fig.S1. The size of the electrode is set to 150 $\mu$m $\times$ 50 $\mu$m $\times$ 50 $\mu$m. It should be pointed out that the overall porosity and total reaction area for these type electrodes are set to nearly the same value, respectively. Thus, we can reveal the effects of the spatial distribution of the porosity and particles size along the longitude direction on the charging performance. The material parameters used in the model are listed as below. The anisotropy diffusion coefficient $D_a$, $D_b$ and $D_c$ are set to $3\times10^{-12}$ m$^2$/s, $3\times10^{-12}$ m$^2$/s and $3\times10^{-14}$ m$^2$/s, respectively. The diffusion coefficient of the electrolyte ($D_i$) is assumed to be isotropic and concentration independent, which is chosen as $1.5\times10^{-10}$ m$^2$/s. The conductivity of the electrolyte ($\kappa$) and the transference number of lithium ion ($t^0$) are also simplified as constant and estimated as 1 S/m and 0.4, respectively(37). The maximum concentration in the particle ($c_{s,max}$) is 31.37 mol/L, and the
initial concentration in the electrolyte is 1 mol/L, respectively\(38\). The initial concentration in the particle \((c_s,0)\) is set as 0.02\(c_{s,\text{max}}\). The reaction rate constant at the particle-electrolyte interface \(k_0\) and the exchange current density at the Li-electrolyte interface \(i_0\) are chosen as 1.76\(\times 10^{-11}\) m\(^{2.5}\)/s/mol\(^{0.5}\) and 2 mA/cm\(^2\), respectively\(39\). The symmetric parameter \((\alpha)\) is generally chosen as 0.5, and the charge of lithium ions \((z_+\) equals to +1. The Faraday constant \((F)\), the ideal gas constant \((R)\) and temperature \((T)\) are 96485 C/mol, 8.314J/mol/K and 298K, respectively. The open circuit potential as a function of the state of charge is obtained from the reference\(39\). In order to numerically solve the above governing equations, the following normalized quantities are introduced. \(c_s^* = c_s/c_{s,\text{max}}, \quad c_l^* = c_l/c_{l,0}, \quad t^* = t D_l / l_0^2, \quad j_n^* = j_n / (c_{l,0} D_l), \quad \phi_l^* = \phi_l / U_m, \quad \mu^* = \mu / (RT)\). Here, \(l_0\) is length of the mesh grid, which equals to 0.25\(\mu\)m. \(U_m\) is the maximum value of the open circuit potential in our simulation. Further, to identify the effect of dual-gradient structure on the charging rate performance, a voxel extracted from the reconstructed 3D graphite electrode is served as the inputted configuration in the particle level model. The same material parameters and normalizing process mentioned above are used in the 3D simulation. Numerical calculation are conducted by MATLAB codes, in which fast Fourier transform algorithm is used to solve the governing equations (Eqs. (8)-(10)). Due to the introduction of the virtual side walls, periodic boundary conditions of the computation box are satisfied. We use at least 4 mesh grids to distinguish the smoothed interface \((0<\Psi_{p}<1, \ 0<\Psi_{w}<1)\) and small dimensionless time step \((1\times 10^{-4})\) to guarantee the numerical accuracy when employing the smoothed boundary method\(36\). The mesh grids are 200\(l_0\)\(\times\)200\(l_0\)\(\times\)600\(l_0\) for 3D simulations. The calculation will be terminated when simulated potential drops below the cutoff voltage (0V). It should be pointed out that only particles and electrolyte are contained the simulation results (as shown in Fig. 1 and Fig. 2), the virtual frame introduced for calculation is not included.
Fig. S1. Model illustration of the random type, the single-gradient type and the dual-gradient type electrode structure and simulated charging process in different electrode structures. (A) Illustration of the representative volume in the electrode. (B) Spatial distribution of the electrode particles with different sizes and porosity in different electrode models. (C) The porosity distribution and (D) the particle size distribution along the thickness direction, respectively. L indicates the distance to the bottom of electrode along the thickness direction. The electrode is evenly divided into five parts along the thickness direction and the average porosity of each part is calculated. (E) Simulated voltage versus state of charge (SOC) curves of different type electrodes under the current density of 3.5 mA/cm². The values of overpotential (ΔV) are calculated by taking the potential difference between the cut-off voltage at fast rates versus corresponding Li⁺ potential at open circuit. The potential at open circuit is as close as possible to the thermodynamic equilibrium. (F) Simulated charging process in BT-dual-gradient type electrode. (G) The ΔV at different charging current densities.
Fig. S2. The preparation illustration of the dual gradient electrode and the corresponding characterizations of the slurry, CuNWs, G@Cu particles. (A) The formation schematic of dual gradient structure in the G@Cu-CuNWs anode. Inset is the optical photograph of the prepared free-standing G@Cu-CuNWs electrode. (B) The viscosity curves of different slurries for the electrode preparation, indicating much lower viscosity of our G@Cu-CuNWs slurry in comparison to traditional graphite anode slurry. (C-E) SEM image of CuNWs (C), graphite particles (D) and G@Cu particles (E), respectively. (F) SEM image and corresponding elementary mapping (carbon and copper) of the G@Cu particle.
Fig. S3. The characterization of G@Cu-CuNWs membrane before calendering. (A, B) The top (A) and bottom surface (B) SEM image of G@Cu-CuNWs membrane, respectively. (C) The particle size distribution of G@Cu particles in the G@Cu-CuNWs electrode along the thickness direction. (D) Powder X-ray diffraction patterns of the graphite and G@Cu particles. (E) Thermal gravimetric analysis (TGA) curves of the graphite particles and G@Cu-CuNWs membrane. The calculated Cu content of G@Cu is 14.7%. After the heating process in TGA test, the graphite was burnt out and Cu was oxidized to copper oxide. According to the left content of copper oxide, the Cu content in the G@Cu-CuNWs can be calculated. (F) The cross-sectional SEM image of the G@Cu-CuNWs membrane after annealing at 450 °C for 2 hours under H₂/Ar (5%/95%) atmosphere. (G) The corresponding magnified SEM image of G@Cu-CuNWs membrane. (H, I) Optical photograph of the areal resistance test of commercial graphite anode (H) and G@Cu-CuNWs anode (I). The M-3 Mini type four-probe tester shows that the areal resistance of the G@Cu-CuNWs anode (65.0 Ω) is lower than that of commercial graphite anode (87.9 Ω).
Fig. S4. Roll pressing of the electrode. (A) Optical photograph of the thick traditional graphite electrode on the copper current collectors after the roll pressing. (B) Optical photograph of current collector free thick graphite anode. (C) Optical photograph of the thick G@Cu-CuNWs anode after the roll pressing. (D-F) The cross-sectional SEM image of the G@Cu-CuNWs electrode rolled by 280 μm (D), 230 μm (E), 180 μm (F) spacing, respectively. The thickness of the electrode was measured as 311 μm (D), 243 μm (E) and 198 μm (F), respectively. (G) The corresponding curve of electrode thickness versus compaction density.
Fig. S5. Electrochemical performance comparison of thick G@Cu-CuNWs and graphite anodes. (A) Initial discharge/charge voltage profiles of the G@Cu-CuNWs and graphite anode in the half cell at the rate of 0.1 C (1 C=370 mA g$^{-1}$). (B) The charge/discharge voltage profiles of the thick G@Cu-CuNWs and graphite anode at different rates. (C) CV curves of the thick G@Cu-CuNWs and graphite anode in the first four cycles. Scan rate: 0.1 mV s$^{-1}$. The reduced polarization and enhanced peak current indicate the excellent reaction kinetics in our dual-gradient structural anode. (D) The cycling performance of the G@Cu-CuNWs and graphite anode over 50 cycles at 0.1 C. (E) The curves of charge voltage versus state of charge (SOC) in the symmetric cell at different charging rates. (F) The rate performance comparison in the symmetric cell.
Fig. S6. 3D reconstruction of the G@Cu-CuNWs and random graphite anode. a, b, Three-dimensional volume rendering of the G@Cu-CuNWs (A) and graphite (B) anode, respectively. A multi-level image threshold method was employed to distinguish the void space (gray scale intensity 0) from other solid materials (gray scale intensity 255). For visual depiction, the binary images are recolored with blue-green (solid materials) and gray (pores). (C, D) The longitudinal cross-sections of the G@Cu-CuNWs (C) and graphite (D) anode, respectively. (E, F) The pore structure extraction from the inside electrode of G@Cu-CuNWs (E) and graphite (F), respectively.
Fig. S7. The porosity and calculated Li$^+$ ion concentration distribution comparison of G@Cu-CuNWs and graphite anode along the thickness direction. (A) The porosity distribution analysis along the thickness direction in the G@Cu-CuNWs and graphite anode. (B) The calculated Li$^+$ ion concentration distribution in the dual gradient G@Cu-CuNWs and random graphite anode along the thickness direction.
Fig. S8. Electrochemical performance evaluation of 70 μm thick G@Cu-CuNWs and graphite anode in the half cell. (A) The rate performance of the G@Cu-CuNWs and graphite anode in the half cell using the Li foil as the counter electrode. (B) The cycling performance of the G@Cu-CuNWs and graphite anode at 1 C. (C, D) Voltage profiles (C) during charging process of the G@Cu-CuNWs and graphite anode with increasing the charging rate from 0.5 to 5 C and the corresponding overpotential comparison. (D). (E) The plots of SOC at galvanostatic state versus the cycle number of the G@Cu-CuNWs and graphite anode with the rate increasing from 1 to 5 C. (F) The comparison of SOC at galvanostatic stage of the G@Cu-CuNWs and graphite anode at different rates.
Fig. S9. The constant current charging ratio of G@Cu-CuNWs electrode with other types of gradient structure at 1C.
Fig. S10. Electrochemical performance evaluation of the G@Cu-CuNWs and random graphite anode in the full cell coupled with the LCO cathode. (A) The first charge/discharge voltage profiles of the G@Cu-CuNWs||LCO and graphite||LCO full cells. (B, C) Cycling performance of the full cells in the voltage range from 3 to 4.3 V for 50 cycles under 1 C (B) and 3 C (C) (1 C = 370 mA g\(^{-1}\)), respectively. The discharging rate was fixed at 0.5 C in all cycling tests. (D, E) The curves of voltage and SOC versus the charge time at the rate of 3 C (D) and 6 C (E), respectively. The required time for any SOC can be extracted from these curves.
Fig. S11. The electrochemical performance comparison of the random structured G@Cu-CuNWs and dual gradient structured G@Cu-CuNWs anode. (A-C) The comparison of electrochemical performance in the symmetric cell. (A, B) The charge voltage profiles at the different charging rate of 3 C (A) and 6 C (B), respectively. (C) The corresponding histogram of SOC in galvanostatic charging process at different C-rate. (D-F) The comparison of electrochemical performance in the full cell. (D,E) The charge voltage profiles at the different charging rate of 3 C (D) and 6 C (E), respectively. (F) The corresponding histogram of SOC in the galvanostatic charging process at the different C-rate.
Fig. S12. Stress–strain curves of G@Cu-CuNWs and Graphite anode without Cu foil.
Table S1. The mass ratio of graphite in the electrode

| Samples            | Mass of current collector (mg cm\(^{-2}\)) | Mass of electrode (mg cm\(^{-2}\)) | Electrode composition (wt.%): (AM\(^a\): BM\(^b\):CM\(^c\)) | Mass ratio of graphite in the electrode (wt.%) | Copper content in the electrode (wt.%) |
|--------------------|------------------------------------------|-----------------------------------|---------------------------------------------------------------|-----------------------------------------------|--------------------------------------|
| Commercial graphite| 8.36                                     | 8.5×2 (Two sides)                 | 96.2:2:1.8                                                   | 64.36                                        | 33.1                                 |
| G@Cu-CuNWs         | 0                                        | 9.8                               | 85.87:0:14.13                                               | 85.87                                        | 14.13                                |

\(^a\) Active material, \(^b\) Binder material, \(^c\) Conductive agent.
Table S2. Capacity comparison of various graphite anodes at different charging rates.

| Mass loading (mg cm$^{-2}$) | 0.2 C $^{SC^a}$ | 0.5 C $^{AC^b}$ | 1 C $^{SC^c}$ | 2 C $^{AC^d}$ | 3 C $^{SC^e}$ | 6 C $^{AC^f}$ | Ref. |
|-----------------------------|-----------------|-----------------|---------------|---------------|---------------|---------------|------|
| Commercial graphite         | 12.1            | 269             | 3.25          | 184           | 2.20          | 84            | 1.01 | 21             | 0.25 | 9             | 0.11 | ---          | 12   |
| a Si nanolayer & EA$^c$ graphite | 7.1             | ---             | ---           | 420           | 2.98          | 335           | 2.38 | 187            | 1.33 | 99            | 0.70 | ---          | 12   |
| Magnetically aligned graphite | 5               | 350             | 1.75          | 284           | 1.42          | 144           | 0.72 | 66             | 0.33 | ---          | ---  | ---          | 9    |
| Graphite with staged porosity | 12.8            | 219             | 2.80          | 91            | 1.17          | ---           | ---  | ---            | ---  | ---          | ---  | 24           |
| KOH-etched graphite         | 5               | 316             | 1.58          | 214           | 1.07          | 140           | 0.62 | 50             | 0.25 | ---          | ---  | ---          | 30   |
| G@Cu-CuNWs                  | 10.3            | 310             | 3.19          | 310           | 3.19          | 301           | 3.10 | 264            | 2.72 | ---          | 193  | 1.99 Our work |

a. Specific capacity, b. Areal capacity, c. Edge-plane-activated
Table S3. The calculation of volumetric energy density of LCO-graphite full cell.

**Volumetric energy density**

\[
\text{Volumetric energy density} = \text{(Areal capacity)} \times \text{(Average voltage)}
\]

**Total thickness**

**Full-cell electrode information**

|                  | LCO    | Graphite | G@Cu-CuNWs |
|------------------|--------|----------|------------|
| Mass loading level (mg cm\(^{-2}\)) | 22.7   | 10.3     | 10.3       |
| Electrode density (g cm\(^{-3}\))    | 3.6    | 1.39     | 1.42       |
| Electrode thickness (µm)               | 63     | 74       | 72         |

**Thickness (µm) (current collector and separator)**

|                                  | Graphite based Full cell | G@Cu-CuNWs based Full cell |
|----------------------------------|--------------------------|-----------------------------|
| 34                               |                          | 24                          |

**Full-cell information**

**Charging rate of 3 C**

|                  | LCO  | Graphite | G@Cu-CuNWs |
|------------------|------|----------|------------|
| Areal capacity (mAh cm\(^{-3}\)) | ---  | 2.98     | 3.04       |
| Average voltage (V)                | ---  | 3.73     | 3.84       |

**Charging rate of 6 C**

|                  | LCO  | Graphite | G@Cu-CuNWs |
|------------------|------|----------|------------|
| Areal capacity (mAh cm\(^{-3}\)) | ---  | 2.52     | 2.95       |
| Average voltage (V)                | ---  | 3.71     | 3.78       |

**Energy density calculation**

|                  | Graphite | G@Cu-CuNWs |
|------------------|----------|------------|
| **Applied C-rate** |          |            |
| 3 C              | 650 Wh L\(^{-1}\) | 734 Wh L\(^{-1}\) |
| 6 C              | 551 Wh L\(^{-1}\) | 701 Wh L\(^{-1}\) |