

# Scalable synthesis of graphitic aggregates for high-rate battery anode

Received: 1 January 2024

Accepted: 16 March 2026

Published online: 17 April 2026

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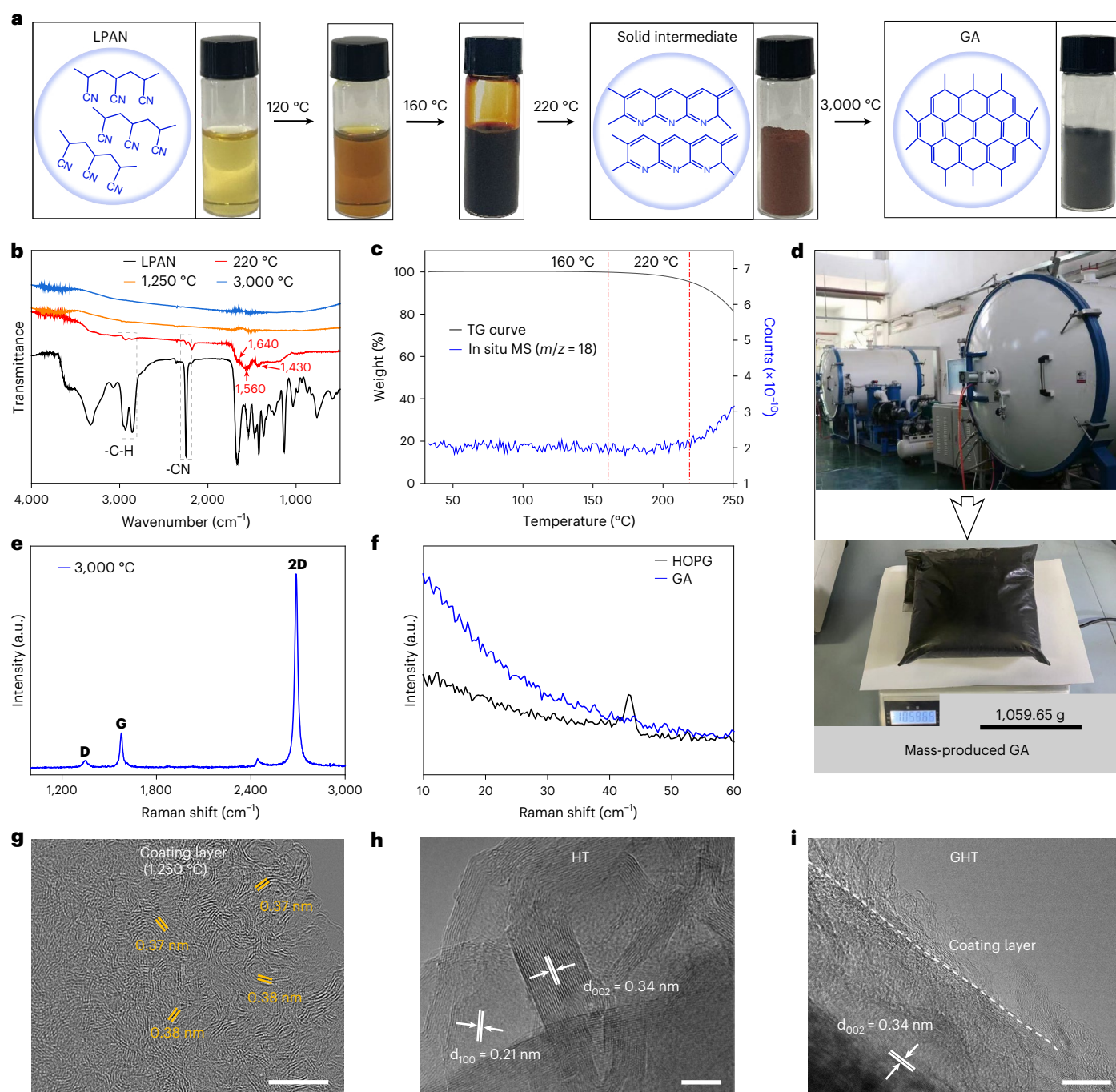
Graphite anodes remain the backbone of lithium-ion batteries, yet their utility is limited by sluggish lithium-ion transport kinetics under extreme conditions. Here we present a scalable method to synthesize graphitic aggregates using liquid-processable, polyacrylonitrile-based precursors, achieving a conformal graphite coating fully compatible with existing industrial workflows. This strategy effectively bypasses high-rate durability bottlenecks by tuning lithium-carbon interactions at the interface. Our findings establish liquid-processable polymer precursors as a high-throughput, manufacturable pathway towards the next generation of high-performance, sustainable energy storage.

For over three decades, graphite has remained the cornerstone of lithium-ion (Li-ion) battery anodes, sustained by its natural abundance, low cost and inherent operational safety<sup>1–4</sup>. Despite this dominance, its performance under extreme conditions—specifically, high-rate charging and sub-zero temperatures—presents a fundamental barrier to meeting modern energy storage demands<sup>5–7</sup>. These limitations are increasingly traced to sluggish Li-ion transport across the electrolyte-graphite interface, where the interplay between interfacial electronic structure and solid electrolyte interphase (SEI) formation remains tightly coupled<sup>8–12</sup>. While diverse strategies have emerged to engineer this interface, they frequently rely on complex, low-throughput synthesis or materials incompatible with large-scale industrial manufacturing<sup>13–16</sup>. Bridging this gap through scalable carbon architectures and synthesis pathways that precisely regulate graphite interfaces thus remains a critical challenge for the future of sustainable battery technologies.

Polymer-derived carbons represent a compelling, yet under-utilized, pathway towards scalable graphitic architectures. Notably, liquid-processable precursors facilitate uniform interfacial coating and batch-level processing, ensuring full compatibility with standard slurry-based electrode fabrication.

Here we report a robust synthesis involving polymerization and controlled oxidation of linear polyacrylonitrile-based molecules into ladder-type precursors, followed by high-temperature carbonization. This approach yields graphitic aggregates (GAs) characterized by a unique combination of short-range graphitic order and long-range structural disorder, synthesized in bulk under industrially relevant conditions. The fluidic nature of the precursor ensures a conformal coating on commercial graphite particles, bridging the gap between advanced materials design and established manufacturing workflows. As the interfacial electronic structure of graphite is critical to the kinetics of Li-ion transport, we employ the GA coating as an interfacial regulator

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**Fig. 1 | Characterizations of GA and modified anodes.** **a**, Scalable GA synthesis route, including low-temperature pre-oxidation and high-temperature carbonization. **b**, Fourier-transform infrared spectra of LPAN, pre-oxidation at 220 °C, solid-state intermediate after calcined at 1,250 °C and the final GA calcined at 3,000 °C. **c**, Thermogravimetric and mass-spectrometric results under  $O_2$  atmosphere, showing the thermogravimetric (TG) curve of LPAN and

the mass-spectrometric (MS) signal of  $m/z = 18$ , which represents  $H_2O$ . **d**, Scalable GA production equipment. **e, f**, Raman spectra of the prepared GA under different frequencies (high frequency, **e**; low frequency, **f**). HOPG, highly oriented pyrolytic graphite. **g**, Transmission electron microscopy image of the LPAN calcined at 1,250 °C. **h, i**, Transmission electron microscopy images of HT (**h**) and GHT (**i**). Scale bars, 10 nm.

rather than as a conventional bulk conductive additive, enabling targeted control over lithium–carbon interactions and SEI formation. Consequently, GA-modified graphite anodes demonstrate robust high-rate durability in practical formats, retaining 87.9% capacity after 1,000 cycles at 1 C and 83.9% after 500 cycles at 3 C within 18650-type cylindrical cells, all while maintaining superior low-temperature performance.

A sustainable, scalable and cost-effective strategy—termed the linear–ladder–planar method—was developed to produce GA. As illustrated in Fig. 1a, the liquid polyacrylonitrile (LPAN) with linear

structure first underwent cyclization to form a solid intermediate at 220 °C. Fourier-transform infrared spectroscopy (Fig. 1b) shows the near-complete disappearance of absorption bands for nitrile groups and carbon–hydrogen vibrations, while three new bands emerge at 1,640, 1,560 and 1,430  $cm^{-1}$ , corresponding to a conjugated aromatic framework. Thermogravimetric and mass-spectrometric analysis (Fig. 1c) revealed minimal weight loss below 160 °C, along with a distinct colour change in LPAN, implying that a condensation reaction occurred. For LPAN, where cyano groups are the only unsaturated

functional groups, it can be inferred that the condensation of cyano groups forms hexatomic rings. A slight weight loss of between 160 °C and 220 °C, together with an increased water signal ( $m/z = 18$ ), demonstrates that cyano group condensation is followed by dehydrogenation. Dehydrogenation of the six-membered ring structure gives rise to a conjugated structure, thereby facilitating the formation of a fused ring system (ladder structure in Fig. 1a). The red solid intermediate shown in Fig. 1a was gradually heated up to 3,000 °C under an inert atmosphere to enhance graphitization, producing a thermodynamically stable planar structure. The method achieves a production capacity of ~20 kg per batch (Fig. 1d). Raman spectroscopy of the 3,000 °C product (Fig. 1e) shows the strongest 2D peak to date<sup>17</sup>, with minimal D-band intensity and low oxygen content (Supplementary Fig. 1), indicating a high degree of graphitization, extensive  $sp^2$  plane and few defects. Low-frequency Raman spectroscopy of highly oriented pyrolytic graphite reveals a characteristic shear mode at 43.4  $\text{cm}^{-1}$  (Fig. 1f). By contrast, our sample exhibits only a near-zero wavenumber inelastic scattering signal, attributed to low-energy excitations or free carriers. This observation aligns with the low-frequency Raman spectra under cross-polarization conditions (Supplementary Fig. 2), indicating the absence of the shear mode in our sample, which originates from a weak  $sp^2$  interlayer interaction. As expected, the transmission electron microscopy images and X-ray diffraction pattern provided in Supplementary Figs. 3 and 4 reveal a graphite-like structure with an expanded interlayer spacing greater than the typical 0.34 nm, which is likely due to weak interlayer interaction. Based on these observations, the sample is identified as GAs. Moreover, the weak interlayer interaction in GAs allows for easy exfoliation into fewer layers of graphene sheets—typically fewer than five layers—using liquid nitrogen and ethyl alcohol, as shown in Supplementary Fig. 5. A gate-to-gate life cycle assessment demonstrated that our linear–ladder–planar pathway, when powered by renewable energy sources, maintains a low ecological footprint (Supplementary Fig. 6 and Supplementary Tables 1 and 2).

Beyond calcining the solid intermediate at 3,000 °C to form low-defect, highly crystalline GA, we also observed the emergence of short-range-ordered GA at 1,250 °C. These aggregates exhibited early-stage graphitization (Supplementary Figs. 7 and 8) and weak interlayer interactions with expanded interlayer spacing (Fig. 1g). Leveraging the unique architecture of short-range-ordered GA and the processability of the LPAN precursor, we modified the graphite anode via the linear–ladder–planar method by blending commercial graphite (high-temperature needle coke, named as HT) with LPAN, followed by pre-oxidation at 220 °C and graphitization at 1,250 °C, yielding a GA-modified HT material named GHT. GHT maintained morphological features like HT, but with a reduced active surface area (Supplementary Fig. 9). Transmission electron microscopy images in Fig. 1h and Supplementary Fig. 10 revealed lattice spacings of 0.34 nm and 0.21 nm for HT, corresponding to the (002) and (100) planes of graphite<sup>18</sup>, respectively. In contrast, GHT displayed a uniform coating structure (Fig. 1i and Supplementary Fig. 11) that maintained electron conductivity (Supplementary Fig. 12).

The electrochemical performance of GHT was first evaluated in coin cells. As shown in Supplementary Fig. 13a, GHT delivers a higher initial charge capacity (350.7  $\text{mAh g}^{-1}$ ) and Coulombic efficiency (96.27%) compared to HT (337.4  $\text{mAh g}^{-1}$  and 93.54%). GHT also displays smaller polarization in its delithiation and lithiation peaks (Supplementary Fig. 13b) and improved cycling stability, retaining 91.71% of its capacity after 200 cycles versus 79.52% for HT (Supplementary Fig. 13c). Voltage profiles further confirm that GHT-based cells maintain both capacity and working voltage during long-term cycling (Supplementary Fig. 13d). Supplementary Fig. 14 depicts that lithium deposition was not observed on the GHT surface, while serious lithium deposition was observed on HT. Rate capability was assessed using two protocols after formation cycling at 0.1 C. Under constant-charge conditions, the GHT-based cell exhibited higher

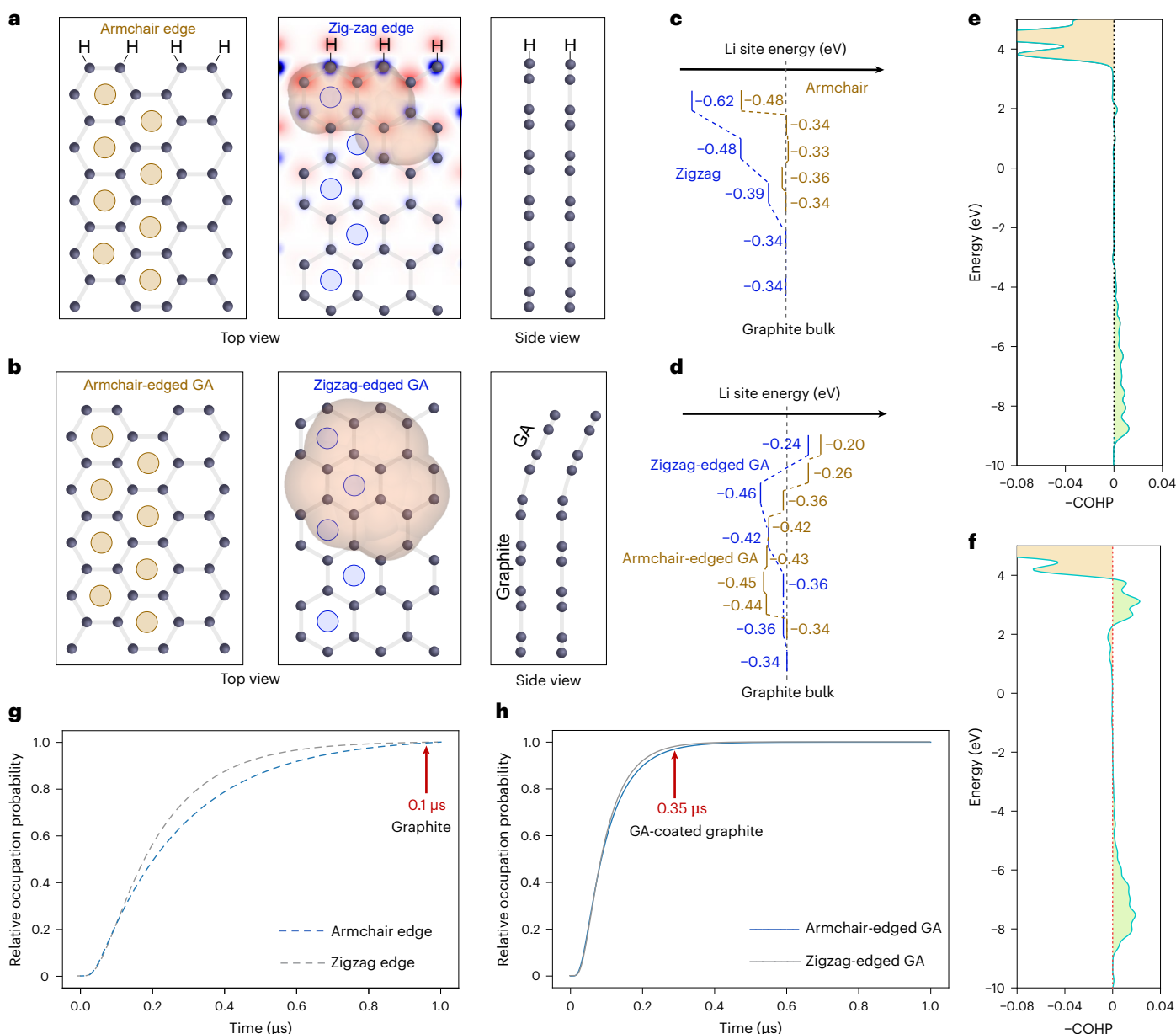
capacities at all rates, whereas the HT-based cell showed rapid capacity decay above 1 C (Supplementary Fig. 13e). At 5 C, the GHT-based cell retained 76.22% of its 0.1 C capacity, notably surpassing the HT cell's 47.45% capacity. With varying charge rates and a constant discharge rate, the GHT-based cell again demonstrated superior rate performance (Supplementary Fig. 13f). These results indicate faster  $\text{Li}^+$  intercalation kinetics for GHT, which helps explain its superior cycling stability. Post-cycling analysis reveals a uniform, stable SEI layer on GHT and suppressed lithium deposition, in contrast to the non-uniform SEI and severe decomposition on HT (Supplementary Fig. 15a–c). Electrochemical impedance spectroscopy confirms faster charge-transfer kinetics at the GHT interface (Supplementary Fig. 15d–g). These improvements are attributed to the GA's coating, which enhances interfacial ion transport and structural stability.

Building on GHT's advantages, we assembled and tested 18,650 cylindrical cells (the results are depicted in Supplementary Figs. 16–18). After 1,000 cycles, the GHT|| $\text{LiNi}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2}\text{O}_2$  cell retained 87.9% of its initial capacity, and even at a 3 C discharge rate, it maintained 83.9%. The cell also showed excellent low-temperature performance, with capacity retentions of 93.2% at 2 C and 92.2% at 3 C when tested at  $-10$  °C. These results highlight the superior cycle life, high-rate capability and low-temperature performance of GHT|| $\text{LiNi}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2}\text{O}_2$  cells.

Density functional theory (DFT) calculations were conducted to clarify the enhanced electrochemical performance of GA-coated graphite anodes (Fig. 2). As shown in Fig. 2a, pristine graphite exposes armchair and zigzag edges where SEI preferentially forms<sup>19</sup>, and Li-ion exhibits stronger binding at edge sites than in the bulk. Li-ion binding energies are  $-0.48$  eV at the armchair edge and  $-0.62$  eV at the zigzag edge, both much higher than the bulk value ( $-0.34$  eV). The strong binding at the zigzag edge originates from topological surface states associated with carbon  $p_z$  orbitals, which stabilize Li-ion and extend partially into the bulk. After GA coating, the surface effect is largely weakened. In the armchair-edged GA and zigzag-edged GA heterostructures (Fig. 2b), Li-ion adsorption at the GA sites is relatively weak ( $-0.20$  to  $-0.24$  eV), and the binding energies that occur at the heterostructures ( $-0.43$  to  $-0.46$  eV) are lower than those at the pristine edges (Fig. 2c). Notably, the absence of spin-polarized surface states at the zigzag-edged GA heterostructures reduces Li-ion binding by  $-0.16$  eV compared with pristine zigzag edges (Fig. 2d). Fig. 2e,f illustrates the crystal orbital Hamilton population of Li-ion binding at the zigzag-edged GA and zigzag edge. There is less covalent bonding below the Fermi level at around  $-8$  eV from the Li–C  $\sigma$  bond, and a greater contribution of the anti-bonding component above the Fermi level at  $-4$  eV for zigzag-edged GA heterostructures, indicating a more ionic bonding nature of Li-ion in the zigzag/GA heterostructure by reducing the surface-state interactions, which was also verified by the local density of states and electron locational function (Supplementary Figs. 20 and 21).

The weakened surface effect leads to a shallower potential well and improved Li-ion mobility. Ab initio molecular dynamics simulations show that Li-ion diffusion is confined near pristine zigzag edges (indicated by the red regions in Fig. 2a,b), but becomes much more delocalized in zigzag-edged GA heterostructures for Li-ion mobility. Markov Chain modelling<sup>20</sup> reveals that Li-ion diffusion in both armchair-edged and zigzag-edged GA is about three times faster than in pristine graphite edges (1.0  $\mu\text{s}$  versus 0.35  $\mu\text{s}$ ), as depicted in Fig. 2g,h. This indicates that GA coating effectively suppresses edge effects, facilitates rapid Li-ion transfer from the interface into the graphite bulk, and thereby enhances ion intercalation kinetics and rate performance.

In summary, we demonstrate a scalable, industrially compatible synthesis utilizing LPAN precursors to engineer unique GAs. This approach enables the formation of conformal, interfacial coatings that fundamentally alter the surface topology of graphite anodes. Our findings establish a manufacturable strategy for the next generation of high-rate, long-life energy storage systems.



**Fig. 2 | DFT calculations.** **a**, Structures of armchair-edged and zigzag-edged graphite. **b**, GA-coated armchair and zigzag heterostructures. The red area shows the region of Li-ion mobility in molecular dynamics simulations. **c,d**, Energy profiles of Li-ion adsorption at different sites in **a** (**c**) and **b** (**d**). **e,f**, Crystal orbital Hamilton population (COHP) of Li binding at the interface site in zigzag-edged

GA heterostructure (**e**) and the zigzag edge (**f**). **g,h**, Relative site occupation probability for Li-ion to reach a site about 19 Å distance from the initial adsorption site after being introduced at time zero at the initial adsorption site, including graphite (**g**) and GA-coated graphite (**h**).

## Methods

GAs were synthesized from a LPAN precursor supplied by Shenzhen Eigen-Equation Graphene Technology Co. Ltd. The LPAN was first heated in air at 220 °C for 3 hours, turning from light yellow to dull red, and then solidifying. The resulting solid was ball-milled and then sintered at 3,000 °C for 8 hours under argon to yield a black powder. To prepare a surface-modified graphite anode, commercial graphite (HT, from the same supplier) was mixed with deionized water (1:1) and 7% LPAN under stirring for 4 hours, followed by spray drying at 180 °C. The mixture was calcined at 220 °C for 3 hours and sintered at 1,250 °C for 5 hours under nitrogen. The final product was labelled as GHT.

## Reporting summary

Further information on research design is available in the Nature Portfolio Reporting Summary linked to this article.

## Data availability

All the relevant data are included in the paper and the corresponding Supplementary Information. Source data are provided with this paper.

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## Acknowledgements

We are grateful for the financial support provided by the National Natural Science Foundation of China (nos. 52202269 and 52203303), the Shenzhen Natural Science Foundation (no. 20220810155330003), the Shenzhen Science and Technology Program (CJGJZD20210408092801005), Shenzhen Key Projects of Technological Research (no. JSGG20200925145800001) and the Department of Education of Guangdong Province (no. 2022ZDZX3018).

## Author contributions

J.H. and J.L. conceived the idea for this project. J.H., X.O., Q.Z. and J.L. designed the experiments and tested the physical and electrochemical properties. C.P., D.X. and X. Yu (Chinese Academy of Sciences) conducted the DFT calculations. P.-H.T. and R.M. collected the low-frequency Raman data. X. Yang (Shenzhen University), S.H., W.Y. and W.X. prepared the electrode materials and performed some of the electrochemical properties. P.L., T.H., X. Yang (Shenzhen University), L.L., S.Y., J.P., B.J., X.R. and Y.L. discussed the results and analysed the data. J.H. and S.Y. wrote and revised the paper.

## Competing interests

The authors declare no competing interests.

## Additional information

**Supplementary information** The online version contains supplementary material available at <https://doi.org/10.1038/s41893-026-01813-y>.

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**Peer review information** *Nature Sustainability* thanks Yingying Zhang and the other, anonymous, reviewer(s) for their contribution to the peer review of this work.

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### Software and code

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#### Data collection

The morphologies of the prepared materials and the cycled electrodes were detected by scanning electron microscope (SEM, JSM 7800F, JEOL). Transmission electron microscopy (TEM, JEOL JEM-F200) were applied to characterize the material structure and SEI layer thickness. Other high resolution TEM images were performed on FEI Talos F200X with different voltages. Aberration-corrected STEM (FEI Titan Cubed Themis G2 300) at the Electron Microscopy Center of Shenzhen University was applied to observe the graphitization of the resulting samples. The graphitic aggregates quality was detected by Raman spectra (Renishaw inVia Raman microscope). The low-frequency Raman spectra were acquired using a microconfocal Raman system (Jobin-Yvon HR800). The objective lens employed had a resolution of 100x (0.9NA) and the excitation energy of the He-Ne laser is 1.96 eV. The experiment adopts 1800 gr/mm holographic grating to ensure high spectral resolution ( $\sim 0.35 \text{ cm}^{-1}$ ). The electronic conductivity comparison between the pristine graphite and the coated sample was performed by 4-Point probes measurement system, and the corresponding pressure is 1.26 kN. The surface area of the prepared samples were measured by Multi-point Method (BSD-PS). The SEI compositions were determined by X-ray photoelectron spectroscopy (XPS, Thermo Fisher Scientific) and fitted by Advantage. The binding energy were calibrated according to the C 1s peak at 284.4 eV. Nitrogen absorption-desorption isotherms were evaluated on a BESTCN 3H-2000PS2 instrument, and the specific surface areas were calculated by the multi-point BET equation. TG-MS was determined by a Netzsch STA449 F3 +Quadrupole Mass Spectrometry (QMS403D). FT-IR spectra was recorded by IR Affinity<sup>®</sup>-1 spectrometer (Shimadzu, Düsseldorf, Germany). Total electron yield (TEY) soft x ray absorption spectrum were measured at the 4B9B beamline in Beijing Synchrotron Radiation Facility (BSRF). The base pressure of the analysis chamber was better than  $3 \times 10^{-10}$  Torr. The photon energy was calibrated by Au 4f core level XPS of clean polycrystalline gold foil which is electrically connected to the sample.

#### Data analysis

Powerpoint and origin were used to conduct the data analysis.

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## Ecological, evolutionary & environmental sciences study design

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Study description	Scalable synthesis of graphitic aggregates for high-rate battery anode
Research sample	Graphitic aggregates, high-rate anode
Sampling strategy	<p>Liquid polyacrylonitrile (LPAN) is the precursor of the graphitic aggregates. During heat to 220°C in air, LPAN was firstly pre-oxidized and trapezoidal, which experienced a distinct color changes from light yellow to dark black, and concentration changes from dilute to concentrated solution. Then, the dark product was heated at 220°C for 3h to realize full peroxidation and molecular planarization, and the corresponding color of the product is dull red. Before calcination at 3000°C, the dull red product needs a ball milling operation. To the end, we obtained a black powder after sintering at 3000°C for 8h under Ar atmosphere, which is proved to be the graphitic aggregates. The whole synthesis process goes through the cyclization and planarization processes from the organic linear molecules.</p> <p>The above graphitic aggregates synthesis route was applied to produce surface modified graphite anode. Commercial graphite (Labeled as HT, received from Shenzhen Eigen-Equation Graphene Technology Co., Ltd.) was stirred with deionized water (1:1) and 7% LPAN precursor for 4h before spray dried at 180°C. The obtained mixture was calcinated at 220°C for 3h and then sintered at 1250°C for 5h under N2 atmosphere. The final product is named as GHT. This technology is well established and is being used to produce tonnage-scale GHT anode material.</p>
Data collection	Sample characterization were conducted on scanning electron microscope (SEM, JSM 7800F, JEOL), transmission electron microscopy (TEM, JEOL JEM-F200), Raman spectra (Renishaw inVia Raman microscope), microconfocal Raman system (Jobin-Yvon HR800), 4-Point probes measurement system, X-ray photoelectron spectroscopy (XPS, Thermo Fisher Scientific), BESTCN 3H-2000PS2 instrument, Vienna Ab initio Simulation Package (VASP), ab-initio molecular dynamics (AIMD) simulation.
Timing and spatial scale	Dec. 2021-Dec.2024
Data exclusions	No data were excluded.
Reproducibility	All attempts to repeat the experiment were successful.

Randomization

Blinding

Did the study involve field work?  Yes  No

## Reporting for specific materials, systems and methods

We require information from authors about some types of materials, experimental systems and methods used in many studies. Here, indicate whether each material, system or method listed is relevant to your study. If you are not sure if a list item applies to your research, read the appropriate section before selecting a response.

### Materials & experimental systems

n/a	Involvement in the study
<input checked="" type="checkbox"/>	<input type="checkbox"/> Antibodies
<input checked="" type="checkbox"/>	<input type="checkbox"/> Eukaryotic cell lines
<input checked="" type="checkbox"/>	<input type="checkbox"/> Palaeontology and archaeology
<input checked="" type="checkbox"/>	<input type="checkbox"/> Animals and other organisms
<input checked="" type="checkbox"/>	<input type="checkbox"/> Clinical data
<input checked="" type="checkbox"/>	<input type="checkbox"/> Dual use research of concern

### Methods

n/a	Involvement in the study
<input checked="" type="checkbox"/>	<input type="checkbox"/> ChIP-seq
<input checked="" type="checkbox"/>	<input type="checkbox"/> Flow cytometry
<input checked="" type="checkbox"/>	<input type="checkbox"/> MRI-based neuroimaging