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**We develop a scalable solid-state synthesis for Si<sub>2</sub>Te<sub>3</sub> and its carbon composite (Si<sub>2</sub>Te<sub>3</sub>@C) to address the challenges of silicon-based anodes in lithium-ion batteries (LIBs). The Si<sub>2</sub>Te<sub>3</sub>@C composite exhibits enhanced electrochemical performance, including high reversible capacity, excellent cycling stability, and improved rate capability, positioning it as a promising candidate for high-performance LIB anodes.**

Layered materials, characterized by strong in-plane covalent bonding and weak out-of-plane van der Waals (vdW) interactions, have garnered significant interest due to their unique physical and chemical properties.<sup>1–3</sup> These materials, including graphite, black phosphorus, transition metal chalcogenides (TMCs), and MXenes, allow for the intercalation of foreign atoms or molecules between layers, making them attractive as electrode materials for lithium-ion batteries (LIBs). Notably, graphite serves as a representative anode material due to its reversible Li-ion intercalation mechanism, while other layered materials, such as phosphides, chalcogenides, carbides, and nitrides, are actively studied for their high capacity and structural advantages, positioning them as promising alternatives for next-generation LIB electrodes.<sup>4–7</sup>

Silicon (Si) has been widely recognized as a promising anode material for LIBs due to its abundance, low redox potential (0.2–0.3 V vs. Li<sup>+</sup>/Li), and exceptionally high theoretical capacity (3578 mA h g<sup>−1</sup> for Li<sub>3.75</sub>Si at room temperature).<sup>8</sup> However, Si-based anodes suffer from severe structural degradation caused by 300–400% volume expansion during lithiation/delithiation processes.<sup>9</sup>

## Layered Si<sub>2</sub>Te<sub>3</sub>-based anodes for high-performance Li-ion batteries†

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This dramatic volume change leads to the mechanical fracture of particles, electrical disconnection of active materials, and the formation of dynamic interfaces at the electrode–electrolyte boundary. Such dynamic interfaces not only degrade electrical contact but also promote undesirable side reactions with the electrolyte, resulting in the continuous growth of the solid electrolyte interphase (SEI) layer, ultimately causing capacity fading and poor cycle life.<sup>10</sup> To address these intrinsic limitations of Si anodes, extensive efforts have been devoted to improving their performance through various strategies, including carbon coatings, composite structures, size and morphology control, and the development of Si-alloys and Si-compounds.<sup>11–14</sup> Among these approaches, Si-alloys and Si-compounds have emerged as particularly promising solutions due to their structural stability and distinct Li-storage mechanisms, which effectively mitigate the effects of volume expansion and improve cycling performance.

Recently, Si<sub>2</sub>Te<sub>3</sub>, a two-dimensional (2D) silicon-based chalcogenide, has attracted considerable attention as a high-performance material for energy storage applications. It exhibits excellent environmental stability, a strain-dependent bandgap, and a high Seebeck coefficient, making it highly versatile.<sup>15,16</sup> Its trigonal layered crystal structure with a stoichiometric ratio of Si/Te = 2/3 supports the formation of natural structural defects through the partial absence of Si–Si pairs, resulting in p-type behavior and enhanced light–matter interactions.<sup>17</sup> These features contribute to improved photoelectric conversion efficiency and a broad photoresponse range, making it suitable for optoelectronic devices as well. Furthermore, the layered structure of Si<sub>2</sub>Te<sub>3</sub> provides large interlayer spacing, which facilitates lithium-ion diffusion during lithiation/delithiation processes, addressing the volume expansion issue associated with conventional Si anodes. Its indirect bandgap nature and photoelectric properties further expand its potential applications beyond energy storage, particularly in optoelectronics. Despite its promising structural and electronic properties, the scalable synthesis of Si<sub>2</sub>Te<sub>3</sub> remains a challenge. Therefore, developing simple and cost-effective methods for producing Si<sub>2</sub>Te<sub>3</sub> is crucial to fully realize its potential as a high-performance anode material in LIBs.

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In this study, we present a facile and scalable approach for synthesizing layered  $\text{Si}_2\text{Te}_3$  through a solid-state process that combines sequential ball-milling (BM) and heat-treatment (HT). While  $\text{Si}_2\text{Te}_3$  exhibits a unique atomic arrangement, its electrochemical performance is largely influenced by its intrinsic properties. To overcome its inherent low electrical conductivity, a composite structure incorporating amorphous carbon is developed. The Li-storage properties of this composite are further evaluated, demonstrating its potential as a high-performance Si-based anode for LIBs. Moreover, its Li-storage mechanism is systematically analyzed using *ex situ* X-ray diffraction (XRD) and high-resolution transmission electron microscopy (HR-TEM) based on the  $dQ/dV$  plots. This work provides a comprehensive understanding of the electrochemical behavior of  $\text{Si}_2\text{Te}_3$ -based composites and offers useful design strategies for next-generation LIB anodes.

Silicon–tellurium (Si–Te) compounds are primarily known to form two distinct phases,  $\text{SiTe}_2$  and  $\text{Si}_2\text{Te}_3$ .<sup>17,18</sup> However, the Si–Te phase diagram identifies  $\text{Si}_2\text{Te}_3$  as the only thermodynamically stable compound in this system (Fig. S1, ESI†).  $\text{Si}_2\text{Te}_3$  crystallizes in a hexagonal structure with a  $P\bar{3}1c$  (no. 163) space group and exhibits lattice parameters of  $a = b = 7.4 \text{ \AA}$  and  $c = 13.5 \text{ \AA}$ . It features a layered crystal structure with each layer having a thickness of  $6.7 \text{ \AA}$ . The layers consist of two parallel planes of tellurium atoms, with Si–Si dimers occupying the inter-layer regions.<sup>17</sup> The interlayer spacing in  $\text{Si}_2\text{Te}_3$  is approximately  $3.2 \text{ \AA}$ , which is comparable to well-known layered materials such as graphene ( $\sim 3.4 \text{ \AA}$ ), black phosphorus ( $\sim 3.2 \text{ \AA}$ ), and  $\text{MoS}_2$  ( $\sim 3.2 \text{ \AA}$ ). This relatively large interlayer spacing facilitates rapid diffusion and reversible accommodation of Li-ions, making  $\text{Si}_2\text{Te}_3$  a promising candidate for LIB anode materials.

$\text{Si}_2\text{Te}_3$  was synthesized *via* a solid-state reaction involving sequential BM and HT processes. The XRD patterns of the synthesized  $\text{Si}_2\text{Te}_3$  matched well with those of the hexagonal  $\text{Si}_2\text{Te}_3$  phase (JCPDS #75-0820), confirming that the material was formed homogeneously without impurity phases (Fig. 1a and Fig. S2, ESI†). To further investigate its structural properties, the XRD pattern was refined using the Rietveld method (Fig. 1b). The corresponding lattice parameters— $\chi^2 = 1.01$ ,  $R_p = 15.5\%$ , and  $R_{wp} = 19.1\%$  (Table S1, ESI†)—showed excellent agreement with experimental data, indicating the high crystallinity of the synthesized material. The morphology and particle size distribution of  $\text{Si}_2\text{Te}_3$  were examined using scanning electron microscopy (SEM) and particle size analysis (PSA). As shown in Fig. S3 (ESI†), the average particle size was approximately  $10.01 \mu\text{m}$ , with irregular particle morphologies. To further validate the homogeneous synthesis, Raman spectroscopy and HR-TEM analyses were conducted. The Raman spectra exhibited three distinct peaks at  $139$ ,  $291$ , and  $504 \text{ cm}^{-1}$ , which were clearly distinguishable from those of Si and Te (Fig. S4, ESI†), confirming the formation of the  $\text{Si}_2\text{Te}_3$  phase. Additionally, HR-TEM images (Fig. S5, ESI†) revealed well-developed  $\text{Si}_2\text{Te}_3$  crystallites with clearly visible diffraction patterns (DPs) corresponding to the hexagonal structure. The observed crystal planes, including  $(\bar{1}\bar{1}4)$ ,  $(110)$ ,  $(\bar{1}\bar{1}2)$ , and  $(\bar{1}\bar{1}6)$ , matched the main peaks detected in the XRD patterns, further

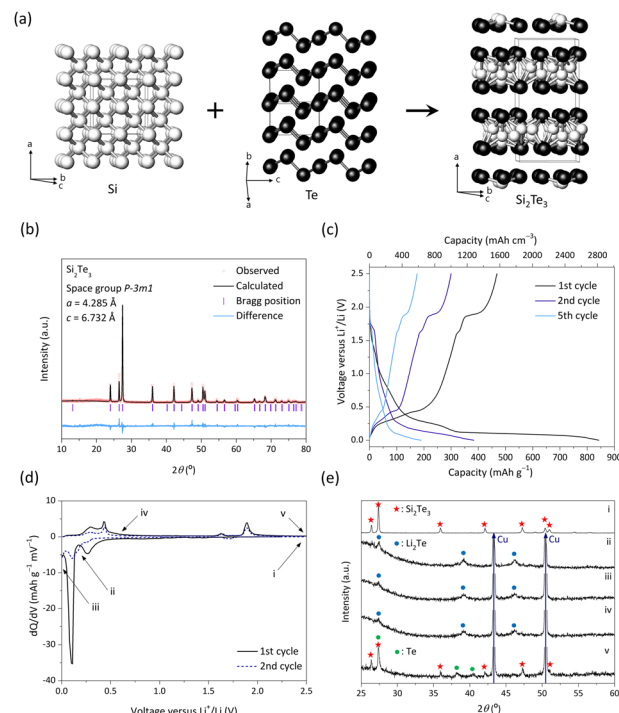


Fig. 1 (a) Crystal structure of layered  $\text{Si}_2\text{Te}_3$ ; (b) Rietveld-refined XRD patterns of  $\text{Si}_2\text{Te}_3$ ; (c) GDC profiles of  $\text{Si}_2\text{Te}_3$  anode at  $100 \text{ mA g}^{-1}$ ; (d)  $dQ/dV$  plots for  $\text{Si}_2\text{Te}_3$ ; (e) *ex situ* XRD patterns of  $\text{Si}_2\text{Te}_3$  anode during first cycle.

verifying the structural integrity and high crystalline quality of the synthesized  $\text{Si}_2\text{Te}_3$ .

To evaluate the electrochemical Li-storage properties of the  $\text{Si}_2\text{Te}_3$  anode, galvanostatic discharge and charge (GDC) profiles were recorded at a current density of  $100 \text{ mA g}^{-1}$  (Fig. 1c). For comparison, Si and Te anodes were also prepared and tested electrochemically using bulk Si and Te powders. The Si anode exhibited a high initial lithiation/delithiation capacity of  $3511/1925 \text{ mA h g}^{-1}$  (Fig. S6, ESI†). However, its capacity rapidly degraded to  $282 \text{ mA h g}^{-1}$  after 10 cycles due to severe volume expansion ( $>300\%$ ) caused by the alloying/dealloying reactions with  $\text{Li}_x\text{Si}$  phases. In contrast, the Te anode demonstrated a lower lithiation/delithiation capacity of  $199/133 \text{ mA h g}^{-1}$  and suffered from poor capacity retention due to its low electrical conductivity. Compared to these materials, the  $\text{Si}_2\text{Te}_3$  anode exhibited an initial lithiation/delithiation capacity of  $843/468 \text{ mA h g}^{-1}$  ( $2816/1563 \text{ mA g cm}^{-3}$ ) with an ICE of  $55.5\%$ . Thus, Li-storage performance of  $\text{Si}_2\text{Te}_3$  is also not sufficient to meet the criteria for high-performance LIB anodes compared with Si-based anodes.

To analyze the Li-storage mechanism of the  $\text{Si}_2\text{Te}_3$  anode, *ex situ* XRD analysis was performed based on the  $dQ/dV$  plot. The  $dQ/dV$  plot exhibited two prominent peaks during the discharge process and three peaks during the charge process (Fig. 1d). *Ex situ* XRD analysis (Fig. 1e) revealed that as the potential decreased from open circuit potential (i) to  $0.17 \text{ V}$  (ii), the  $\text{Si}_2\text{Te}_3$  phase disappeared, and only the  $\text{Li}_2\text{Te}$  phase (JCPDS #77-2147) was detected, confirming the conversion reaction of  $\text{Si}_2\text{Te}_3$  into  $\text{Li}_2\text{Te}$  and amorphous Si. Further analysis



at 0 V (iii) revealed that the  $\text{Li}_2\text{Te}$  phase remained. To verify the formation of the Li-Si phase after full lithiation, the  $dQ/dV$  plots for Si and  $\text{Si}_2\text{Te}_3$  anodes were compared (Fig. S7, ESI<sup>†</sup>).<sup>19</sup> The overlapping peak positions confirmed that the formation of the  $\text{Li}_{3.75}\text{Si}$  phase was consistent in both anodes. Upon charging to 0.6 V (iv), the  $\text{Li}_2\text{Te}$  phase persisted, suggesting partial recombination during the initial charging process. At the fully charged state (2.5 V, v), the  $\text{Si}_2\text{Te}_3$  phase reappeared. However, the Te phase was also detected, indicating that the  $\text{Si}_2\text{Te}_3$  phase was partially recombined, with residual Te and amorphous Si coexisting. These results demonstrate that the  $\text{Si}_2\text{Te}_3$  anode undergoes a reversible conversion reaction, although the presence of residual Te and amorphous Si suggests partial phase reconstruction during the charging process.

To further enhance the Li-reversibility and reactivity of  $\text{Si}_2\text{Te}_3$ , we designed a  $\text{Si}_2\text{Te}_3@\text{C}$  composite by combining the as-synthesized  $\text{Si}_2\text{Te}_3$  with carbon black using a BM process. Carbon-supported composites are well-known to improve ionic and electronic conductivity, accommodate large volume changes, and suppress the agglomeration of nanoscale Li-active materials during repeated cycling.<sup>20,21</sup> The introduction of conductive carbon matrices not only facilitates charge transfer kinetics but also stabilizes the structural integrity of the  $\text{Si}_2\text{Te}_3$  particles, enabling prolonged cycle life and rate performance. The XRD pattern of the  $\text{Si}_2\text{Te}_3@\text{C}$  composite revealed broadened diffraction peaks, indicating a reduction in crystallinity during the composite formation process (Fig. S8, ESI<sup>†</sup>). This suggests that the BM process induced a partial amorphization of the material, which could contribute to enhanced electrochemical properties by providing shorter diffusion paths for Li ions. HR-TEM analysis further confirmed that the synthesized  $\text{Si}_2\text{Te}_3@\text{C}$  composite consists of ultrafine nanocrystallites ( $\sim 5\text{--}7$  nm) uniformly embedded within an amorphous carbon matrix (Fig. 2a–c). The DPs obtained from selected area electron diffraction (SAED) matched well with the hexagonal crystal structure of  $\text{Si}_2\text{Te}_3$ , supporting the structural stability of the nanocrystallites within the composite. Energy-dispersive X-ray spectroscopy (EDS) mapping derived from scanning TEM (STEM) images verified the homogeneous distribution of elemental Si (yellow) and Te (green) throughout the carbon matrix (blue), highlighting the well-mixed composition of the composite. In addition, the average particle size of  $\text{Si}_2\text{Te}_3@\text{C}$  was approximately 6.8  $\mu\text{m}$ , with irregular particle morphologies (Fig. S9, ESI<sup>†</sup>).

Fig. 2d shows the GDC profiles of the  $\text{Si}_2\text{Te}_3@\text{C}$  anode at a current density of 100  $\text{mA g}^{-1}$ . The initial lithiation/delithiation capacities were 816  $\text{mA h g}^{-1}$  (1746  $\text{mA h cm}^{-3}$ ) and 559  $\text{mA h g}^{-1}$  (1196  $\text{mA h cm}^{-3}$ ), respectively. Considering the first irreversible capacity of the carbon component (30 wt%, 110  $\text{mA h g}^{-1}$ , Fig. S10, ESI<sup>†</sup>), the  $\text{Si}_2\text{Te}_3@\text{C}$  composite exhibited significantly improved Li reversibility and reactivity compared to the pristine  $\text{Si}_2\text{Te}_3$  anode. In addition, electrochemical impedance spectroscopy (EIS) and the corresponding Nyquist plots were analyzed (Fig. S11, ESI<sup>†</sup>). The charge transfer resistance ( $R_{ct}$ ) of the  $\text{Si}_2\text{Te}_3@\text{C}$  nanocomposite anode was measured as 34.9  $\Omega$ , which is significantly lower than that of the pristine  $\text{Si}_2\text{Te}_3$  anode (59.7  $\Omega$ ). Furthermore, the nanocomposite exhibited a higher Li-ion diffusion coefficient of  $1.23 \times 10^{-15} \text{ cm}^2 \text{ s}^{-1}$ , compared to  $1.11 \times 10^{-14} \text{ cm}^2 \text{ s}^{-1}$  for the

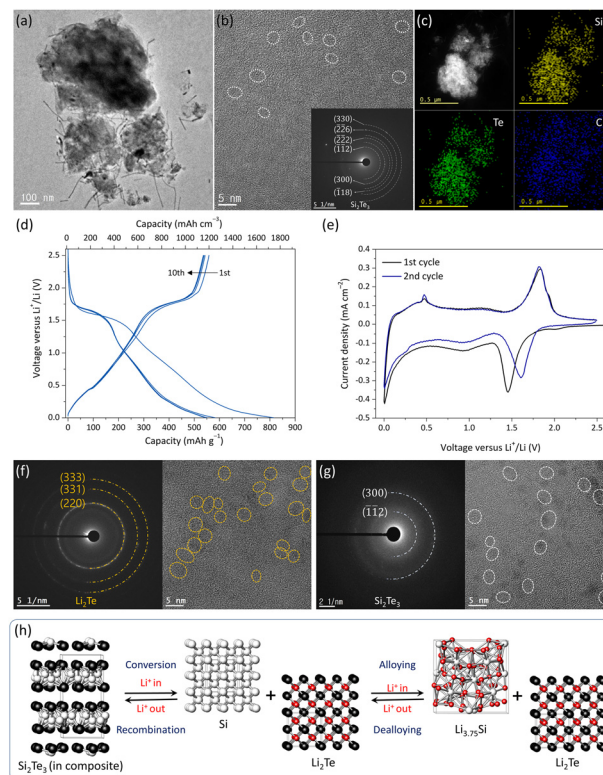


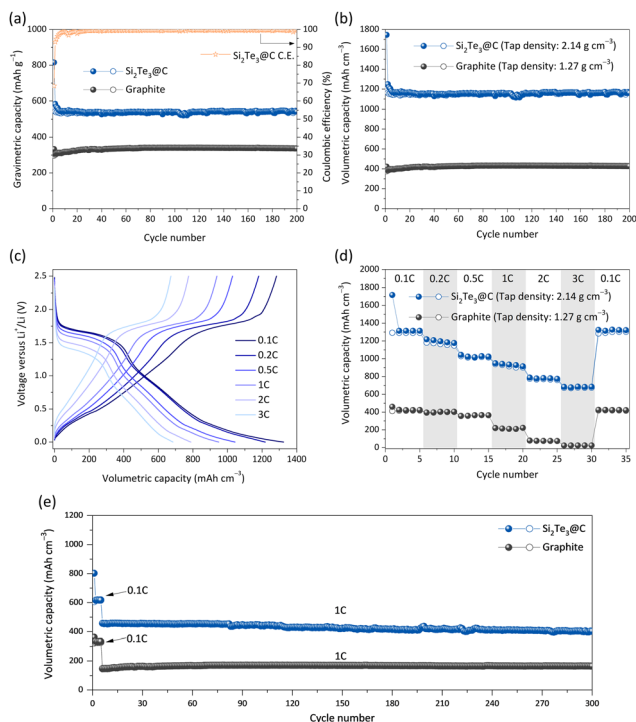
Fig. 2 (a) Bright field TEM image, (b) HR-TEM image with corresponding DPs and, (c) STEM image with corresponding EDS mapping images of  $\text{Si}_2\text{Te}_3@\text{C}$ ; (d) GDC profiles of  $\text{Si}_2\text{Te}_3@\text{C}$  at 100  $\text{mA g}^{-1}$ ; (e) CV plots of  $\text{Si}_2\text{Te}_3@\text{C}$ ; *ex situ* HR-TEM images with corresponding DPs of  $\text{Si}_2\text{Te}_3@\text{C}$  after (f) full discharged potential (0 V) and (g) full charged potential (2.5 V); (h) schematic of the crystallographic phase change mechanism of  $\text{Si}_2\text{Te}_3@\text{C}$ .

pristine  $\text{Si}_2\text{Te}_3$ , as calculated from the  $Z_{re}$ -angular frequency ( $\omega^{-1/2}$ ) relationship in the low-frequency region.

To investigate the Li-storage mechanism, *ex situ* XRD analysis was performed at selected potentials based on the  $dQ/dV$  plot and cyclic voltammetry (CV) results. The CV plot displayed two distinct peaks during Li insertion/extraction, suggesting a conversion-type reaction mechanism (Fig. 2e). Although XRD patterns were collected at five cut-off potential points, all patterns exhibited an amorphous nature, likely due to the low crystallinity of  $\text{Si}_2\text{Te}_3$  within the composite (Fig. S12, ESI<sup>†</sup>). Thus, to investigate the structural changes of  $\text{Si}_2\text{Te}_3@\text{C}$ , *ex situ* HR-TEM analysis is performed and the results are shown in Fig. 2f and g. At the fully discharged potential (0 V), well-dispersed nanocrystallites of  $\text{Li}_2\text{Te}$  (5–8 nm) and amorphous  $\text{Li}_{3.75}\text{Si}$  phases were observed, confirming that  $\text{Si}_2\text{Te}_3$  was fully converted into these phases during the discharge process (Fig. 2f). In contrast, at the fully charged potential (2.5 V), only the  $\text{Si}_2\text{Te}_3$  phase was detected, indicating that  $\text{Li}_{3.75}\text{Si}$  and  $\text{Li}_2\text{Te}$  recombined to reform  $\text{Si}_2\text{Te}_3$  with 5–7 nm nanocrystallites during the charging process (Fig. 2g and h).

These findings confirm that the  $\text{Si}_2\text{Te}_3@\text{C}$  nanocomposite undergoes a reversible conversion reaction, where the Li-storage process is facilitated by the conducting carbon matrix. The carbon matrix not only improves electronic conductivity but also provides structural stability, effectively buffering the





**Fig. 3** (a) Gravimetric and (b) volumetric capacities vs. cycle number for  $\text{Si}_2\text{Te}_3\text{@C}$  and graphite anodes at  $100 \text{ mA g}^{-1}$ ; (c) GDC profiles of  $\text{Si}_2\text{Te}_3\text{@C}$  anode at different C-rates (1C:  $550 \text{ mA g}^{-1}$ ); (d) rate capability of  $\text{Si}_2\text{Te}_3\text{@C}$  (1C:  $550 \text{ mA g}^{-1}$ ) and graphite (1C:  $300 \text{ mA g}^{-1}$ ) anodes; (e) cycling performance of  $\text{Si}_2\text{Te}_3\text{@C}$  (1C:  $550 \text{ mA g}^{-1}$ ) and graphite (1C:  $300 \text{ mA g}^{-1}$ ) anodes at 1C-rate.

volume changes associated with the lithiation/delithiation processes. In addition, the formation of the  $\text{Li}_2\text{Te}$  phase during lithiation, derived from  $\text{Si}_2\text{Te}_3$  in the composite, could contribute to the electrochemical performance of LIB anodes.<sup>22</sup>

Fig. 3a and b illustrate the electrochemical performance of the  $\text{Si}_2\text{Te}_3\text{@C}$  composite anode compared with a commercial graphite anode at a cycling rate of  $100 \text{ mA g}^{-1}$ . The  $\text{Si}_2\text{Te}_3\text{@C}$  composite exhibited a highly reversible gravimetric/volumetric capacity of  $816/559 \text{ mA h g}^{-1}$  and  $1746/1196 \text{ mA h cm}^{-3}$ , respectively, which are higher than those of the graphite anode ( $332/296 \text{ mA h g}^{-1}$  and  $421/376 \text{ mA h cm}^{-3}$ ). In addition, the reversible capacity was well-retained after 200 cycles, maintaining approximately 97.3% capacity retention with high Coulombic efficiency per cycle. The rate capability of the  $\text{Si}_2\text{Te}_3\text{@C}$  composite anode, as a function of the C-rate (1C =  $550 \text{ mA g}^{-1}$ ), is shown and compared with that of the graphite anode (Fig. 3c, d, and Fig. S13, ESI†). At cycling rates of 0.1, 0.2, 0.5, 1, 2, and 3C, the  $\text{Si}_2\text{Te}_3\text{@C}$  composite displayed high reversible capacities of 1291, 1180, 1029, 938, 775, and  $672 \text{ mA h cm}^{-3}$ , respectively, outperforming the graphite anode. Moreover, during long-term cycling performance of the  $\text{Si}_2\text{Te}_3\text{@C}$  composite anode at a cycling rate of 1C ( $550 \text{ mA g}^{-1}$ ), the reversible capacity remained at  $402 \text{ mA h g}^{-1}$  ( $861 \text{ mA h cm}^{-3}$ ) with stable capacity retention of 88% after 300 cycles (Fig. 3e). This excellent electrochemical performance can be attributed to the ultrafine  $\text{Si}_2\text{Te}_3$  nanocrystallites (5–7 nm) embedded within the amorphous carbon support, which effectively provide short Li-ion diffusion paths, enhance ionic and electronic conductivity, and

mitigate the effects of volume expansion during lithiation/delithiation processes (Fig. S14, ESI†).

In summary, this study demonstrates the successful synthesis of layered  $\text{Si}_2\text{Te}_3$  through a scalable solid-state process combining sequential BM and HT. To address the inherent limitations of  $\text{Si}_2\text{Te}_3$ , including low electrical conductivity and structural instability, a  $\text{Si}_2\text{Te}_3\text{@C}$  composite was developed by incorporating amorphous carbon. The carbon matrix effectively enhanced electronic conductivity, provided structural stability, and mitigated volume changes during lithiation/delithiation processes. Electrochemical evaluation revealed that the  $\text{Si}_2\text{Te}_3\text{@C}$  composite exhibited high reversible capacities ( $559 \text{ mA h g}^{-1}/1196 \text{ mA h cm}^{-3}$ ) and excellent cycling stability, retaining  $\sim 97.3\%$  of its initial capacity after 200 cycles. It also demonstrated superior rate performance, maintaining  $\sim 402 \text{ mA h g}^{-1}$  ( $861 \text{ mA h cm}^{-3}$ ) at 1C after 300 cycles with 88% capacity retention. *Ex situ* XRD and HR-TEM analyses confirmed a reversible conversion reaction mechanism involving the formation and recombination of  $\text{Li}_2\text{Te}$  and  $\text{Li}_{3.75}\text{Si}$  phases, facilitated by the conductive carbon matrix. These findings highlight the potential of  $\text{Si}_2\text{Te}_3$ -based composites as high-performance anode materials for next-generation LIBs and provide valuable insights for the design of layered chalcogenide materials with enhanced electrochemical properties.

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## Data availability

The data supporting this article have been included as part of the ESI.†

## Conflicts of interest

There are no conflicts to declare.

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