

RESEARCH ARTICLE

Electron Donor-Acceptor Systems With Black Phosphorus and Tellurium for High-Performance Interfacial Lithium Diffusion and Storage

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ABSTRACT

Black phosphorus (BP), with high theoretical capacity and electrical conductivity, has emerged as a promising candidate for an advanced energy storage system. However, its huge volume expansion caused by the solubility of intermediate discharge products, i.e., Li_xP , leads to significant capacity fading. Herein, a specially designed BP/Te@C anode with a 3D network structure is constructed via interfacial electron transfer between BP and Te, forming a typical electron donor-acceptor system. Besides, Tellurium, with metalloid properties and high ductility, not only strengthens the interfacial interaction within BP but also significantly enhances the overall electrical conductivity. 3D tomography reconstruction and ToF-SIMS confirm the structural integrity and stability of this unique architecture during long-term cycling. Additionally, theoretical calculations reveal a special interfacial lithium diffusion and storage mechanism in BP/Te@C composites, which contributes to the grain size refinement and the improved utilization of BP and Te during cycling. BP/Te@C anodes deliver a capacity of 734.4 mAh g^{-1} at 3 A g^{-1} with well cycling performance, along with the low-temperature adaptability (-20°C). $\text{LiFePO}_4\|\text{BP/Te@C}$ full cells achieve an outstanding power density of 4051.6 W kg^{-1} while maintaining a high-capacity retention of 90.7%. This work builds an ingenious protocol for designing robust phosphorus-based anodes with superior electrochemical performance.

1 | Introduction

Lithium-ion batteries (LIBs) have gradually emerged as the predominant energy storage solution for portable electronics and electric vehicles, owing to their high energy density, excellent cycle stability, and superior specific capacity [1, 2]. However, as energy storage requirements continue to evolve, the performance limitations of conventional carbon-based anode materials have become increasingly evident. This has driven significant research interest in developing next-generation anode materials with higher capacity and enhanced cycling durability [3].

Black phosphorus (BP), a thermodynamically stable allotrope of phosphorus, offers distinct advantages over other forms of phosphorus, e.g., red phosphorus (RP), white phosphorus, and purple phosphorus, such as high chemical stability and electrical conductivity. BP exhibits three key merits that make it a promising candidate for advanced LIB anodes. First, the layered structure with a large interlayer space of $5.2 \text{ \AA}$ provides plenty of active sites for metal-ion storage. Thus, BP possesses an exceptionally high theoretical specific capacity of 2596 mAh g^{-1} , which significantly surpasses that of commercial graphite (372 mAh g^{-1}) and lithium titanate (175 mAh g^{-1}) [4, 5]. Second, its moderate lithiation

potential (~ 0.7 V vs. Li^+/Li) strikes a favorable balance between energy efficiency and safety by mitigating the risk of lithium dendrite formation, while maintaining competitive energy density, which is commonly observed in silicon-based anodes operating near 0.2 V [6, 7]. Third, BP demonstrates superior carrier mobility and electrical conductivity compared to red phosphorus, thereby facilitating the kinetics of electrochemical redox reactions during the charge and discharge process [8]. These obvious advantages set BP as a focal point in the pursuit of next-generation anode materials for LIBs [9, 10]. Nevertheless, the practical implementation of BP anodes faces several critical challenges. For example, the intrinsic ionic conductivity of phosphorus is extremely low ($\sim 10^{-12}$ $\text{S}\cdot\text{m}^{-1}$), which usually results in the sluggish charge transfer kinetics during lithiation/delithiation [11]. Second, substantial volume expansion ($\sim 300\%$) of BP occurs upon lithium insertion, leading to a mechanical degradation of the electrode, particle pulverization, and instability of the solid electrolyte interphase (SEI), which collectively contribute to rapid capacity fading [12]. Third, the lithiation mechanism of BP anodes typically proceeds through a multi-step reaction pathway ($\text{P} \rightarrow \text{Li}_x\text{P} \rightarrow \text{Li}_3\text{P}$), which produces soluble intermediate species (lithium phosphides, Li_xP), thus causing irreversible capacity fading and reduced Coulombic efficiency [13].

To solve these significant drawbacks of BP anodes, it is necessary to construct a rational design of BP-based composite with high electrical conductivity and improve both structural stability and rate capability. Traditional strategies include spatial confinement of nano-phosphorus with a carbon component and chemical oxidation with a Lewis acid [14]. Ball-milling is a commonly used method for the confinement of nano-phosphorus, which is cost-efficient and has a widespread application. For example, Sun and Haghighat-Shishavan ball-milled RP with graphite and carbon nanotubes, respectively, and yielded a high capacity of 1849 and 1681 mA h g^{-1} after 100 and 400 cycles, respectively [15, 16]. During ball-milling, it is found that RP can transform into BP, implying the high thermodynamic stability of BP instead of RP. Polymer component, e.g., PANI, is also used for the confinement of BP [17]. However, the reported ball-milling is time-consuming and only provides weak interaction between BP and the carbon component, lacking a well-designed structure.

In addition to the structural design of BP anodes, the underlying mechanism of Li^+ diffusion and storage during cycling is also important to achieve high-performance LIBs. Commonly, it is confirmed that BP will interact with Li^+ by an alloying process during discharge, forming Li_xP , e.g., LiP , Li_2P , and then Li_3P , and the discharge products transform back to phosphorus when fully charged [18, 19]. Other studies also show that additional Li species, e.g., LiP_7 , Li_3P_7 , Li_4P_3 , can be theoretically formed during alloying [20]. Thus, a solid study should be carried out to study the specific reaction mechanism in BP anodes. Besides, previous studies always pay lots of attention to the chemical composition changes during reaction, but ignore the structural evolution of BP anodes, which is more important to understand the relationship between “structure” and “electrochemical properties”.

Herein, we design a BP-based composite by incorporating tellurium (Te) as a functional block into the traditional phosphorus/graphene (P/C) matrix via fast ball-milling. Tellurium, with high electrical conductivity (2×10^2 $\text{S}\cdot\text{m}^{-1}$) and metalloid

property, can provide additional electrons to BP and form a robust electron donor-acceptor system instead of the traditional $\text{p}-\pi$ conjugation in P/C. Notably, tellurium possesses metal-like ductility, enabling it to function as an effective binder that improves the adhesion between BP and C components. Furthermore, 3D tomography reconstruction and ToF-SIMS characterizations verify this distinguished and robust configuration during long-term cycling. Specifically, C components form the basic 3D framework, while BP and Te are confined in nanochambers with a 2D diffusion pathway. Additionally, electrochemical tests and theoretical calculations reveal a special interfacial lithium diffusion and storage mechanism in BP/Te@C composites, where lithium ions prefer to be adsorbed in the interlayers of BP/Te instead of the surface. This unique diffusion passway contributes to the grain size refinement and the improved utilization of BP and Te during cycling. Consequently, BP/Te@C anodes deliver a capacity of 734.4 mAh g^{-1} at 3 A g^{-1} with well cycling performance, as well as the wide temperature adaptability from -20°C to 20°C . $\text{LiFePO}_4\|\text{BP/Te@C}$ full cells achieve an outstanding power density of 4051.6 W kg^{-1} with a high-capacity retention of 90.7%.

2 | Results and Discussion

Typically, BP/Te@C composites were synthesized via a two-step high-energy ball milling process (details in Experimental Section, Supporting Information). Here, home-made BP crystals were provided by a chemical vapor transportation (CVT) method to ensure high quality instead of being oxidized. Additional carbon components, including carbon nanotubes (CNTs) and graphene sheets, were proportionally added to form the most stable BP/Te@C composites with the best electrochemical behavior (Figure S1). Figure 1a presents the X-ray diffraction (XRD) patterns of BP, Te, and BP/Te@C composite, respectively. As shown in Figure 1a, XRD patterns of BP/Te@C exhibit characteristic diffraction peaks at 23.1° , 27.7° , and 40.6° , which mainly correspond to the standard patterns of hexagonal crystal system tellurium (PDF No.36-1452), indicating that the well-crystalline structures of Te are preserved after ball milling. The carbon peak mainly appears as a broad peak, which is overlapped with the characteristic peak of Te at 23.1° and 27.7° . Besides, it should be noted that the characteristic peaks of orthorhombic BP (PDF No.47-1626) are imperceptible, probably due to the amorphous and nanocrystalline feature of BP after high-energy ball milling, which has also been reported in previous work [15]. Therefore, the specific content of BP in BP/Te@C has been tested by inductively coupled plasma optical emission spectroscopy (ICP-OES) and thermogravimetry analysis (TGA), which refers to a weight percentage of 8.12 wt.% for BP and 45.42 wt.% for Te (Figure S2 and Table S1). Figure 1b exhibits the Raman spectra of BP, Te, and BP/Te@C. Here, the spectrum of BP/Te@C displays three distinct peaks at 344.4, 417.8, and 446.1 cm^{-1} , which are assigned to the A_g^1 , B_g^2 , and A_g^2 vibrational modes of BP, respectively [12]. Additionally, two peaks at 106.9 and 127.3 cm^{-1} correspond to the A_1 and E_2 phonon modes of Te [21]. The observed overall redshift in Raman bands of BP/Te@C suggests a reduction in grain size of both BP and Te to the nanoscale after ball milling, which is consistent with the XRD results and previous reports [12]. To intuitively observe the interface of BP and Te in BP/Te@C, Figure 1c shows the low-resolution transmission electron microscopy (TEM) image

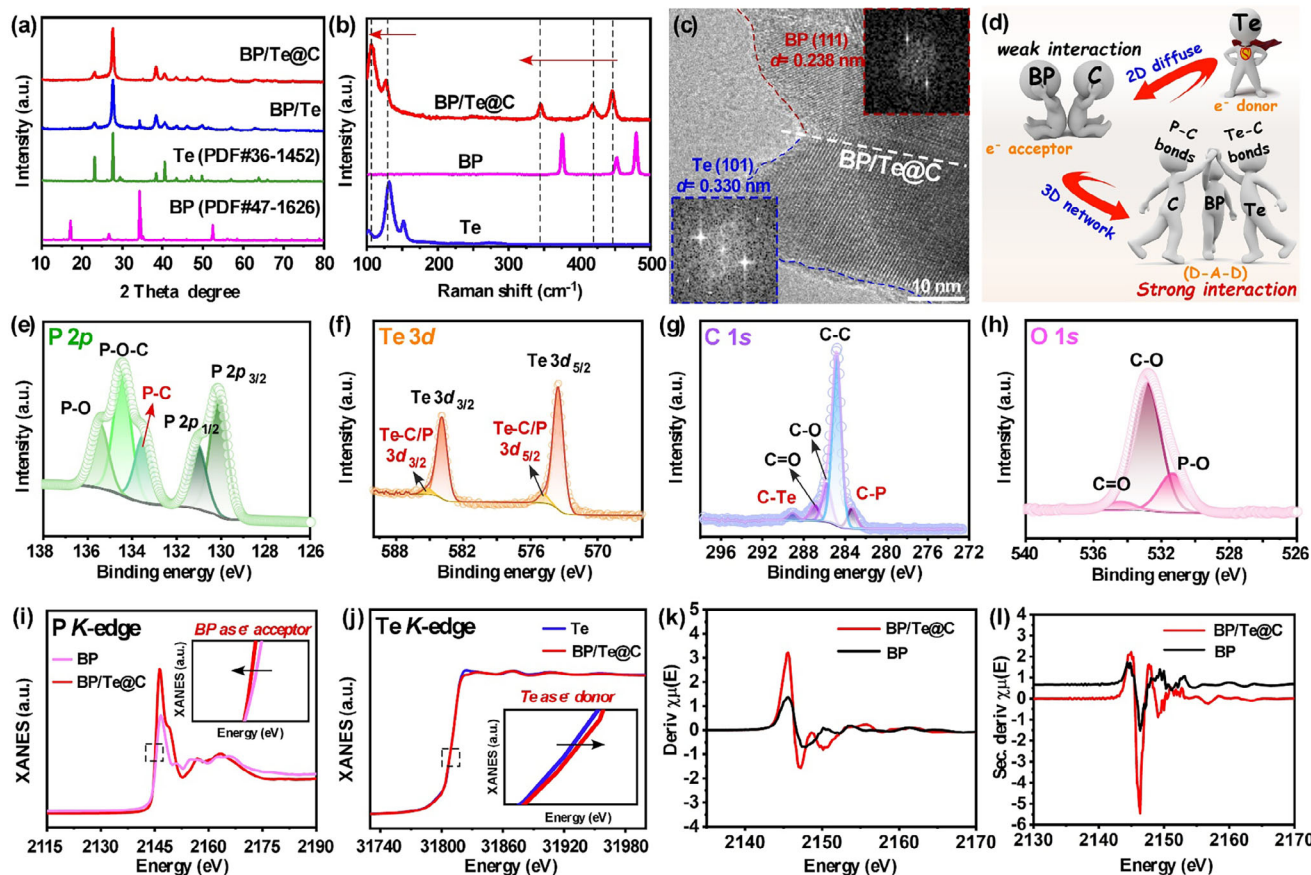


FIGURE 1 | Characterizations of the electron donor-acceptor system for BP/Te@C. (a) XRD spectra of BP/Te@C, BP/Te, Te, and BP. (b) Raman spectra of BP/Te@C, Te, and BP. (c) Low-resolution TEM image of BP/Te@C to show the interface, inset: corresponding FFT patterns. (d) Schematic illustration of the electron donor-acceptor system for BP/Te@C. (e–h) P 2p, Te 3d, C 1s, and O 1s XPS spectra of BP/Te@C, respectively. (i–j) P and Te K-edge XANES spectra for BP and BP/Te@C, respectively. (k–l) The first-order and second-order differentiation of the synchrotron radiation curves of the P K-edge of BP and BP/Te@C.

of BP/Te@C, in which two different crystal domains refer to lattice fringes with the interplanar spacings of $d = 0.330$ and $d = 0.238$ nm, corresponding to Te (101) [21] and BP (111) lattice plane [22], respectively. A clear interface can be observed in Figure 1c, which implies the electron transfer between Te and BP. Detailed observations of the structural arrangement are displayed in Figure 2 by 3D tomography reconstruction. Moreover, high-resolution X-ray photoelectron spectroscopy (XPS) characterizations have been conducted on BP/Te@C, BP, and Te to observe the chemical environment and elemental states (Figure 1e–h and Figure S3). For BP/Te@C, P 2p XPS spectra can be mainly deconvoluted into P–C, P–O–C, P–O, P 2p_{1/2}, and P 2p_{3/2} peaks. When compared to pure BP, the BP/Te@C composite exhibits additional P–C and P–O–C bonds, which indicate the successful bonding between BP and the carbon component [23]. In addition, for Te 3d spectrum of BP/Te@C, characteristic peaks which are observed at 585.3 and 574.9 eV can be assigned to Te–C/Te–P bonds [24], while peaks at higher binding energy (583.9 and 573.5 eV) correspond to Te 3d_{3/2} and Te 3d_{5/2}, respectively [25, 26]. Oppositely, for Te 3d spectrum of pure Te, only Te–Te bond is present. Figure 1g shows the C 1s spectrum of BP/Te@C, in which the characteristic peak at 289.0 eV refers to Te–C bonds [26, 27] and the peak at 283.36 eV refers to P–C bonds [23]. This demonstrates the strong interaction between the carbon

component and BP/Te, which greatly enhances the structural stability of BP/Te@C during cycling.

To further probe the specific electronic structure of the as-prepared samples, synchrotron-based X-ray absorption near-edge spectroscopy (XANES) is employed to observe the charge transfer in BP/Te@C. Figure 1i displays the P K-edge spectra of pristine BP and BP/Te@C composites. Compared with pure BP, BP/Te@C shows a red shift, implying that the valence of P becomes negative. This is due to the fact that BP accepts electrons (acceptor) from Te or carbon component, which is in good agreement with the aforementioned XPS results. Besides, the Te K-edge spectra of pure Te and BP/Te@C hybrids are displayed in Figure 1j. Oppositely, compared to pure Te, the spectrum of BP/Te@C composite shows a blue shift, indicating the valence of Te becomes more positive in BP/Te@C. This should be related to the fact that Te donor electrons go to BP, which is also consistent with XPS spectra in Figure 1f. Moreover, the first-order differentiation of the synchrotron radiation curves of BP/Te@C and BP are shown in Figure 1k. The derivative $\chi_{\mu}(E)$ values of BP/Te@C are larger than those of BP, which is due to the confinement of the carbon component for BP/Te in BP/Te@C, which can prevent it from being oxidized to phosphorus-containing oxides or phosphoric acid derivatives. Therefore, the synchrotron source can easily

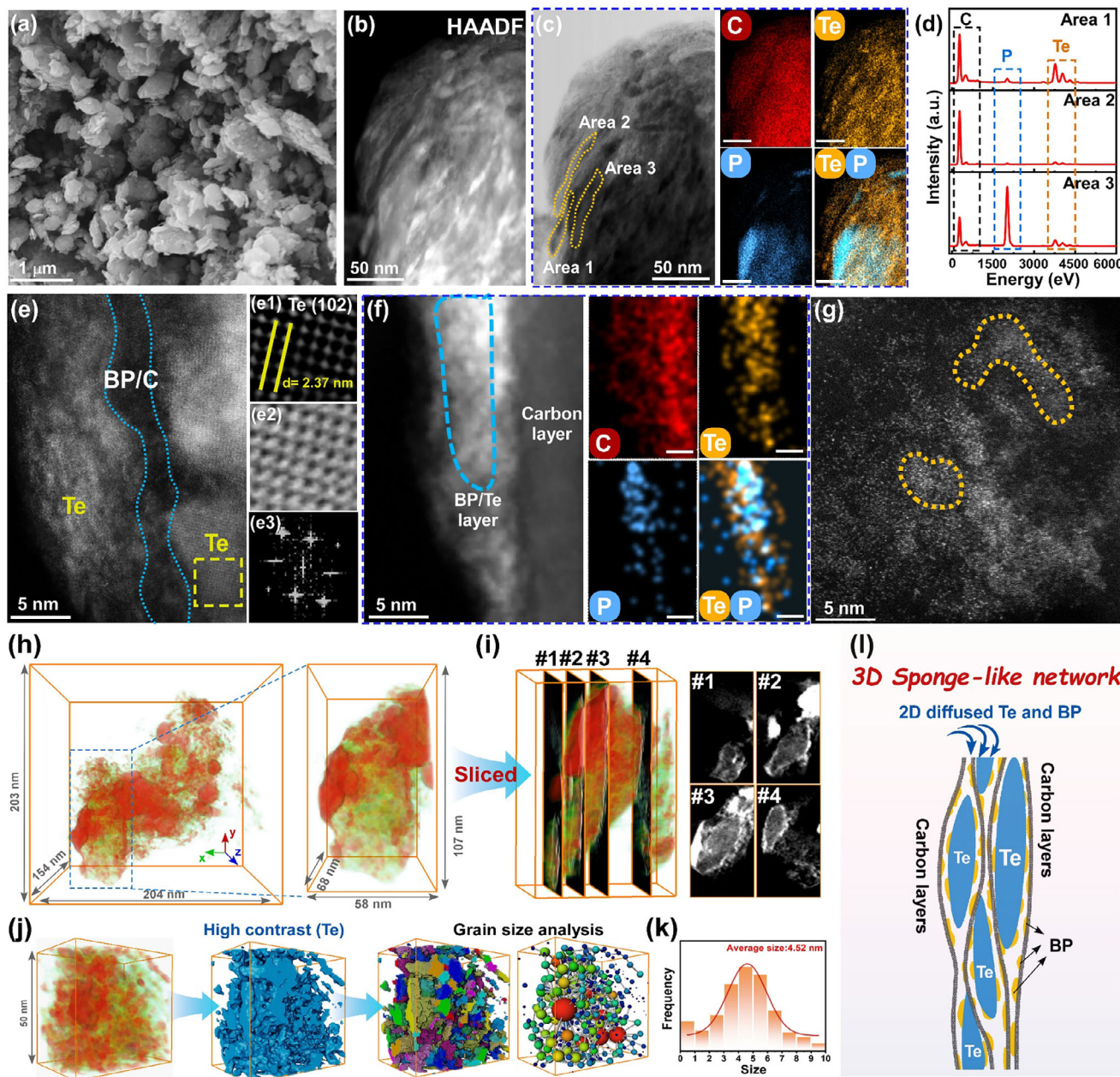


FIGURE 2 | Structural configurations of BP/Te@C before cycling. (a) SEM image of BP/Te@C. (b) and (c) STEM-HAADF images of BP/Te@C, as well as the corresponding elemental mappings of Te, P, and C. (d) EELS spectra of the corresponding areas in (b). (e) High-resolution STEM and FFT images of BP/Te@C. (f) STEM-HAADF image and the corresponding elemental mappings to show the interface BP/Te and C layers. (g) High-resolution STEM-HAADF images of BP/Te@C to show the distribution of BP/Te nanoclusters. (h) A sub-volume extraction of BP/Te@C by the 3D tomography technique. (i) Orthogonal cross-sections of reconstructed BP/Te@C. (j) and (k) Tomography-based segmentation of high-contrast Te and the corresponding analysis of grain size distribution. (l) Schematic plot of the structural configuration of BP/Te@C materials.

penetrate pure BP and offer smaller $\chi\mu(E)$ values [28]. For the second-order differentiation of the synchrotron adiation curves in Figure 1l, the trends of these two samples are consistent with the first-order curves, and the $\chi\mu(E)$ values of BP/Te@C are larger than those of pure BP. Similarly, this indicates that the well confinement of BP/Te@C. Therefore, the comprehensive studies of XPS and XAF results provide a preliminary framework of the composition and electronic environment in BP/Te@C (Figure 1d). Here, due to the addition of Te, which possesses metalloid properties with high conductivity, it can offer electrons to BP and facilitate the interface interaction, i.e., Te and C act

as the electron donors, while BP acts as the electron acceptor. Benefited from this electron D (Te)-A (BP)-D (carbon component) system, a strong 3D network has been constructed, which will be discussed in detail in the following part.

Aiming to unravel the microstructure, high-resolution TEM based on the double aberration correction and 3D tomography reconstruction has been carried out in Figure 2. First, Figure 2a shows the macroscopic morphology of BP/Te@C by scanning electron microscopy (SEM). It can be observed that BP/Te@C shows a distinguished layered structure with relatively uniform

lateral diameter after ball milling. This is probably due to the unique and excellent ductility of Te, which possesses a unique anisotropic trigonal crystal structure and is composed of aligned molecular chains connected by van der Waals bonds. This layered structure of BP/Te@C provides a large specific surface area, which significantly benefits the wettability of the electrolyte (Figure S4), thereby improving the Li⁺ storage and transmission efficiency.

Figure 2b presents low-resolution high-angle annular dark-field scanning transmission electron microscope (STEM-HAADF) images of BP/Te@C and the corresponding energy dispersive X-ray spectroscopy (EDX) mappings of BP, Te, and C. Similarly, a layered distribution of Te and P can be well observed. Besides, Figure 2c provides the EDS mapping results of Te, P, C, and mixed Te/P according to the area in Figure 2b, along with the electron energy loss spectroscopy (EELS) results for three regions shown in Figure 2d. It can be clearly seen that before cycling, Te and P are separately distributed in the microscope. For the outside region (Area 2), the main composition is carbon component, while for the inner region (Area 1 and 3), it is Te and P separately dominated. These results verify the layer-by-layer structure in pristine BP/Te@C materials. Furthermore, Figure 2e exhibits the high-resolution observation of the layered structure of pristine BP/Te@C materials by STEM-HAADF characterizations. Here, the P/C layers (dark regions) are sandwiched between the Te layers, which can be confirmed by atomic phase images in Figure 2e1 and e2. Also, the lattice fringes of bright regions in Figure 2e are confirmed to be the Te (102) crystal plane by fast Fourier transformation (FFT) (Figure 2e3). Then, with the help of high-resolution STEM-HAADF and EDS mapping characterizations, we demonstrate this closely stacked layer-by-layer structure from the side view. In Figure 2f, it can be seen that BP and Te layers uniformly adhere to the carbon layers, forming a 2D nanochamber by carbon component. Additionally, Figure 2g shows that Te clusters present a uniform distribution in pristine BP/Te@C after ball milling. To further confirm the specific grain size of Te in pristine BP/Te@C materials, 3D tomographic reconstruction is performed (Figure 2h). Typically, HAADF-STEM images are collected at intervals of 2° within a range of 150° for tomographic reconstruction (Experimental Section, Supporting Information). The resulting reconstructed structural units and HAADF images at different rotation angles are shown in Figure S5a. The reconstructed front view shape closely resembles the HAADF image captured at a 70° angle, validating the effectiveness and authenticity of the reconstruction. Figures S5b presents the front, right, and top views of the reconstructed unit, which occupies the bounding box with length $x = 204$ nm, width $z = 154$ nm, and height $y = 203$ nm, respectively. Subsequently, a sub-volume extraction operation (with the bounding box of $x = 58$ nm, $z = 68$ nm, $y = 107$ nm) is performed on the reconstructed BP/Te@C (Figure 2h). Figure 2i provides the orthogonal cross-sections of reconstructed BP/Te@C, which are sequentially marked with No. 1, 2, 3, and 4. In these orthogonal slices, it can be found that high-contrast areas (refer to Te) are mixed with the low-contrast areas (refer to BP/C) (Figure S6), forming a sponge-like structure, which indicates the well-constructed 3D network in BP/Te@C. Furthermore, as shown in Figure 2j, the segmentation based on the contrast in the reconstructed box is conducted to analyze the grain size of Te in pristine BP/Te@C. Here, the high-contrast volume (Te) is

separated from the low-contrast volume (BP/C). Figure S7 illustrates the selection of sub-volumes, where regions with relatively continuous particles are selected for analysis. Subsequently, a watershed algorithm, applied to a distance map derived from the segmented image, is employed to delineate boundaries between adjacent yet topologically distinct features, such as individual particles or interconnected void networks. This process effectively separates the continuous solid or pore volume into numerous distinct segments amenable to quantitative analysis. Following segmentation, these discrete Te volumes are uniquely labeled and color-coded for visualization (blue box in Figure 2j and Figures S8–S9). To further represent the volumetric distribution and interconnectivity of these segmented phases, a volume-and-connectivity-equivalent ball-and-stick model is generated (right panel in Figure 2j and Figure S10). Here, larger segmented volumes are depicted with warmer colors (larger spheres), while smaller volumes are shown with cooler colors (smaller spheres). Besides, sticks between spheres indicate the connectivity between neighboring volumes. Statistical analysis of the feature sizes within the segmented volume yields average diameters of 4.52 nm for Te clusters (Figure 2k). These quantitative results further confirm that Te and BP components in BP/Te@C after ball milling have reached an astonishing nanoscale level with a homogeneous mixture.

Therefore, the aforementioned studies provide a comprehensive view of the structural configuration of BP/Te@C before cycling (Figure 2l), i.e., a 3D sponge-like network in microscale with nanochambers impregnated by 2D diffused Te and BP. Synergistically, the 3D network constructed by the carbon component can promote the overall conductivity, while enclosed active materials (BP and Te) can be fully utilized for Li⁺ storage. Benefited from the high ductility (Figure S11) and electron-donating properties of Te, the 2D diffused active materials in nanochambers can effectively buffer the volume change. Thus, this loose and continuous structure of BP/Te@C is crucial for adapting the large volume expansion caused by both BP and Te, and significantly improves the Li⁺ diffusion kinetics during cycling, consequently enhancing the long-term stability and rate performance of LIBs.

To reveal the advantages of this unique 3D configuration of BP/Te@C in electrochemical behavior, the structural evolution of BP/Te@C during cycling has been studied by 3D tomography reconstruction (Figure 3). Figure 3a–f provide the STEM-HAADF images and the corresponding grain size analysis of Te in BP/Te@C after cycling numbers (e.g., 50, 100, and 500). Inset in Figure 3a presents the HAADF image of pristine BP/Te@C, which appears as a relatively intact and dense structure. However, upon cycling, significant morphological transformations are observed. After 50 cycles of the charge and discharge process, examination of the materials in their charged state reveals that the structural configuration gradually becomes loose and porous, which tends to form small clusters (Figure 3a). This phenomenon persisted until the 500th cycle (Figure 3c,e). The progressive structural evolution indicates that the material underwent dynamic and continuous structural adaptation and reconstruction during the cycling process.

While 2D STEM-HAADF imaging provides valuable morphological insights into the structural evolution, its projection nature limits the precise spatial localization of features like pores (i.e.,

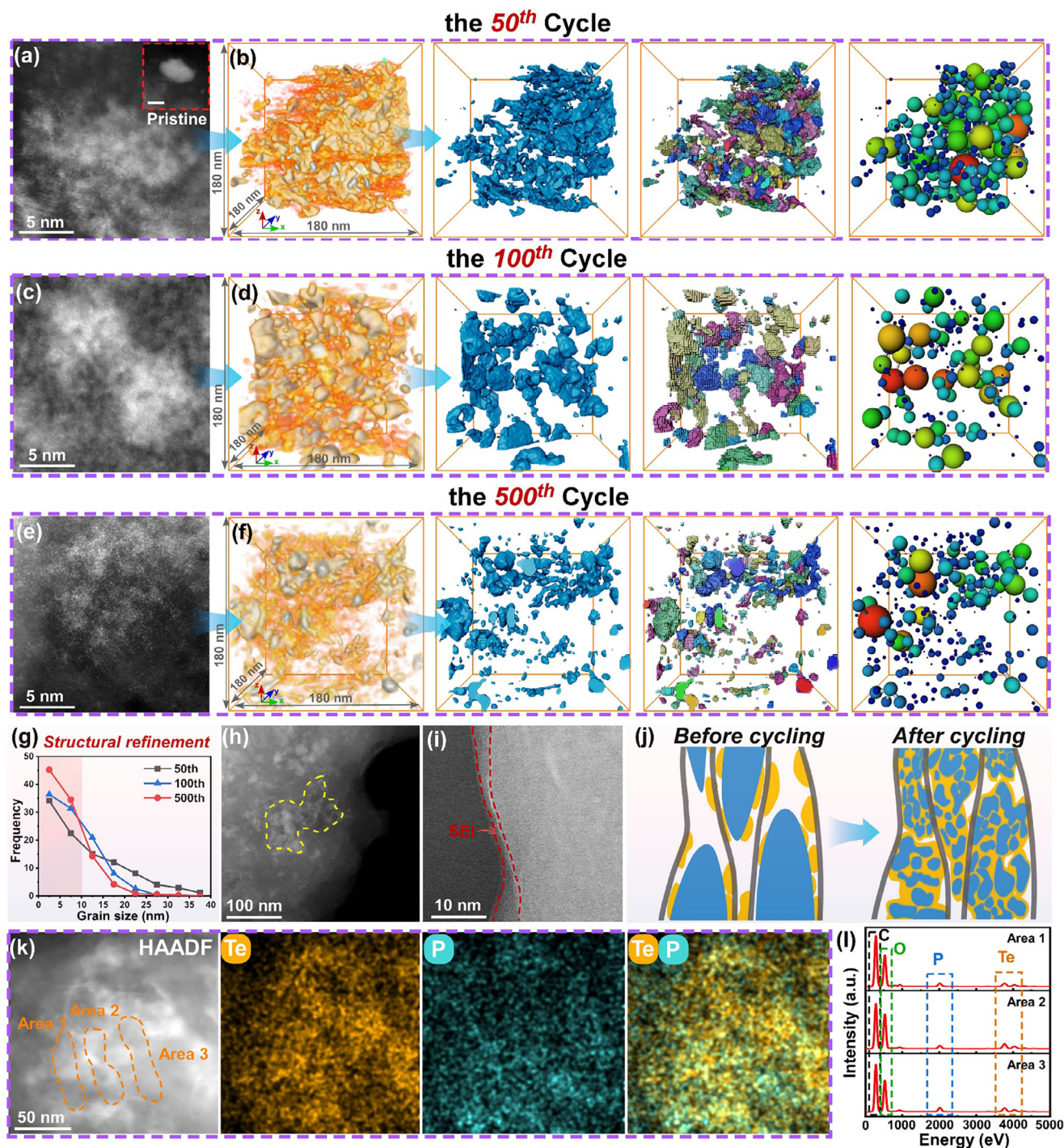


FIGURE 3 | Structural configurations of BP/Te@C after cycling. (a), (c) and (e) STEM-HAADF image of BP/Te@C after 50, 100, and 500 cycles of charge and discharge process, respectively. Inset in (a): STEM-HAADF image of pristine BP/Te@C. Scale bar: 5 nm. (b), (d) and (f) Tomography-based segmentations of high-contrast Te in BP/Te@C after 50, 100, and 500 cycles of charge and discharge process, respectively. (g) Analysis of grain size distribution according to the tomography-based segmentations. (h) Low-resolution STEM-HAADF image of BP/Te@C after 500 cycles. (i) SEI coated on BP/Te@C after 500 cycles. (j) Schematic plot of the structural evolution of BP/Te@C during cycling. (k) STEM-HAADF and EDS mappings of BP/Te@C after 500 cycles. (l) EELS spectra of different regions in (k).

differentiating surface vs. internal voids). To overcome this limitation and obtain a comprehensive 3D understanding of the structural evolution, ex situ 3D tomography reconstruction is performed on BP/Te@C particles after 50, 100, and 500 electrochemical cycles (Figure 3b,d,f). Besides, STEM-HAADF images from various tilt angles as a precondition for tomographic

reconstruction are shown in Figures S12a, S13a, and S14a. The acquired tilt series are then used to reconstruct 3D volumes, showcasing the three standard views. To facilitate the quantitative comparison, sub-volumes of equivalent dimensional sizes are extracted from the reconstructed models (Figures S12c, S13c, and S14c). From these sub-volumes, 3D surface models (right panels of

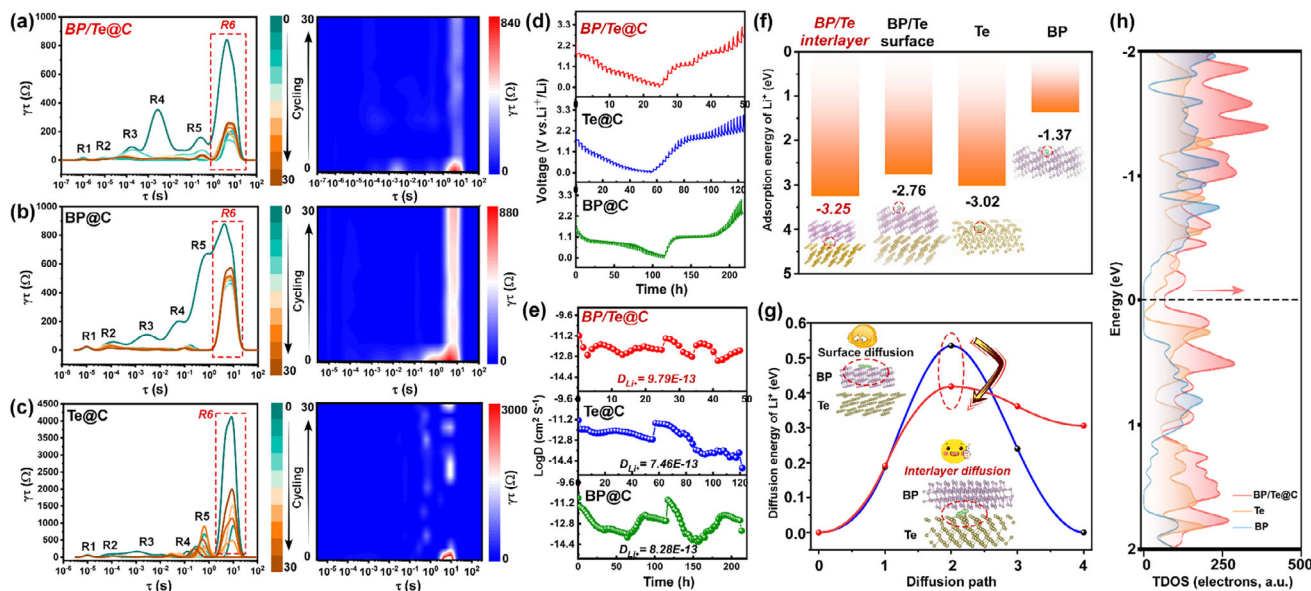


FIGURE 4 | The interlayer lithium diffusion and storage of BP/Te@C. (a–c) In situ EIS and DRT patterns of BP/Te@C (a), BP@C (b), and Te@C (c), respectively. (d) GITT curves of BP/Te@C, BP@C, and Te@C anodes. (e) Plots of Li^+ diffusion coefficient (D_{Li^+}) as a function of the state during the lithiation/delithiation process. (f) Adsorption energy of Li^+ on the surface of BP/Te@C, BP@C, and Te@C. (g) Diffusion energy of Li^+ on BP/Te interlayer and BP/Te surface. (h) Total density of states (PDOS) of BP/Te@C, BP@C, and Te@C.

Figure 6b,d,f) are generated by the same strategy used in Figure 2j (Figures S15–S23, Video S1), and a statistical analysis of the grain size of Te after cycling is conducted on the feature sizes within the segmented volumes (Figure 3g). After 50 cycles, the grain size distribution frequency is relatively wide, and grains with a diameter (d) exceeding 30 nm can still be found. After 100 cycles, the grain size distribution frequency becomes concentrated, and the average diameter gradually decreases. However, there is still a certain proportion of grains with $d = 20\text{--}30$ nm that can be found. Interestingly, grains with $d \geq 20$ nm can be seldom found in samples after 500 cycles of charge and discharge, while grains with $d \leq 10$ nm dominate the products. The above results indicate that the structure of BP/Te@C is continuously refined during cycling, which is consistent with the 2D STEM-HAADF images. Additionally, Figure 3h presents the porous structure with nanoscale holes of BP/Te@C after 500 cycles of charge and discharge, probably due to volume expansion during cycling. Besides, a stable and dense solid electrolyte interface (SEI) is in situ formed on the surface of discharge products after cycling (Figure 3i), of which the composition will be detailedly revealed in Figure 5. This compact SEI effectively inhibits the volume expansion, thereby significantly enhancing the cycling stability of LIBs. Additionally, the surface and cross-section morphology of the electrodes after fast charging were characterized by SEM (Figure S24). The SEM analysis shows that after cycling, the BP/Te@C electrode maintains a smooth, dense surface with a significantly lower volumetric expansion (144%) compared to the cracked BP@C electrode (243%), demonstrating that the addition of Te improves Li^+ adsorption and kinetics to ensure uniform lithiation and reduce stress during fast charging. Figure 3j schematically shows the structural evolution of BP/Te@C during cycling. Initially, carbon component forms a continuous 3D network with BP and Te confined in nanochambers of carbon layers, which provides structural support and pathways for e^-/Li^+ . After cycling, BP and Te in nanochambers undergo self-

adaptation and refinement, intermingling with each other, and forming a sharp contrast to the separated state before cycling. Figure 3k proves the homogeneous intermixture of BP and Te after cycling by EDS mapping of Te and P. Moreover, Figure 3l shows the EELS spectra of C, P, and Te for different areas extracted from Figure 3k. It can be seen that in all selected regions, the intensities of P, Te, and C remain consistent, verifying that BP and Te have been interpenetrated with each other after cycling.

Furthermore, to deeply understand the underlying reason for the homogeneous intermixture of BP and Te after cycling, the capability of Li^+ diffusion and storage in BP/Te@C has been explored by electrochemical test and theoretical illustrations. Figure 4a–c show the in situ electrochemical impedance spectroscopy (EIS) tests and corresponding distribution of relaxation times (DRT) fitting analyses of three samples, i.e., BP/Te@C, BP@C, and Te@C (details in Experimental Section, SI). It is observed that the impedance gradually decreases with increasing cycle numbers, and the impedance of BP/Te@C becomes even smaller than that of the other two reference samples after 30 cycles. This indicates the faster Li^+ diffusion and smaller charge transfer resistance in BP/Te@C. According to the DRT analysis, BP/Te@C exhibits six distinct peaks (denoted as R1 to R6) within the time scale (τ) range of 10^{-6} to 102 s, where each peak corresponds to a specific electrochemical reaction process. The last peak, i.e., R6, located in the range of 1 to 100 s, is associated with diffusion impedance and charge transfer resistance. Given that R6 is the most prominent peak, it suggests that the electrochemical reactions of all three samples are primarily diffusion-controlled. R1 corresponds to contact resistance, which reflects the low contact resistance and excellent structural conductivity of BP/Te@C. R2 represents the impedance of Li^+ transport across the SEI layer, implying the formation of more resistive SEI layers in BP@C and Te@C. Besides, R5 and R6 fall within the diffusion resistance

region, further confirming the superior diffusion performance of BP/Te@C [29, 30].

The Li^+ diffusion coefficients (D_{Li^+}) at various states of charge are determined using GITT tests. Figure 4d displays typical voltage relaxation profiles for the three electrodes during lithiation and delithiation, while the voltage change during relaxation after each current pulse reflects the overpotential of the corresponding reaction step. The specific D_{Li^+} values of different samples are calculated based on Fick's second law (Figure 4e). It shows that Li^+ diffusion in BP/Te@C ($D_{\text{Li}^+} = 9.79 \times 10^{-13}$) are significantly higher than that of the other two samples, confirming the improved Li^+ diffusion kinetics. This enhancement is attributed to efficient stress dissipation and fast interface diffusion provided by a 3D network of BP/Te@C during cycling, which will be revealed in Figure 4f,g. To further investigate the specific Li^+ diffusion pathway in BP/Te@C, density functional theory (DFT) calculations are employed to systematically examine the electrochemical behaviors of four distinct configurations: BP/Te interlayers, BP/Te surface, Te, and BP (details in Experimental Section). Figure 4f shows the adsorption energy of Li^+ on different models. Among them, BP/Te interlayers exhibit the lowest adsorption energy of Li^+ (−3.25 eV), which is significantly lower than that on BP/Te surface (−2.76 eV), Te (−3.02 eV), and BP (−1.37 eV). These indicate that Li^+ prefers to adsorb in BP/Te interlayers, effectively providing more active adsorption sites for Li^+ adsorption and significantly promoting the surface adsorption process of Li^+ . Additionally, the diffusion pathway and energy barriers of Li^+ for BP/Te interlayers and BP/Te surface are further calculated (Figure 4g). Here, BP/Te interlayers show a significantly lower diffusion barrier than that of the BP/Te surface, suggesting that the heterointerface is conducive to the rapid migration of Li^+ . Combining the advantages of both adsorption and diffusion, BP/Te@C exhibits superior Li^+ diffusion and storage characteristics, thereby significantly enhancing the electrochemical properties of LIBs. Besides, a charge enrichment phenomenon occurring on the BP/Te interlayers is unveiled via the differential charge density map coupled with Bader analysis in Figure S25. It is distinctly observed that Te functions as an electron donor (e^- · donor), while BP serves as an electron acceptor (e^- · acceptor), which further confirms the electron D-A system of BP/Te@C. Quantitative calculations further demonstrate that the charge transfer of Li^+ across the BP/Te interlayer amounts to −0.87e, which attests to the lower charge transfer energy barrier between the BP/Te interlayer and facilitates the Li^+ diffusion kinetics. Figure 4h provides the total density of states (TDOS) of different models. It can be observed that BP/Te@C occupies more electrons near the Fermi level when compared with that of BP@C and Te@C, indicating the enhanced electrical conductivity of BP/Te@C.

After revealing the advantages of interfacial Li^+ diffusion and storage in BP/Te@C, the potential Li^+ storage mechanism is further probed via ex situ X-ray photoelectron spectroscopy (XPS) in Figure 5a–c. Here, the reversible Li^+ insertion/extraction process in BP/Te@C is confirmed by the periodic attenuation and enhancement of Te—Te/Te—C (Figure 5b) and P—P bond signals (Figure 5c). In the high-resolution Te 3d XPS spectra, the binding energies of Te—Te/Te—C bonds shift toward lower position during discharge and recover to higher energies during charge. Concurrently, as lithiation progressed, the relative

intensity of Te—Te bonds gradually decreased compared to Te—C bonds. Oppositely, this trend is reversed during delithiation, where the intensity of Te—Te bonds returns. Additionally, in the high-resolution P 2p XPS spectrum (Figure 5c), the intensity of P—P bonds exhibits corresponding attenuation and recoveries during deep discharge and charge, further validating the reaction reversibility [31, 32]. Besides, ex situ XRD tests also demonstrate the as-formed discharge products of BP/Te@C anodes (Figure 5d and Figure S26). Here, the intensities of representative Te peaks at 27.4° , 38.2° , 40.3° , and 43.2° rapidly diminished with lithiation and became negligible at ~ 0.1 V, which indicates the transformation of Te to Li_2Te . Upon delithiation, these peaks gradually appear, showing the reverse conversion of Li_2Te to Te [21]. Moreover, at the pristine state, characteristic peaks of BP can be weakly observed at 26.3° and 34.6° , which vanish with lithiation, signifying the transformation of P to Li_xP . However, a distinct peak for LiP_5 (33.4°) emerges at the fully discharged state (0.1 V) and disappears after charging, confirming the reversible conversion of P. Notably, the peak near 23.0° arises from overlapping diffraction signals of Te, Li_2Te , and Li_3P . To clarify the trend, ex situ XRD data at 1.7, 1.3, 0.1, 1.8, and 2.0 V are further separately analyzed in Figure S26. Here, peaks at 27.4° , 38.2° , and 40.3° reflect the lithiation/delithiation of Te, while peaks at 16.36° and 32.42° correspond to the formation of LiP_5 during lithiation of BP, which disappears during delithiation. Besides, for the peak at 23.6° , a superposition of Li_2Te and Li_3P , showing their emergence during lithiation and disappearance during delithiation. For the peak at 46.06° , overlapping of Te and Li_2Te , it exhibits strong intensity before Te lithiation and weakens during lithiation, showing a prominent Li_2Te peak after full lithiation at 0.1 V. Therefore, the aforementioned ex situ XPS and XRD results indicate that BP and Te in BP/Te@C anodes can form Li_xP and Li_2Te_x during discharge, while provide solid Li_2Te and Li_3P as the final discharge products. Importantly, it should be pointed out that Li_xP and Li_2Te_x are soluble in electrolyte [21, 33], thus showing no obvious XRD diffraction peaks. Besides, the miscibility of these soluble intermediate products can further refine the grain size of pristine anodes during long-time cycling, which has been proved by 3D tomography reconstruction in Figure 3.

In addition, the solid electrolyte interphase (SEI) of BP/Te@C, which is critical for interface compatibility, has been characterized by time-of-flight secondary ion mass spectrometry (ToF-SIMS). Figure 5f demonstrates that a thick organic-dominated SEI is formed on the BP@C anode, which contains species such as CHO_2^- , C_2HO^- , and C_4HO^- (Figure S27). In contrast, BP/Te@C (Figure 5e) features a more compact and elastic SEI layer with an outer region dominated by inorganic components, including LiF_2^- , LiTe^- , LiP^- , LiCO^- , and Li_2F_3^- (Figure S27). Besides, BP/Te@C anode shows a more robust confinement for P^- when compared with BP@C, implying the excellent long-term stability during cycling. Depth profiles of characteristic functional groups in BP/Te@C (Figure 5h and Figure S28) validate the results from 3D ToF-SIMS depth profiling. Notably, the uniform distribution of LiTe^- and LiP^- can be observed in BP/Te@C, with their concentrations increasing with depth, confirming successful internal lithiation. Te is not only a part of SEI, but also indirectly promotes the formation of a more stable, rich in inorganic components, and dense passivation layer by improving the electrochemical interface. These findings indicate

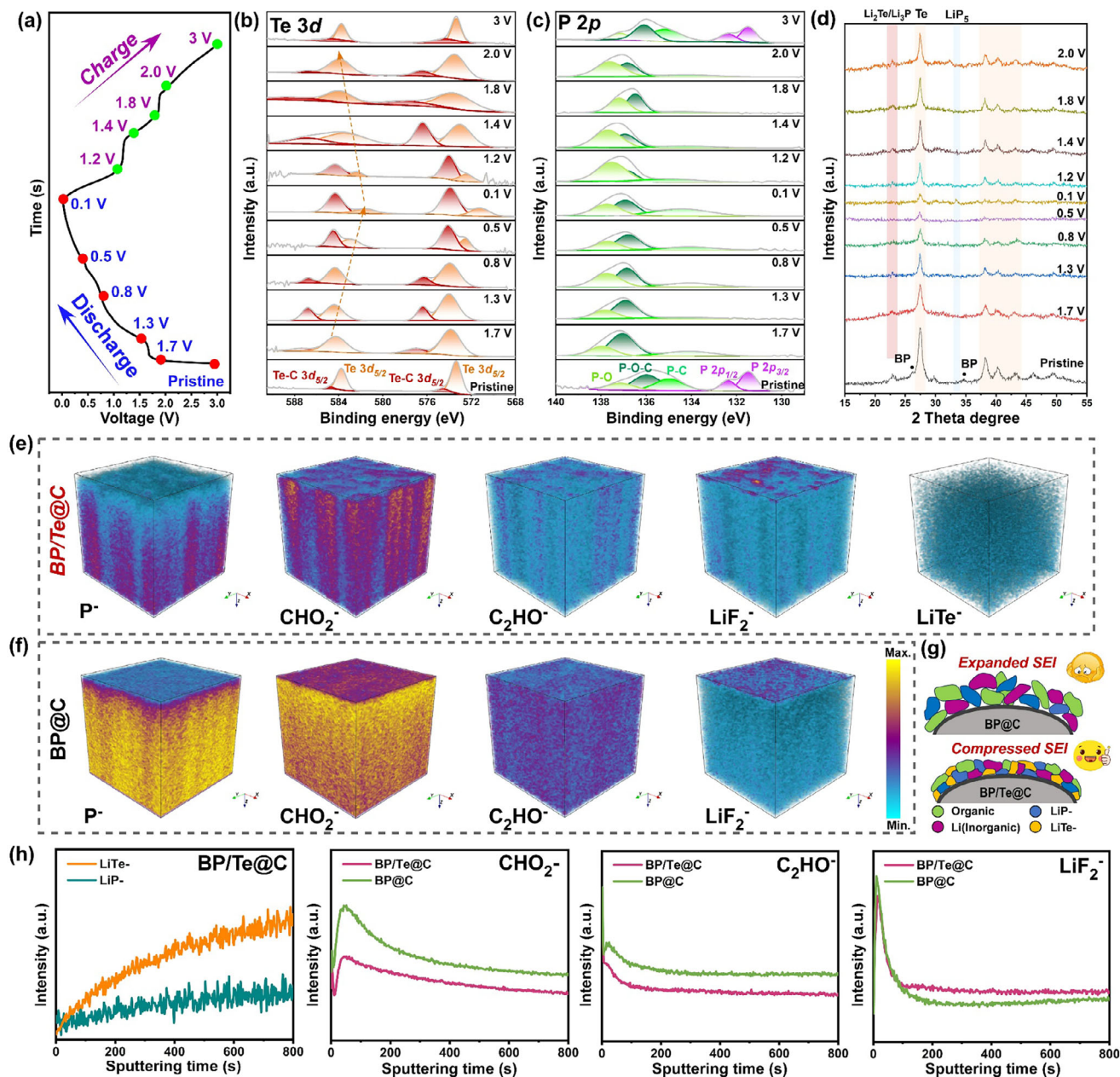


FIGURE 5 | Electrochemical reaction mechanism and in situ products of BP/Te@C anode. (a) Representative charge/discharge curves of BP/Te@C anode for XRD and XPS tests. (b, c) High-resolution ex situ XPS spectra of Te 3d (b) and P 2p (c) refer to the corresponding charge/discharge states of BP/Te@C anode. (d) Ex situ XRD curves of BP/Te@C anode. (e, f) 3D rendering visualization of the selected secondary-ion fragments of BP/Te@C (e) and BP@C (f) by ToF-SIMS. (g) Schematic plot of SEI components for BP/Te@C and BP@C. (h) Normalized depth profile of representative secondary ion fragments obtained from BP/Te@C anode.

that excessive electrolyte decomposition on BP@C yields a thick, loosely packed SEI layer, whereas BP/Te@C forms a uniform, thin, and robust SEI structure (Figure 5g) with the additional Te component, thus suppressing the dendrite growth and improving Li⁺ diffusion [34, 35].

The overall electrochemical performance of BP/Te@C is systematically evaluated by tests on half cells and full cells (Figure 6). Figure 6a presents the first-5th cyclic voltammetry (CV) profiles of BP/Te@C scanned at 0.1 mV s⁻¹ within the voltage window of 0.01–3.0 V. During the anodic sweep, an anodic peak emerges at 1.96 V, corresponding to the delithiation process of Li₂Te → Te

(P1). The anodic peaks at 1.28 and 1.10 V (P2, P3) are attributed to the delithiation of Li₃P → P. In the cathodic sweep, cathodic peaks at 1.72 and 1.60 V (P4, P5) correspond to the formation of polymeric lithium telluride (Li₂Te_x) and Li₂Te, respectively [36, 37]. A broad cathodic peak centered at ~0.6 V is assigned to the lithiation of P to Li₃P [18, 38], indicating the high reversibility and favorable cyclability of BP/Te@C. Figure 6b displays the CV curves of BP/Te@C at varied scan rates (0.01–3.0 V). Five distinct peaks (P1–P5) were selected to calculate the *b*-values via the relationship between peak current and scan rate, yielding *b*-values of 0.62, 0.46, 0.85, 0.60, and 0.81, respectively. These results confirm the synergistic contribution of diffusion-controlled and

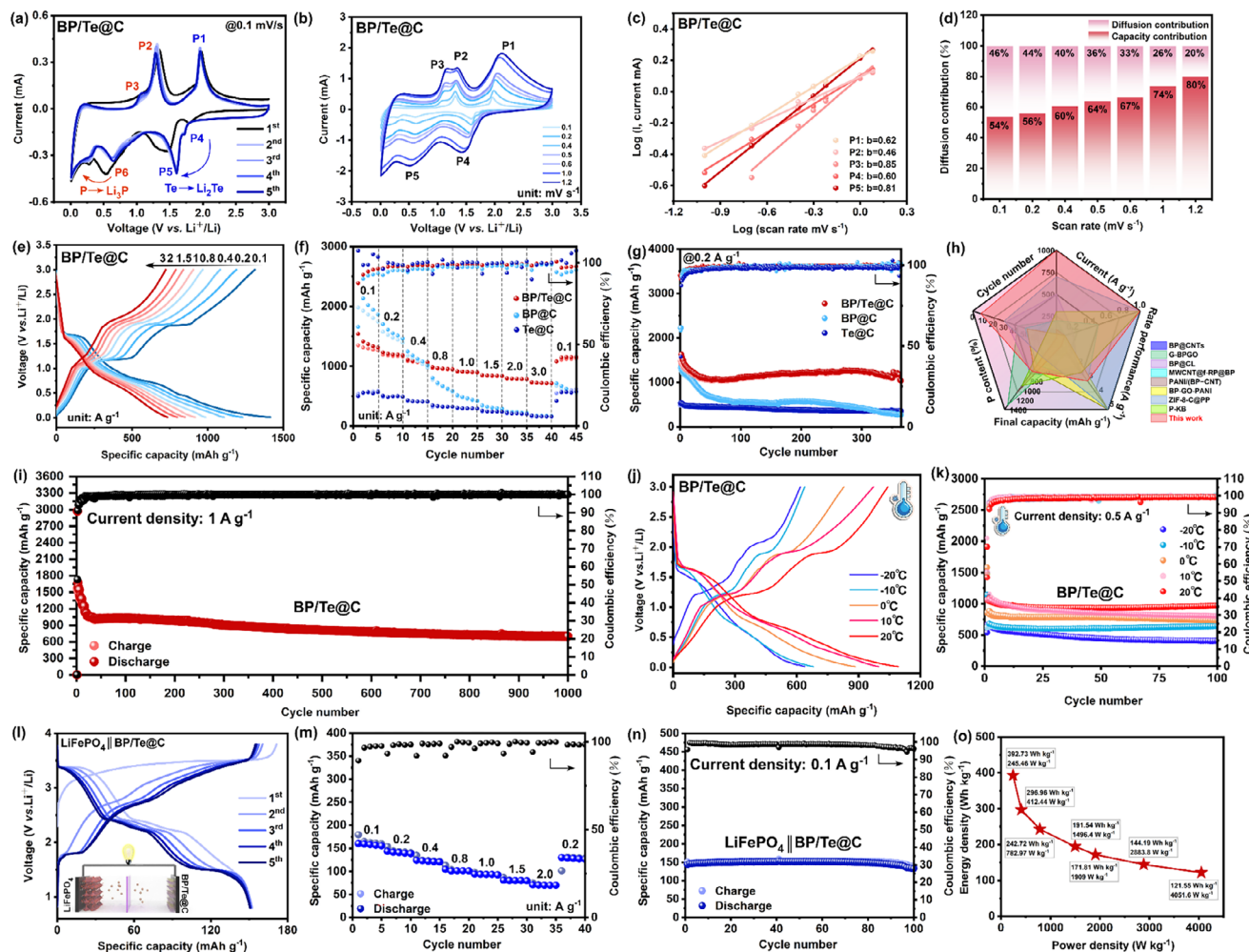


FIGURE 6 | Electrochemical performance. (a) CV curves of BP/Te@C at a scan rate of 0.1 mV s⁻¹ during the first-5th cycles (half cell). (b) CV curves of the BP/Te@C at different scan rates. (c) Log(i) vs. log(v) plots of redox peaks in CV curves of BP/Te@C. (d) Diffusion and capacitance contribution at different scan rates of BP/Te@C. (e) GCD curves of BP/Te@C at different current densities. (f) Rate performances. (g) Cyclic performance of BP/Te@C, BP@C, Te@C at 0.2 A g⁻¹. (h) Comparison of the electrochemical performance of BP/Te@C with reported anode materials. (i) Long-term cycling performance of BP/Te@C at 1 A g⁻¹. (j, k) GCD curves(j) and cyclic performance (k) of BP/Te@C at different temperatures. (l) GCD curves of LiFePO₄||BP/Te@C full cells during the first-5th cycles. Inset: schematic plot of the structure of LiFePO₄||BP/Te@C full cell. (m) Rate performances of LiFePO₄||BP/Te@C. (n) Cyclic performance of LiFePO₄||BP/Te@C. (o) Power and energy densities at varying current densities for LiFePO₄||BP/Te@C.

pseudocapacitive processes in BP/Te@C (Figure 6c). Besides, the capacitance contribution ratio of the electrode (Figure 6d) increases from 54% to 80% as the scan rate rises from 0.1 to 1.2 mV s⁻¹. It indicates that the pseudo-capacitive behavior plays a significant role in the capacitance contribution, especially at high scan rates [39].

Figure 6e shows the galvanostatic charge-discharge (GCD) curves of the BP/Te@C at different current densities, and Figure 6f compares the rate performance of BP/Te@C, BP@C, and Te@C. Although BP@C delivers a high specific capacity of 2133.9 mAh g⁻¹ at 0.1 A g⁻¹, it retains only 173.5 mAh g⁻¹ at 3 A g⁻¹ with poor reversibility (711.1 mAh g⁻¹ upon recovery to 0.1 A g⁻¹). Te@C exhibits better reversibility but retains merely 158.1 mAh g⁻¹ at 3 A g⁻¹. In contrast, BP/Te@C initially delivers 1333.4 mAh g⁻¹, retains 734.4 mAh g⁻¹ at 3 A g⁻¹, and recovers to 1150.7 mAh g⁻¹ at 0.1 A g⁻¹, demonstrating the excellent rate performance of BP/Te@C. It should be noted that BP/Te@C occupies the

smallest amount of BP (8.1 wt.%, refer to Figure S2 and Table S1) in our control experiments under identical conditions. We conducted 1000-cycle GCD measurements on 1:1-BP/Te@C, 3:1-BP/Te@C, and 5:1-BP/Te@C (BP: Te, mole ratio) at a current density of 1 A g⁻¹. The results demonstrate that 1:1-BP/Te@C delivers a stable specific capacity of 700 mAh g⁻¹ after cycling, while 3:1-BP/Te@C and 5:1-BP/Te@C retain capacities of 243.2 and 208.5 mAh g⁻¹, respectively, indicating a pronounced decline in capacity retention with increasing BP content. It can be found that 1:1-BP/Te@C delivers the highest specific capacity, indicating the full utilization of BP in BP/Te@C due to the featured structure of the 3D network (Figure S1 and Table S4). Figure 6g compares the long-cycle performance of different anodes. After 350 cycles at a current density of 0.2 A g⁻¹, the specific capacities of BP@C anode, Te@C anode, and BP/Te@C anode are 270.6, 360.1, and 1111.1 mAh g⁻¹, respectively. This indicates the excellent cycling stability of the BP/Te@C anode. Figure 6i illustrates that after being active at low current density for 20 pre-cycles, BP/Te@C

anode maintains the specific capacity of 700.0 mAh g⁻¹ over 1000 cycles at 1 A g⁻¹ with an average Coulombic efficiency (CE) approaching to 100%. These indicate robust cycling stability and high charge utilization. The rate/cycling performance of BP/Te@C is comparable and even superior to most reported counterparts (Figure 6h and Table S2). This performance is due to the synergy where the carbon network offers a robust framework, and tellurium activates kinetics and interface, enabling high-rate and stable cycling.

Additionally, low-temperature tests further validate the wide-temperature applicability of BP/Te@C anode (Figure 6j,k). At 0.5 A g⁻¹, the specific capacities retain 971.3, 805.7, 737.1, 646.0, and 408.1 mAh g⁻¹ after 100 cycles at 20, 10, 0, -10, and -20°C, respectively, with average CEs near 100%. These confirm the adaptability of BP/Te@C anode over a broad temperature range. The practical feasibility of BP/Te@C is further evaluated in a full cell paired with the commercial LiFePO₄ cathode (details in Experimental Section). Figure 6l shows the first-5th GCD curves of LiFePO₄||BP/Te@C (operating voltage of 0.6–3.8 V), with an inset depicting the full-cell configuration. Figure 6m depicts the rate performance of LiFePO₄||BP/Te@C. The above results exhibit a high specific capacity of 160.3 mAh g⁻¹ at 0.1 A g⁻¹ and 138.7 mAh g⁻¹ at 0.2 A g⁻¹. As the current density increases, the full cells retain a specific capacity of 70.7 mAh g⁻¹ at 2.0 A g⁻¹, and show excellent reversibility, maintaining a specific capacity of 130.2 mAh g⁻¹ when the current density is restored to 0.2 A g⁻¹. These demonstrate the excellent rate performance of LiFePO₄||BP/Te@C. After 100 cycles at 0.1 A g⁻¹ (Figure 6n), the specific capacity still maintains 133.0 mAh g⁻¹ with a high-capacity retention of 90.7%, demonstrating the exceptional cycling stability of full cells. Based on the mass of active materials, the LiFePO₄||BP/Te@C full cells achieve a high energy density of 121.55 Wh kg⁻¹ and a power density of 4051.6 W kg⁻¹ (Figure 6o), outperforming the recently reported phosphorus-based full cells (Figure S29 and Table S3). These results underscore the promising practical application potential of BP/Te@C, which offers a high-performance interfacial lithium diffusion and storage by the unique structural configuration.

3 | Conclusion

In summary, this work reports a distinctive interfacial lithium diffusion and storage mechanism in phosphorus-based anode with a unique 3D network configuration. Taking advantage of the metalloid property and high ductility of additional tellurium, an electron donor–acceptor (D–A) system is formed between black phosphorus, tellurium, and carbon component (BP/Te@C), which possesses a 3D network structure where BP/Te are confined in nanochambers with a 2D diffusion pathway. Specific 3D tomography reconstruction and ToF-SIMS characterizations verify this distinguished and robust configuration during long-term cycling. Additionally, electrochemical tests and theoretical calculations illustrate the interfacial lithium diffusion and storage mechanism in BP/Te@C composites, which further contributes to the grain size refinement and the improved utilization of BP and Te during cycling. Consequently, BP/Te@C anodes deliver a specific discharge capacity of 734.4 mAh g⁻¹ at 3 A g⁻¹ and exhibit well cycling performance over 350 cycles, as well as the well low-temperature adaptability (-20°C). LiFePO₄||BP/Te@C

full cells achieve an outstanding power density of 4051.6 W kg⁻¹ with a high-capacity retention of 90.7%. This work provides a design strategy for advanced BP-based anodes toward next-generation energy storage systems, e.g., sodium-ion batteries and potassium-ion batteries.

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Conflicts of Interest

The authors declare no conflict of interest.

Data Availability Statement

Data sharing not applicable to this article, as no datasets were generated or analysed during the current study.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section.

Supporting File 1: adfm75285-sup-0001-SuppMat.docx.

Supporting File 2: adfm75285-sup-0002-VideoS1.mp4.