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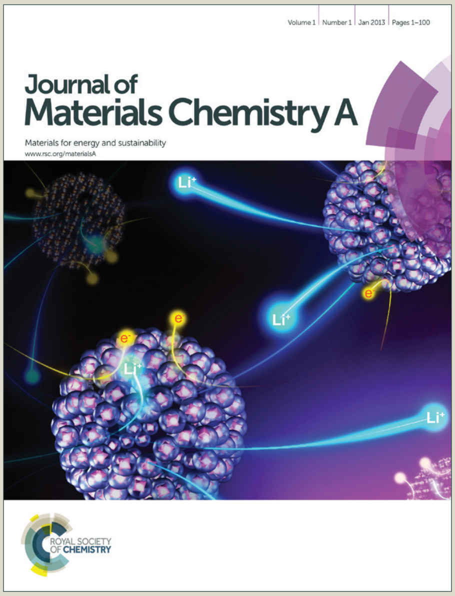

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Lithium-Tellurium Batteries Based on Tellurium/Porous Carbon Composite

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Abstract: For the first time, lithiation/delithiation behavior of tellurium at room temperature was investigated using tellurium/porous carbon (Te/C) composite electrodes. The Te/C composite is synthesized by infusion of liquid tellurium into porous carbon host at a high temperature of 600 ^{o}C under vacuum. Owing to the physical confinement of Te by porous carbon matrix, the Te/C electrode is capable to deliver a reversible volumetric capacity as high as 1400 mAh/cm^3 (224 mAh/g) at 312 mA/cm^3 (50 mA/g) current and maintain 87% of initial capacity after 1000 cycles. Even when the current increases to 12480 mA/cm^3 (2000 mA/g), the Te/C electrode can still provide a volumetric capacity of 500 mAh/cm^3. The high volumetric capacity, long cycle life and good rate capability make Te/C composite a promising electrode for Li-ion batteries.

Keywords: Tellurium, porous carbon, cathode, energy storage, lithium-ion batteries

Introduction

During the last two decades, rechargeable lithium-ion batteries (LIBs) have been wildly used as the power sources for various mobile electronics, including cellular phones, laptop computers and digital cameras.[1-3] In recent years, the rapid development of electric vehicles (EVs), hybrid electric vehicles (HEVs) and large-scale renewable energy storage calls for batteries with high energy/power density, superior cycling and reliability stability, as well as low cost.[4-6] Recently, significant progress has been realized in the development of high capacity electrodes for LIBs. Due to the high capacity, materials based on elements in period 5 (In, Sn, Sb) and group 16 (chalcogens group: S and Se)[7-13] in the period table have been wildly investigated as anodes and cathodes for Li-ion batteries, respectively. However, the lithiation/delithiation behavior of tellurium (Te), the element crossing the period 5 and group 16, has not been studied yet. Owing to a large volume change of these high capacity electrodes, these materials will be cracked or even pulverized into small powders after lithiation/delithiation, thus resulting in capacity decline. The most effective method is to disperse these materials into a porous carbon matrix to mechanically confine these cracked materials, thus improving the cycling stability.

In chalcogens group, sulfur has been extensively studied as a cathode material for Li-S batteries, because of their low cost and high theoretical gravimetric energy density (2570 Wh/kg).[14] However, sulfur cathodes are challenged by three major issues:[15-17] First, the electronic conductivity of S and formed Li_2S are very low, resulting in a poor rate capability and low utilization of active materials. Second, the large volume expansion after lithiation (~80%) brings to particle pulverization and structure reorganization. Third, the dissolution of high order polysulfide intermediates into electrolyte and subsequent migration and reaction with Li anode leads to the transport of low-order polysulfides back to the cathode side, resulting in "shuttle effect". Therefore, current Li-S batteries suffer from low Coulombic efficiency and rapid capacity decay, blocking the commercialization of S cathode in battery industries. Over the years, great efforts have been devoted to address these problems and many strategies have been proposed, such as impregnating sulfur into the porous carbon,[18, 19] encapsulating sulfur with graphene oxide[17] or conductive polymers,[20, 21] entrapping sulfur into the yolk-shell structure,[16] or reducing the solubility of polysulfides by alternative electrolyte or additives.[22]

Recently, it has been reported that selenium (Se), the element in chalcogens family (sulfur, selenium, tellurium), can also be treated as a perspective candidate for cathode material.[23-25] With similar chemical properties but higher electronic conductivity (Se: 1×10^{-4} S/m, S: 5×10^{-16} S/m), lithium-selenium (Li-Se) batteries can deliver a reversible gravimetric capacity of 675 mAh/g theoretically. However, although Se exhibits promising advantages compared with S, the electronic conductivity is still not high enough. Thus, inspired by this, tellurium, another element in the chalcogens family, which possess the highest electronic conductivity among all non-metallic materials (2×10^2 S/m), endows it further advantages compared with sulfur and selenium, such as high utilization rate and better kinetics during charge/discharge process. Despite the gravimetric capacity of tellurium electrode (Li_2Te: 419 mAh/g) is lower than that of selenium (Li_2Se: 675 mAh/g) and sulfur (Li_2S: 1672 mAh/g), the higher density provides it a high volumetric capacity (2621 mAh/cm^3 based on density of 6.24 g/cm^3), which is comparable to that of selenium (3253 mAh/cm^3 based on density of 4.82 g/cm^3) and sulfur (3467 mAh/cm^3 based on density of 2.07 g/cm^3). In fact, the battery packing space is usually limited in portable devices and EV/HEV, thus volumetric capacity is actually more important than gravimetric capacity.[24, 26] Therefore, the superior electronic conductivity as well as high volumetric capacity makes tellurium a potential candidate for electrode material in lithium-ion batteries.

In this study, a novel Te/C composite was synthesized by encapsulating liquid tellurium into the host of porous carbon at a high temperature of 600 ^oC under vacuum. For the first time, the lithiation/delithiation behavior of Te/C composite was investigated in 1 M $LiPF_6$ in a mixture of ethylene carbonate/diethyl carbonate (EC/DEC, 1:1 by volume) electrolyte at room temperature. The as prepared Te/C electrode can deliver and maintain a reversible volumetric capacity of 1400 mAh/cm^3 (224 mAh/g) at a current rate of 50 mA/g with a Coulombic efficiency up to 99% for 1000 cycles. As the current density increases from 50 mA/g to 1000 mA/g, the capacity retained about 47%, and 35% after current density increases up to 2000 mA/g. The high volumetric capacity, long cycle life, excellent Coulombic efficiency and good rate capability of Li-Te battery demonstrates that tellurium is a perspective electrode material candidate for high-energy storage and large-scale applications.

Experimental Section

Synthesis of Tellurium/Porous Carbon Composite

The Te/C composite was synthesized by impregnating liquid tellurium into the porous carbon (Advanced Chemical Supply Inc., USA) matrix through a vacuum-liquid-infusion method. Typically, solid tellurium and porous carbon were mixed with a ratio of 1:1 by weight and sealed in a glass tube under vacuum. Heat treatment at 600 ^{o}C was conducted to melt the solid tellurium (melting point is 450 ^{o}C) and infuse it into the porous carbon.

Material Characterization

Scanning electron microscopy (SEM) images were taken using a Hitachi SU-70 analytical ultra-high resolution SEM (Japan). Transmission electron microscopy (TEM) images were taken by JEOL (Japan) 2100F field emission TEM. Thermogravimetric analysis (TGA) was carried out using a thermogravimetric analyzer (TA instruments, USA) with a heating rate of 10 ^{o}C min^{-1} in air. X-ray diffraction (XRD) pattern was recorded by Bruker Smart1000 (Bruker AXS Inc., USA) using CuKα radiation. Brunauer–Emmett–Teller (BET) specific surface area and pore size/volume were tested using N_2 absorption on Micromeritics ASAP 2020 (Micromeritics Instrument Corp., USA). Raman measurements were performed on a Horiba Jobin Yvon Labram Aramis using a 532 nm diode-pumped solid-state laser, attenuated to give ~900 μW power at the sample surface.

Electrochemical Measurements

Te/C composite was mixed with carbon black and sodium alginate binder to form slurry at the weight ratio of 70:15:15. The electrode was prepared by casting the slurry onto aluminum (Al) foil using a doctor blade and drying at room temperature overnight followed by heating at 100 ^{o}C in vacuum oven overnight. The same method was used to fabricate the pure tellurium and porous carbon electrodes. The thickness of the coating on Al is approximately 20 μm and the loading of electrode is about 1 mg/cm^2. The electrode was cut into circular pieces with diameter of 1.2 cm for coin-cell testing. Li-ion batteries were assembled with lithium as the counter electrode, 1 M $LiPF_6$ in a mixture of ethylene carbonate/diethyl carbonate (EC/DEC, 1:1 by volume) as the electrolyte, and Celgard®3501 (Celgard, LLC Corp., USA) as the separator. Electrochemical performance was tested using an Arbin battery test station (BT2000, Arbin Instruments, USA). Capacity was calculated on the basis of the mass of Te. GITT measurements were carried out by applying a pulse constant current (50 mA/g) with duration of 0.25 h, followed by 6 h relaxation to reach an equilibrium potential. Cyclic voltammograms at a scan rate of 0.1 mV/s between 0.8 and 2.5 V (vs. Li/Li^+) were tested using Solartron 1260/1287 electrochemical interface (Solartron Metrology, UK).

Results and discussion

Figure 1a and b show the SEM and TEM images of Te/C composite, which exhibits a well-formed flake-like shape. As compared with the pure porous carbon, illustrated in the inserted figure of Figure 1a, no morphology change is observed after tellurium is infused into the porous carbon host, implying that most of the tellurium has been filled inside the porous carbon, which has been confirmed by the high resolution TEM images of Te/C composite, see Figure 1c. The elemental mapping images (Figure 1d, e and f) reveal that tellurium is homogeneously distributed in the porous carbon matrix and no tellurium accumulation is observed on the carbon surface, illustrating a perfect Te infiltration into porous carbon. The uniform distribution and free of surface accumulation as well as excellent electronic conductivity of carbon and tellurium itself ensure a high utilization of active material and good kinetics during electrochemical reactions.

The content of tellurium in Te/C composite is revealed to be 50 wt% by the TEM elemental analysis (Figure 1g), which is further confirmed by the TGA measurement results as shown in Figure 1h. There is no weight loss observed from room temperature to 350 ^{o}C, indicating that the composite is stable up to 350 ^{o}C in air, and no Te oxidation and carbon decomposition reaction occurred in 25-350 ^{o}C. The weight loss from 350 to 480 ^{o}C is the combination effects of weight loss from the carbon oxidation to carbon dioxide, $C + O_2 = CO_2$ and weight increase due to the oxidation of tellurium, $Te + O_2 = TeO_2$. The Te content can be determined to be 50% using the following equation:

$$\text{Te (wt\%)} = \frac{\text{molecular weight of Te}}{\text{molecular weight of TeO}_2} \times \frac{\text{final weight of TeO}_2}{\text{initial weight of Te/C}} \times 100$$

The tellurium impregnation into porous carbon is also evidenced by the Brunauer–Emmett–Teller (BET) analysis, shown in Figure 2a. The specific surface area is calculated from the adsorption data in the relative pressure range of 0.05 to 0.3. Total pore volumes were calculated from the amount of adsorption at a relative pressure, P/P_0, of 0.90. Micro-pore volumes were calculated using the *t*-plot method. The porous carbon has a large BET surface area of 1416 m^2/g and high pore volume of 0.44 cm^3/g. After infusion of tellurium, the BET surface area decreased dramatically to 353 m^2/g, and the pore volume declined to 0.06 cm^3/g, indicating that most of the pores have been occupied by Te. This result could also be confirmed by the pore size distribution curves (Figure 2b) which were determined from the adsorption branches of the isotherms using density functional theory (DFT), i.e., apparent decrease of the pore size was observed after porous carbon was infused by Te.

The infused tellurium in porous carbon exists in crystal structure as demonstrated by the XRD patterns of Te/C composite in Figure 2c. For comparison, the XRD patterns of both pristine Te and pure porous carbon are also shown in Figure 2c. The Te/C composite displays well-defined diffraction pattern as the pristine tellurium (JCPDA File No. 79-0736). The nature of tellurium in composite is further characterized using Raman spectroscopy. For comparison, porous carbon (C), pristine Te (Te), and manually mixed Te-C composite (Te&C) at the same ratio to the Te/C composite in room temperature, are also analyzed using Raman spectroscopy, (Figure 2d). For porous carbon, two broad peaks at 1350 cm^{-1} and 1590 cm^{-1} are attributed to the disordered (D) and graphitic (G) carbon, demonstrating that the porous carbon is partially graphitized. Typical Raman spectrum pattern of pristine tellurium is characterized by three peaks from 80 to 700 cm^{-1}, which are probably attributed to E_2 bond-bending, A_1 bonding-bending and A_1 bond-stretching modes of the crystalline trigonal tellurium, with a highly anisotropic crystal structure consisting of helical chains of covalently bound.[27, 28] For Te/C composite, after infusing tellurium into porous carbon host, two small peaks from 400 to 700 cm^{-1} disappear, although the vibration peaks around 117 cm^{-1} of Te remains,, implying the Te not only physically confined in porous carbon matrix, but also chemically bonded to carbon stabilizing tellurium in the supporting carbon matrix. The chemical bonding is probably induced by high temperature infusion process since the Te peak at 400 to 700 cm^{-1} still exists in Te&C mixture prepared at room temperature (Figure 2d). The mixed Te&C exhibits simple addition of tellurium and carbon Raman spectra separately, no any changes on the forms of Te itself. Therefore, the confinement of the porous carbon at high temperature remarkably altered the structure of tellurium.

The electrochemical performance of the Te/C composite was investigated in coin cells using lithium metal as the counter electrode, and 1 M $LiPF_6$ in a mixture of ethylene carbonate/diethyl carbonate (EC/DEC, 1:1 by volume) as electrolyte. All electrochemical experiments are conducted at room temperature, and all capacity is calculated based on the mass or volume of tellurium. For Te/C composite, the current density was calculated on the basis of total weight of tellurium and porous carbon. Figure 3 shows the charge/discharge profiles of Te/C composite between 0.8-2.5 V at a current density of 50 mA/g, with the comparison of both pristine Te and porous carbon electrodes at the same voltage range and current density. As demonstrated in Figure 3a, pristine Te electrode displays a voltage plateau at 1.6 V in discharge and at 1.8 V in charge, with a discharge volumetric capacity of 1685 mAh/cm^3 (270 mAh/g) and charge volumetric capacity of 936 mAh/cm^3 (150 mAh/g) in the first cycle. The plateau at 0.9 V is attributed to decomposition of electrolyte and formation of solid electrolyte interphase (SEI). For Te/C composite (Figure 3b) , two slope-shaped voltage plateaus locating at 1.6 V and 1.4 V in discharge, 1.7 V and 1.9 V in charge process are observed. The extra voltage plateau at 1.4 V for discharge and 1.7 V for charge cannot be attributed to carbon since no voltage plateau above 1.0 V is observed in electrode made by pure porous carbon, see Figure 3c. The transition from well-defined single plateau of pure Te to two slope-shaped plateaus of Te/C is probably due to the interaction between Te and porous carbon, which is also observed in S/C and Se/C composites.[17, 19-20] The interaction between Te and porous carbon was confirmed by Raman measurement in Figure 2c. The Te/C composite delivers a volumetric capacity of 6739 mAh/cm^3 (1080 mAh/g) in first discharge and 1872 mAh/cm^3 (300 mAh/g) in first charge process. The large irreversible capacity formed in the first cycle is mainly attributed to the formation of SEI film on the porous carbon,[25] which is confirmed by the high irreversible capacity of porous carbon itself in Figure 3c. Since the porous carbon only provides 6 mAh/cm^3 volumetric capacity (20 mAh/g) (Figure 3c), the contribution from carbon to the total capacity of Te/C is negligible.

Aside from charge/discharge profiles, cyclic voltammetry (CV) actually reveals more details about the electrochemical reaction during the lithiation/delithiation process. Figure 4a demonstrates the CV of the Te/C composite at a scan rate of 0.1 mV/s between 0.8 V and 2.5 V. Two redox couples are observed, with the reduction/oxidation peaks locating at 1.6/1.9 V, and 1.4/1.7 V separately, which is in good agreement with the double slope plateaus of charge/discharge curves in Figure 3b. After the first cycle, the two cathodic peaks shift to a higher voltage, while the anodic peaks shift slightly to a lower voltage, reducing the potential hysteresis for lithiation and delithiation. The reduction of overpotential after first cycle has been observed in many high capacity electrodes, which is believed to be associated with the relaxation of stress/strain induced by volume expansion after first lithiation through deformation.[25] Moreover, the stable cathodic/anodic peaks after the first cycle imply a high cycling stability.

The cycling performance of Te/C composite is investigated by galvanostatic charge and discharge of the Te/C composite electrodes between 0.8 V and 2.5 V at a current density of 50 mA/g for 1000 cycles, as shown in Figure 4b. The Te/C composite provides a volumetric capacity of 2022 mAh/cm^3, corresponding to a specific capacity of 324 mAh/g in the first delithiation process, which is close to the theoretical capacity of Te. In addition, a reversible volumetric capacity of 1400 mAh/cm^3 (224 mAh/g) is still retained even after 1000 cycles, with the Columbic efficiency always approaching up to 99% except the first cycle, indicating an excellent cycling stability. For comparison, the cycling stability of pristine Te electrode is also tested under the same electrochemical condition, which, however, suffered severely from rapid capacity fading, retaining only 7% of the initial capacity after mere 20 cycles. Therefore, after mechanical confinement and chemical stabilization of tellurium in the supporting carbon host, the Te/C composite possesses superior electrochemical performance with a high volumetric capacity and long cycle life.

In addition to the good cycling stability, the Te/C electrode also displays a high rate capability. As shown in Figure 4c, when the current density increases from 50 to 1000 mA/g and 2000 mA/g, the volumetric capacity of the Te/C composite retains about 47% and 35% of capacity at 50 mA/g, respectively. Even when the Te/C composite is charged/discharged at 4000 mA/g, 17% capacity retention can still be obtained. The superior volumetric capacity, high Columbic efficiency, long cycle life and good rate capability of Te/C composite demonstrates that tellurium is a promising electrode material candidate for Li-ion batteries, especially in large-scale energy storage applications.

To investigate the reaction kinetics of Te/C composite at different lithiation/delithiation states, the over potential and reaction resistance of Te/C electrodes during charge/discharge were analyzed using the galvanostatic intermittent titration technique (GITT). During the GITT measurements, the Te/C electrodes were charged/discharged by a series of constant current pulse of 50 mA/g with equal duration period of 0.25 h. Following each pulse, the cells were left at open circuit state for 6 h to reach the equilibrium potentials. Figure 5a demonstrates the potential response of Te/C electrode during GITT measurement in Li-ion batteries. The dotted-lines represent the equilibrium open circuit potentials (OCPs). The reaction resistance at different Li ion insertion and extraction levels is also calculated by dividing the over potential by the pulse current (Figure 5b). The overpotential and reaction resistances of Te/C electrode shows two state increases with state of charge/discharge. For lithiation, the reaction resistance level-off after 50% of discharge, while the resistance rises expeditiously at the end of charge. The different behavior of reaction resistance between charge and discharge may be attributed to the electronic contact resistance that decreases when volume expansion during lithiation and increases with volume contraction during delithiation.

Conclusion

In summary, a novel tellurium/porous carbon (Te/C) composite is synthesized at a high temperature of 600 ^{o}C under vacuum. The designed Te/C composite is employed, for the first time, as electrode material in lithium-ion batteries, which exhibits excellent electrochemical performance in commercialized carbonate-based electrolyte. Owing to the physical confinement and chemical stabilization of tellurium in the supporting carbon matrix, the Te/C electrode can deliver a reversible volumetric capacity of 1400 mAh/cm^3 (224 mAh/g) and a high Coulombic efficiency near 99% over up to 1000 cycles without any further capacity decay. The high volumetric capacity, long cycle life, excellent Coulombic efficiency and good rate capability of Te/C composite demonstrate that Li-Te batteries are promising candidates for applications in large-scale and high-energy storage.

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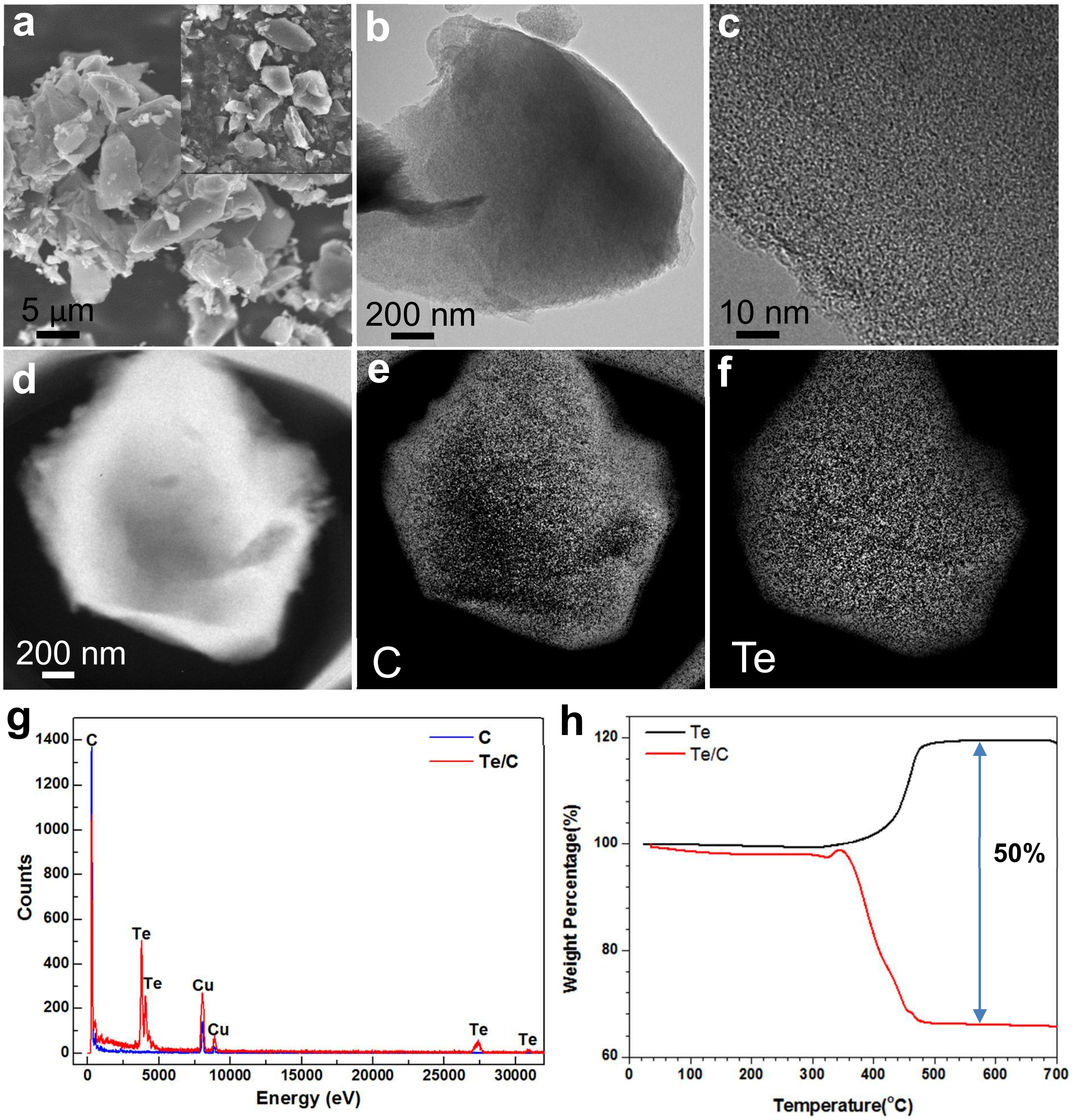

Figure 1. (a) SEM images of the tellurium-impregnated porous carbon composite, inserted image is the pure porous carbon. (b) and (c) TEM images of Te/C composite. (d) TEM and elemental mapping images of Te/C composite: (e) carbon and (f) tellurium. (g) TEM elemental analysis. (h) TGA results of Te/C composite and pristine Te.

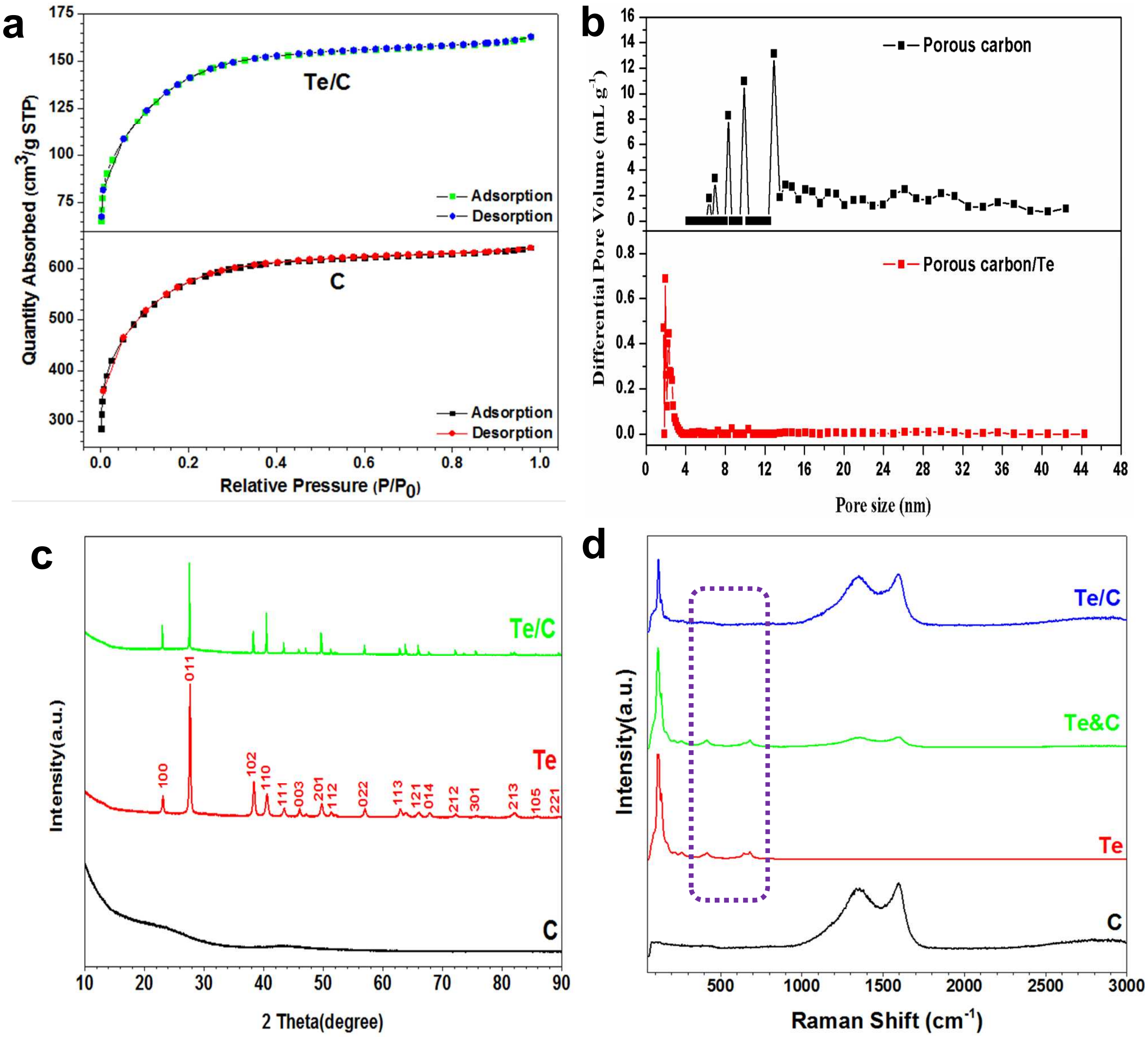

Figure 2. (a) BET Adsorption and desorption curve of the porous carbon and Te/C composite. (b) DFT pore size distribution of porous carbon and Te/C composite. (c) XRD pattern of porous carbon, pristine Te and Te/C composite. (d) Raman spectra of porous carbon, pristine Te, manually mixed Te&C, and Te/C composite.

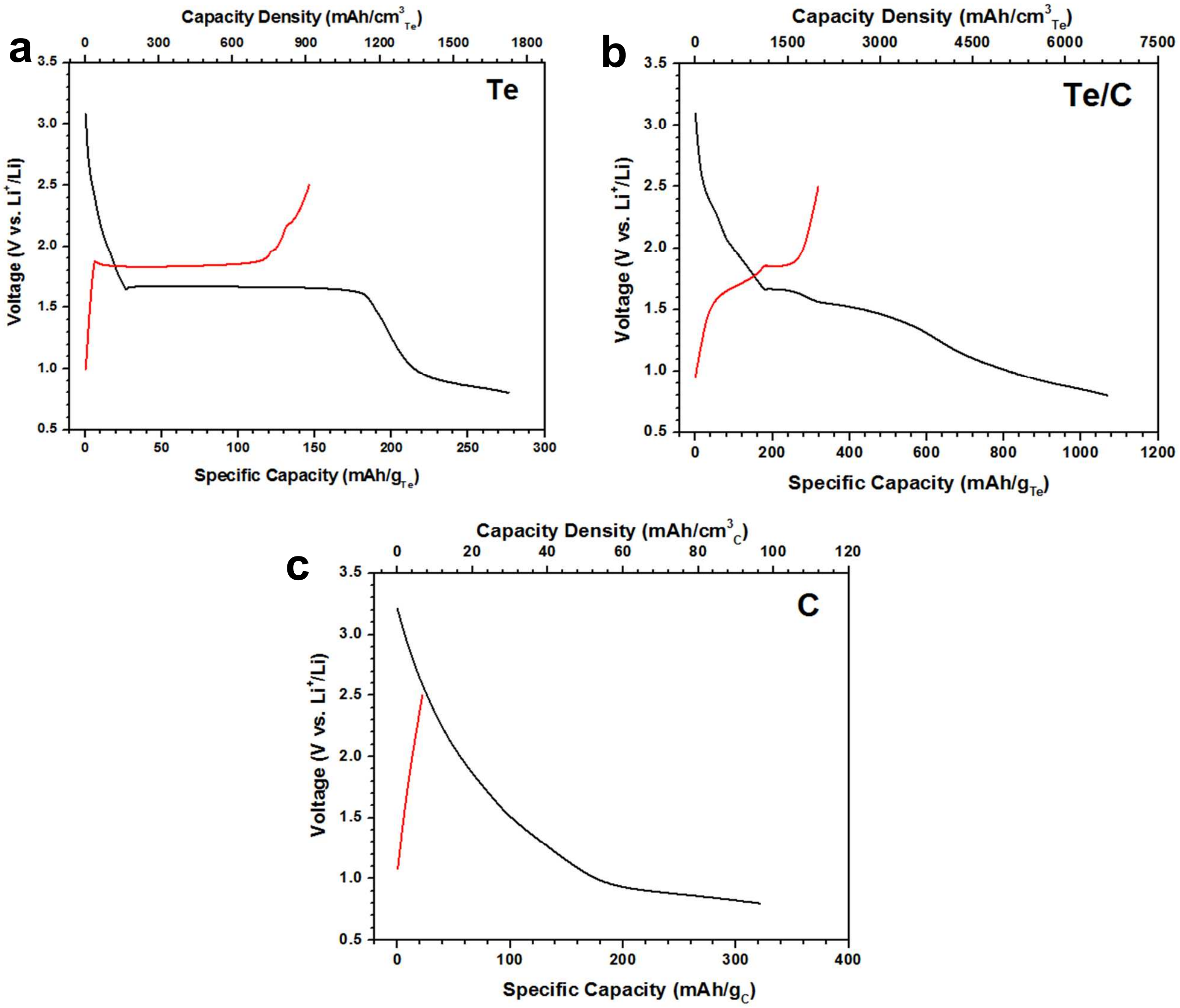

Figure 3. Charge/discharge profiles at the first cycle of (a) pristine Te electrode, (b) Te/C composite electrode and (c) porous carbon electrode.

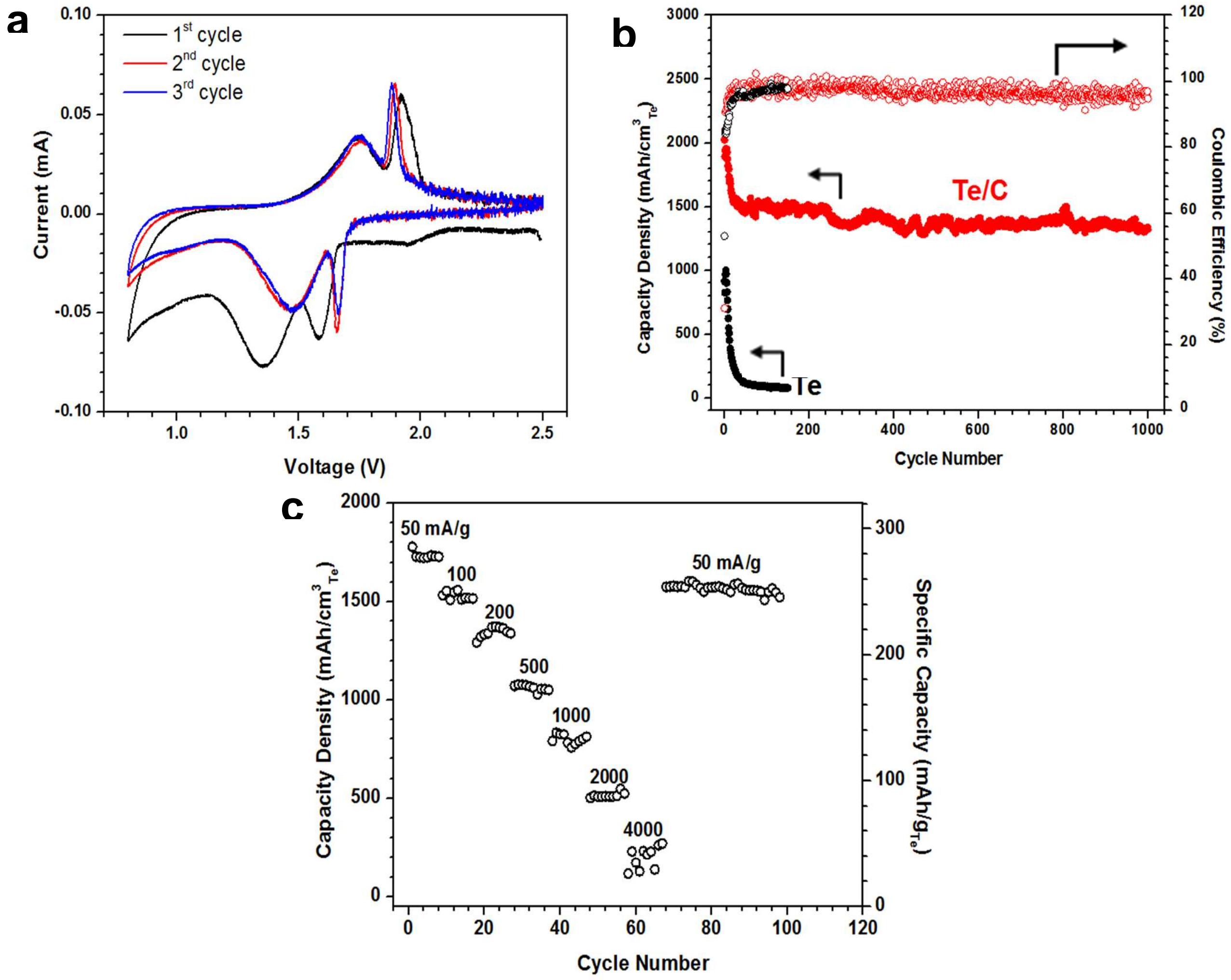

Figure 4. (a) Cyclic voltammograms of the Te/C composite electrode in the initial three cycles. (b) Cycling performance and Coulombic efficiency of the Te/C composite and pristine Te electrode. (c) Rate capability of the Te/C composite electrode in Li-Te batteries.

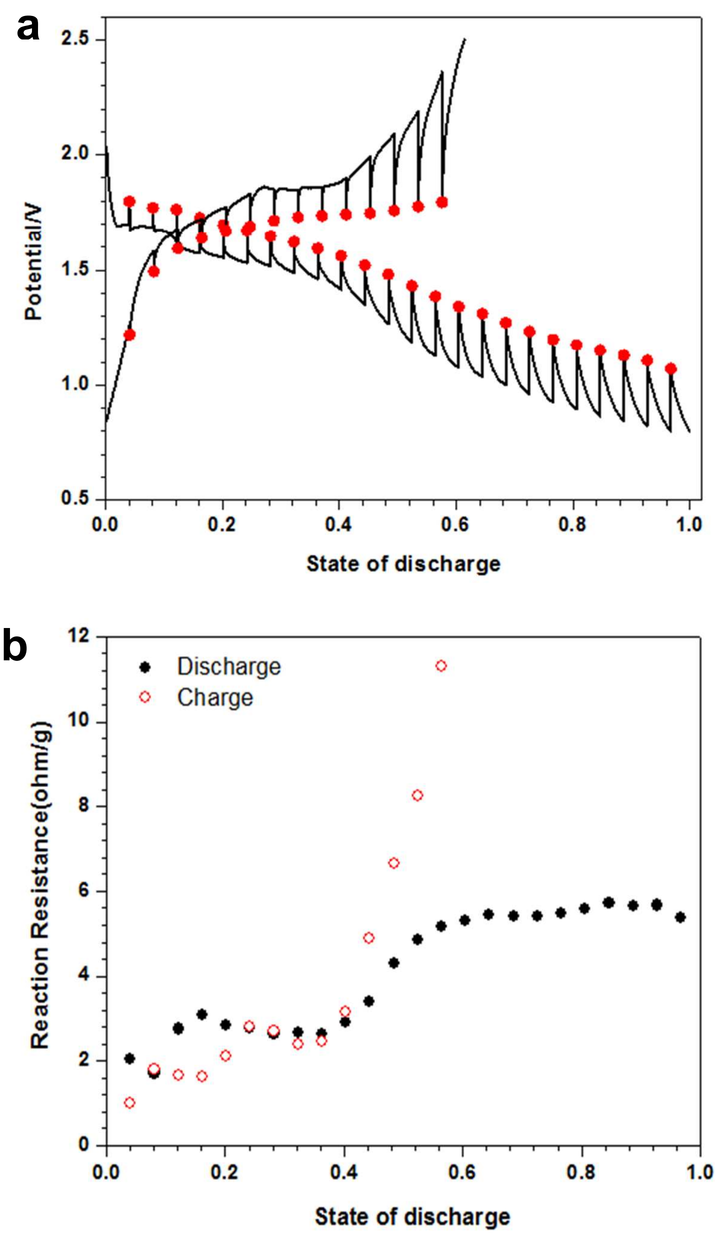

Figure 5. (a) Potential response and (b) reaction resistance of the Te/C composite electrode during GITT measurement. Note: current density was calculated on the basis of total weight of porous carbon and Te.