Electronic Supplementary Material (ESI) for Inorganic Chemistry Frontiers.

Hierarchical Interlayer-Expanded $MoSe_2$/N-doped Carbon Nanorods for High-Rate and Long-Life Sodium and Potassium Ion Batteries

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Experimental section

Preparation of MoO_3•EDA nanorods and MoO_3 nanorods [1, 2]: Dissolved 1.24 g $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$ and 0.84 g ethylenediamine (EDA) in 30 mL water in a round-bottomed flask under stirring. 1 mol L^{-1} hydrochloric acid was dropwise added into the above solution until appearing white precipitate. Then, the formed suspension was heat-treated at 50 ºC in oil bath for 4 h. The products were filtered and washed with water and ethanol for 3 times, dried at 60 ºC overnight to obtain MoO_3•EDA nanorods. Dissolved 1.4 g $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$ in 30 mL water and 6 mL concentrated nitric acid into a 100 mL hydrothermal synthesis reactor, heated at 200 ºC for 20 h, then MoO_3 nanorods were obtained,

Preparation of $MoSe_2$/N-C nanorods and $MoSe_2$/C composites: Weighting 0.2 g MoO_3•EDA and equal molar quantities of selenourea, dissolve them in 30 mL water, stirring until dissolved., the formed suspension was heat-treated at 200 ºC for 12 h in 50 mL stainless still. The obtained product was annealed in Ar/H_2 atomosphere at 600 ºC for 12 h to be as-prepared $MoSe_2$/N-C nanorods. The control $MoSe_2$/C composite was synthesized using the same selenide process with addition of glucose.

Materials characterization: The XRD patterns of samples were obtained on X-ray powder diffraction (Philips) with Cu Kα radiation. Microstructure morphology is characterized by field-emitting scanning electron microscope (FESEM, JEOL-JSM-6700F). nanostructure morphology is characterized by transmission electron microscopy (TEM, Hitachi H7650 and HRTEM, JEOL 2010). Raman spectral was collected by Confocal Laser Micro-Raman spectrometer (JYLABRAM-HR). The elemental valence of samples was collected by X-ray photoelectron spectroscopy (XPS) on ESCALAB 250 spectrometer with Al Kα radiation.

Electrochemical measurements: The anode electrode was prepared by ball-milling the mixture of active material, super P and polyvinylidene fluoride (PVDF) with N-methyl pyrrolidinone (NMP) as solvent. The weight ratio of these component is 7:2:1. The well-mixed slurry was pasted to Cu foils and dried in a vacuum oven at 80 °C overnight. Each electrode was cut into circular plate with area of $1cm^2$ and showed a loading mass density of 1.5 mg cm^{-2}. All 2016 coin cells were assembled in a high-pure Ar-filled glove box with Na/K foil as counter electrode. The electrolyte used in these SIBs is 1 M $NaClO_4$ dissolved in ethylene carbonate and dimethylcarbonate (EC/DMC, volume ratio 1:1). The electrolyte used in these PIBs is 0.8 M KPF_6 dissolved in ethylene carbonate and

diethylcarbonate (EC/DEC, volume ratio 1:1). Cyclic voltammetry curves and Nyquist plots were performed on Chenhua electrochemical workstation (CHI 660E, Shanghai). The electrochemical performance involving cycle stability and rate capability was recorded on LAND-CT2001A instruments.

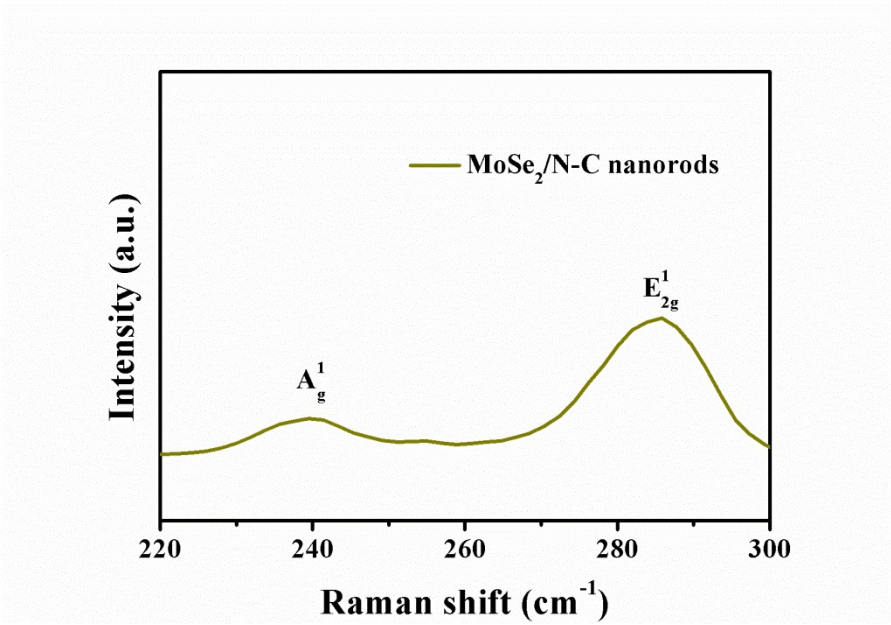

Figure S1. The Raman spectra of the $MoSe_2$/N-C nanorods.

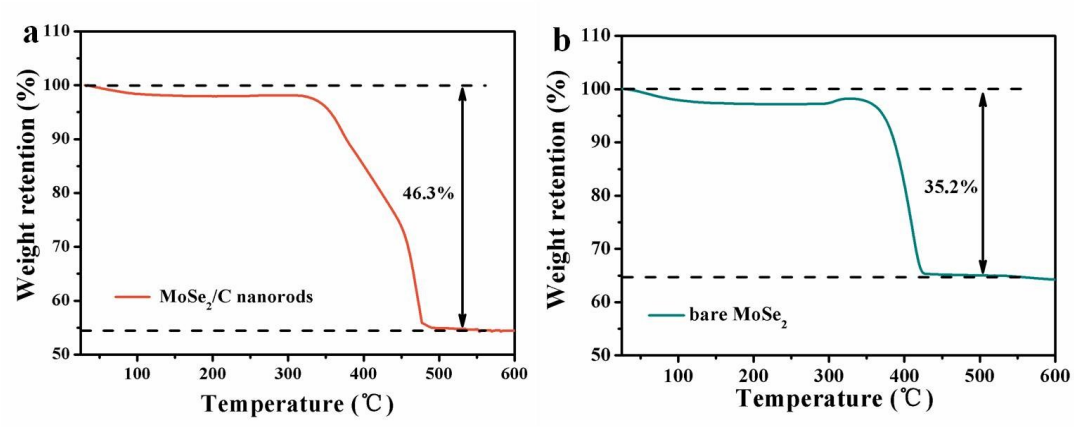

Figure S2. The TG curves of $MoSe_2$/N-C nanorods and bare $MoSe_2$.

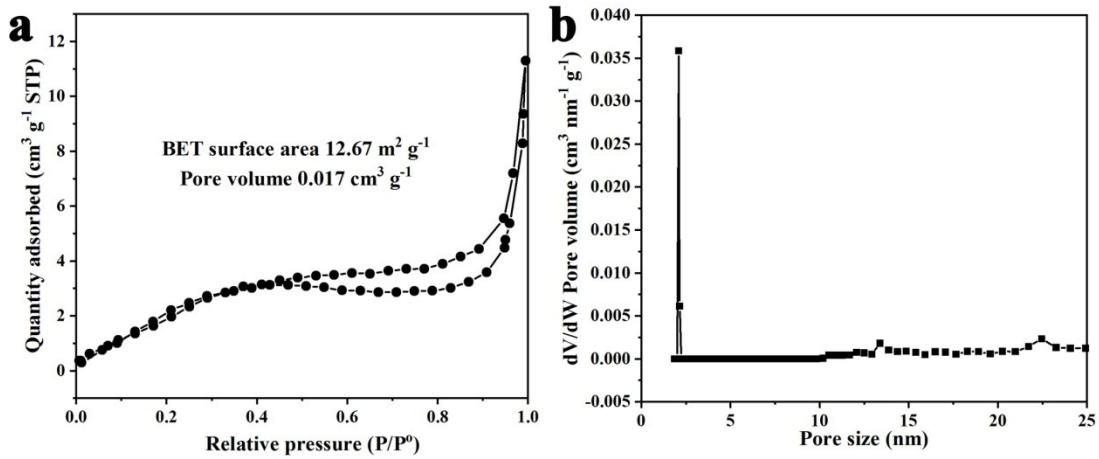

Figure S3. The nitrogen adsorption-desorption isotherm and pore-size distribution curve.

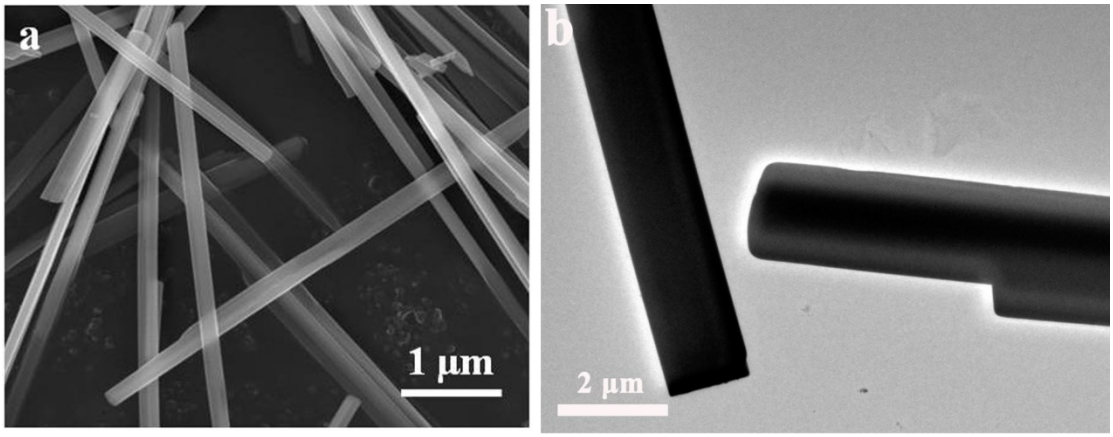

Figure S4. (a) SEM and (b) TEM images of the MoO_3•EDA precursor.

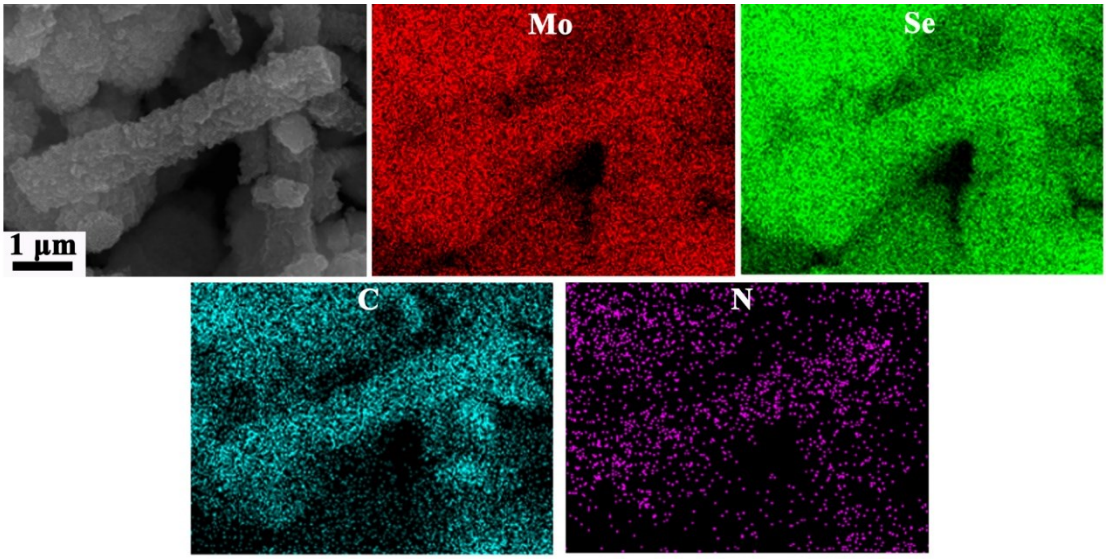

Figure S5. SEM image and EDS mapping image of $MoSe_2$/N-C nanorods composite.

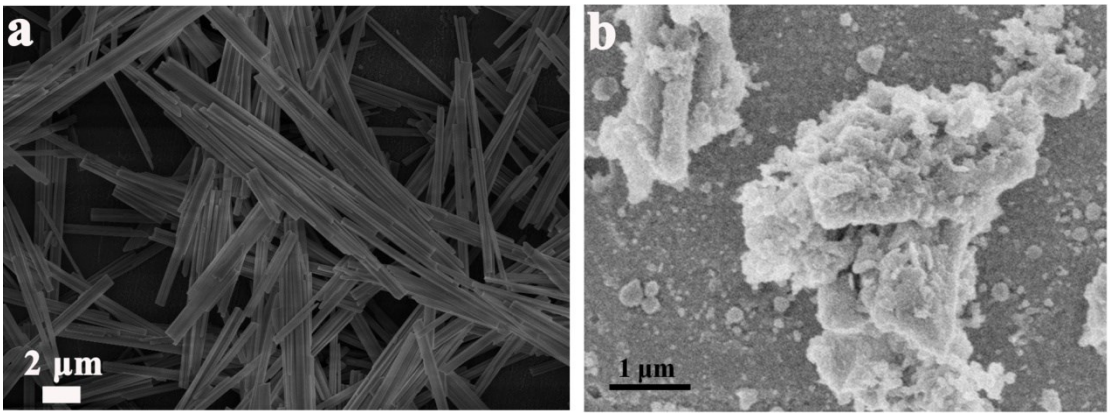

Figure S6. (a) The SEM images of MoO_3 nanorods precursor and (b) $MoSe_2$/C composites.

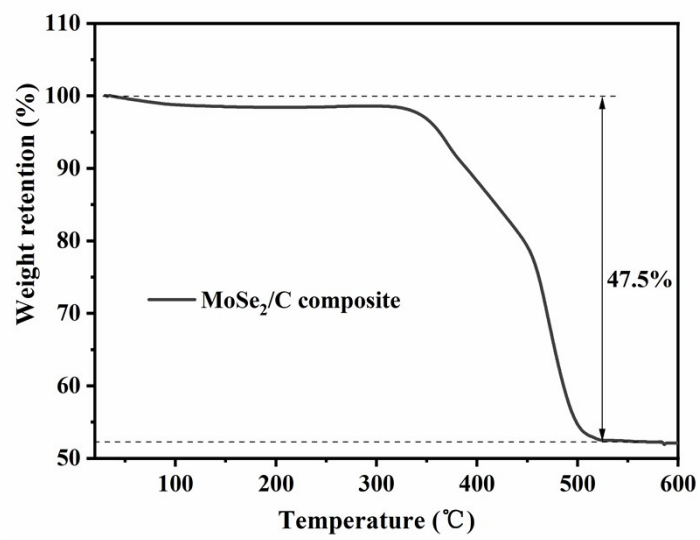

Figure S7. The TG curve of $MoSe_2$/C composite.

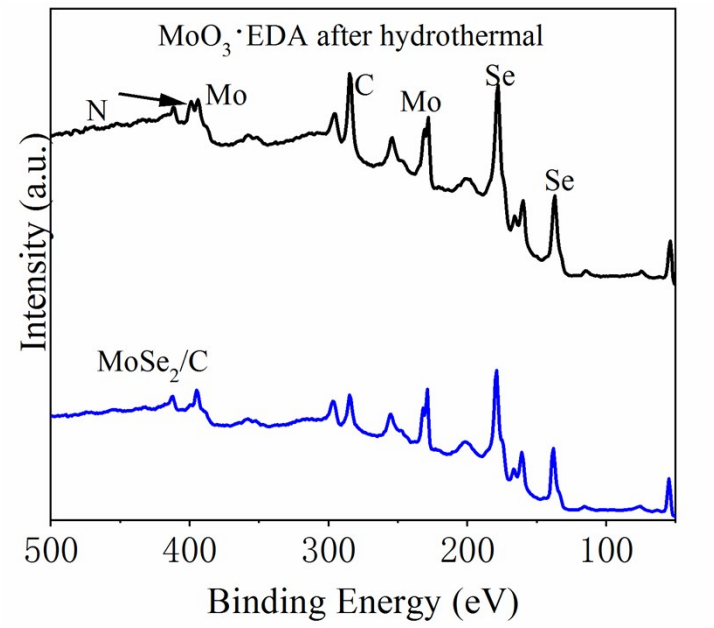

Figure S8. XPS spectra of MoO_3•EDA after hydrothermal and $MoSe_2$/C composite.

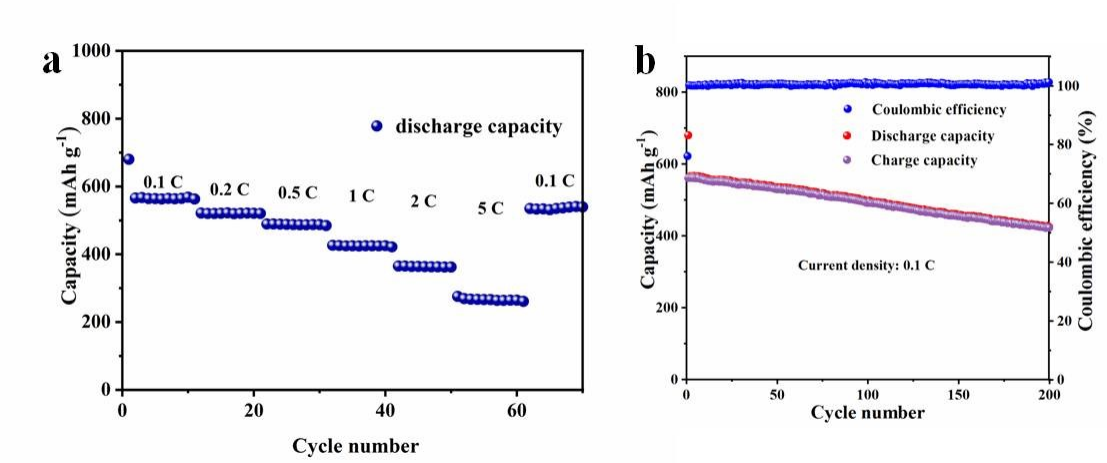

Figure S9. Electrochemical behavior of the $MoSe_2$/C composite in SIBs. (a) rate performance, (b) cycle performance under a rate of 0.1 C.

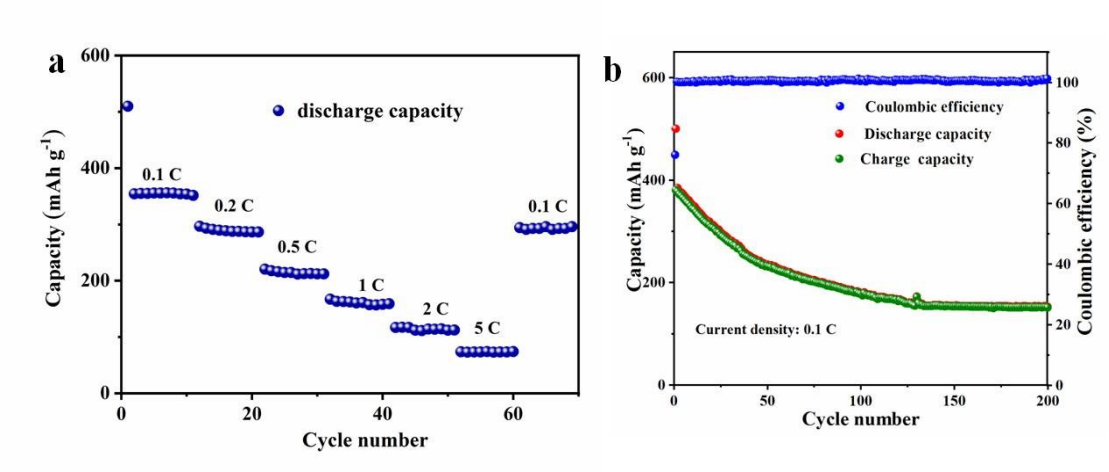

Figure S10. Electrochemical behavior of the $MoSe_2$/C composite in PIBs. (a) rate performace, (b) cycle performance under a rate of 0.1 C.

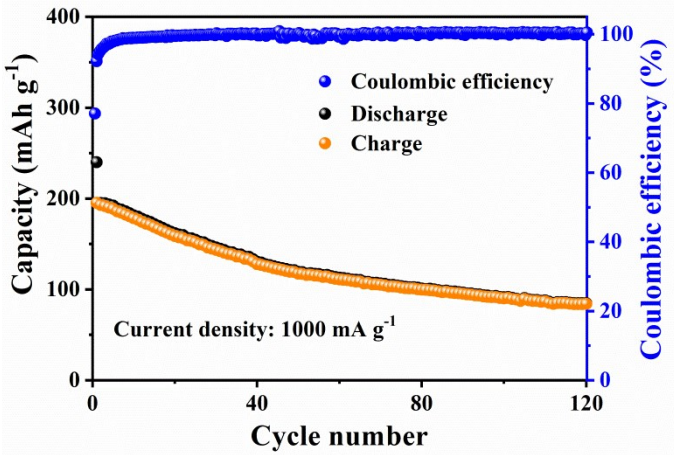

Figure S11. The cycle stability of $MoSe_2$/C composite for potassium storage at a high current density of 1 A g^{-1}.

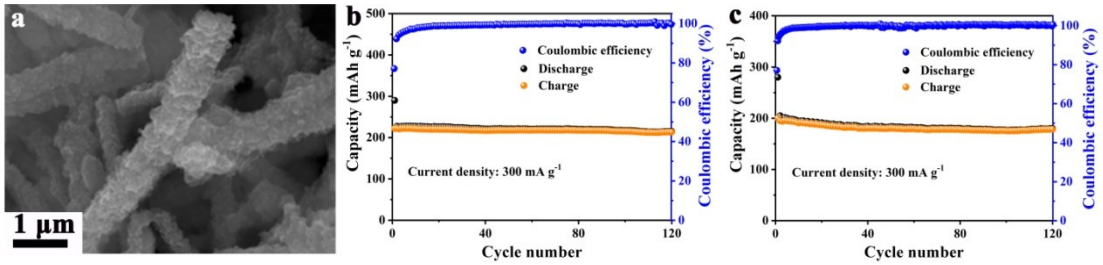

Figure S12. a) SEM image of obtained N-doped carbon, b) and c) cycle performance of N-doped carbon in SIBs and KIBs at a current density of 300 mA g^{-1}.

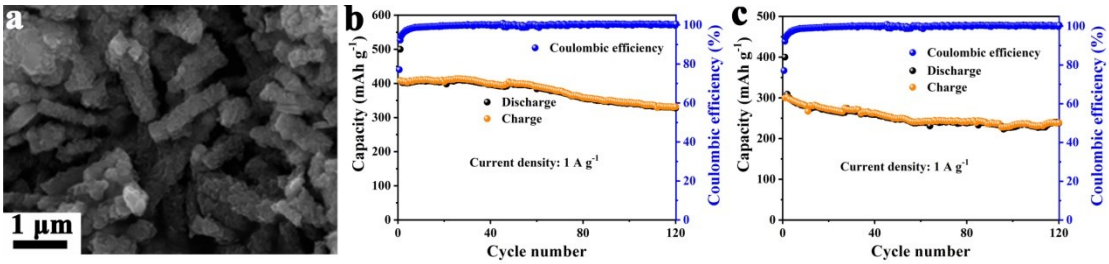

Figure S13. a) SEM image of heat-treated $MoSe_2$/N-C nanorods, b) and c) cycle performance of as-obtained hierarchical rod-like $MoSe_2$ in SIBs and KIBs at a current density of 1 A g^{-1}.

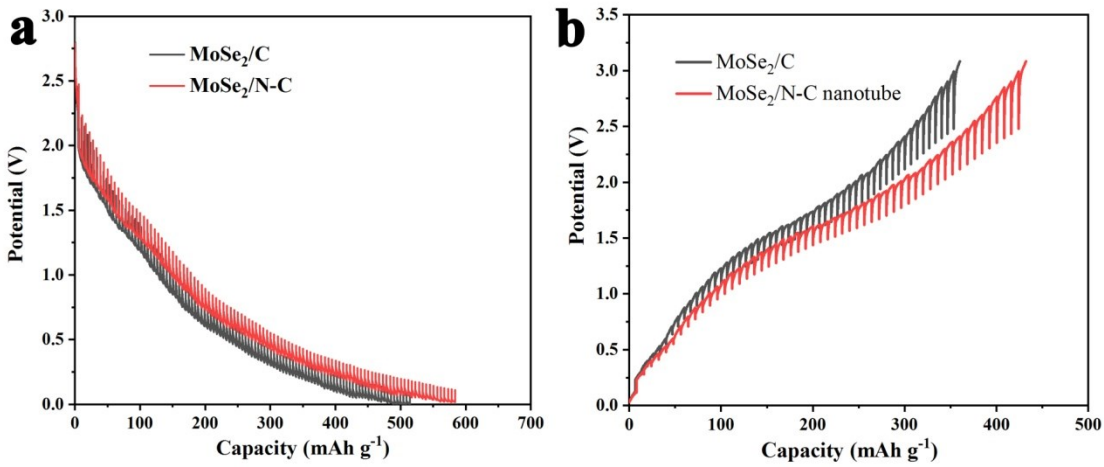

Figure S14. GITT curves of $MoSe_2$/N-C nanorods and $MoSe_2$/C during initial discharge and charge process, respectively.

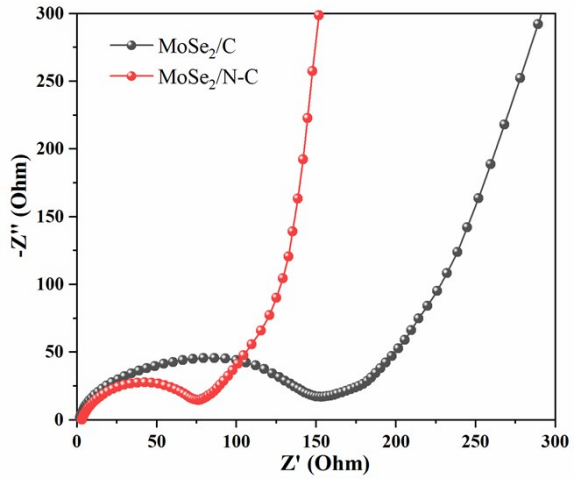

Figure S15. Nyquist plots of $MoSe_2$/N-C nanorods and $MoSe_2$/C.

Reference

1 S. Zhuo, Y. Xu, W. Zhao, J. Zhang and B. Zhang, Hierarchical Nanosheet‐Based MoS2 Nanotubes Fabricated by an Anion‐Exchange Reaction of MoO3‐Amine Hybrid Nanowires, *Angew. Chem. Int. Edit.* 2013, **52**, 8602-8606.

2 X. Zhang, X. Li, J. Liang, Y. Zhu and Y. Qian, Synthesis of MoS2@ C nanotubes via the kirkendall effect with enhanced electrochemical performance for lithium ion and sodium ion batteries, *Small*, 2016, **12**, 2484-2491.