

Interfacial covalent bonding of MXene-stabilized Sb₂Se₃ nanotube hybrid with fast ion transport for enhanced sodium-ion half/full batteries

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Experimental Section

Synthesis of Sb₂S₃: 4 mmol SbCl₃, 8 mmol L-cysteine and 8 mmol Na₂S·9H₂O were dissolved in 80 mL deionized water and stirred for 3 hours to form a uniform solution. The above solution was transferred to a 100 mL hydrothermal kettle and kept at 180 °C for 12 h. Cooling to room temperature, the brown product was centrifuged with deionized water and ethanol for three times and vacuum dried overnight at 60 °C.

Synthesis of Sb@NC: 100 mg of Sb₂S₃ and 50 mg of dopamine hydrochloride were dissolved in 100 ml buffer solution (10 mM), stirred for 6 h, centrifuged three times with deionized water and ethanol, and dried overnight in vacuum at 60 °C. The product Sb₂S₃@polydopamine (Sb₂S₃@PDA) was annealed at 500 °C (3 °C/min) Ar/H₂ (95vol %/5vol %) for 1 h to form Sb@NC.

Synthesis of Sb₂Se₃@NC: Sb@NC and selenium powder mass ratio 1:2 mixed evenly, then annealed at 500 °C (5 °C/min) in N₂ atmosphere for 2 h to form Sb₂Se₃@NC.

Synthesis of Sb₂Se₃@NC/MXene: MAX(Ti₃AlC₂) freestanding thin films were purchased from Nanjing/Jiangsu XFNANO Materials Tech Co., Ltd. (No. XFK07). 100 mg Sb₂Se₃@NC was ultrasonic dispersed in 100 ml deionized water for

30 min to form a uniform solution, then 10 mg MXene was added to the above solution and stirred for 24 h. Finally, solution was freeze-dried at $-55\text{ }^{\circ}\text{C}$ for 48 h until all the water sublimated, and the remaining powder was $\text{Sb}_2\text{Se}_3@\text{NC}/\text{MXene}$.

Material Characterization: The as-prepared samples are in rounded analysis by a large number of characterization equipments, such as conventional scanning electron microscopy (SEM, Hitachi, SU-9010), X-ray diffraction (XRD, D/max-2000, Cu $K\alpha$). Also transmission electron microscopy (TEM, Tecani G-20) with high resolution-TEM (HREM), XPS (ESCALAB, 250Xi) and thermogravimetric analysis (TGA; STA 449 F3) are extensively used. A Micromeritics ASAP 2020b system was used to quantify the Brunauer–Emmett–Teller (BET) surface area at 77 K.

Electrochemical Measurements: The electrode is prepared by $\text{Sb}_2\text{Se}_3@\text{NC}/\text{MXene}$, Super P, polyvinylidene fluoride and N-methylpyrrolidone. Their weight ratios are 70%, 20% and 10%, respectively. Then the slurry is evenly applied to the copper sheets and dried for 12 h at $100\text{ }^{\circ}\text{C}$ in vacuum oven. The electrode sheets are then pressed under a force of about 10 MPa and cut it into circular sheets. Afterwards, it is assembled into coin-type cells in glove box. Sodium sheet is the counter electrode. The 1.0 M NaPF_6 in 1,2-dimethoxyethane is the electrolyte. Cycle performance and rate performance are tested on Neware battery test system (0.01–3.0 V). CV is performed out on the electrochemical workstation. For full cells preparation, the $\text{Sb}_2\text{Se}_3@\text{NC}/\text{MXene}$ anode is presodiated in a half-cell for 3 cycles at 0.1 A g^{-1} in voltage range of 0.01–3.0 V to ensure the formation of a stable solid electrolyte interphase film.

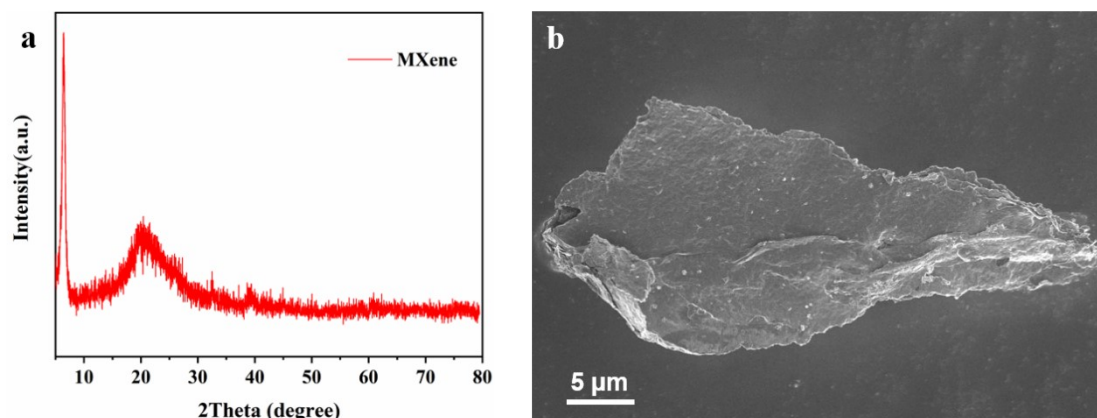


Figure S1. (a) XRD pattern. (b) SEM image of MXene.

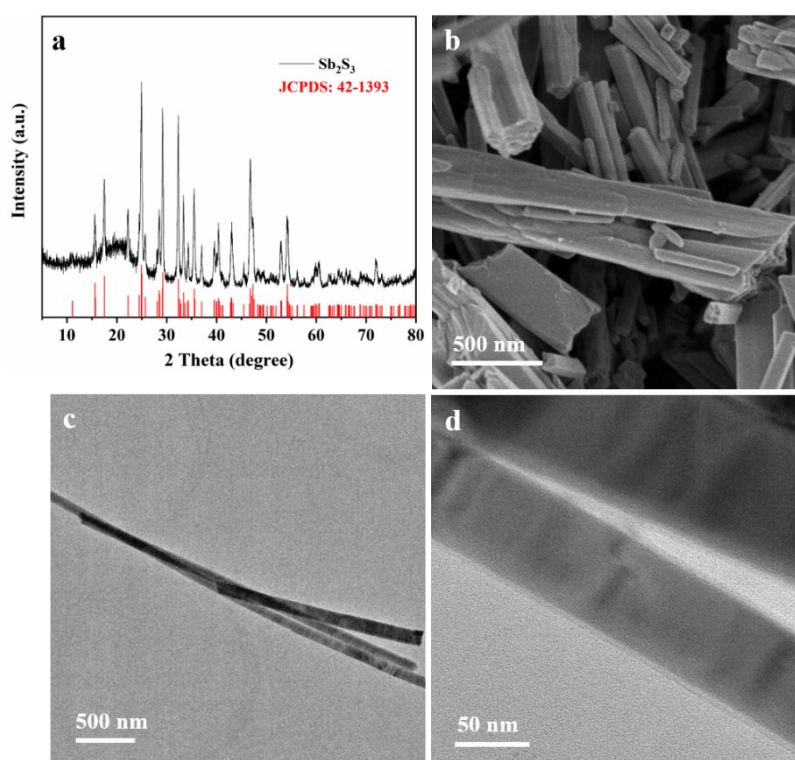


Figure S2. (a) XRD pattern. (b) SEM image. (c, d) TEM images of Sb_2S_3 .

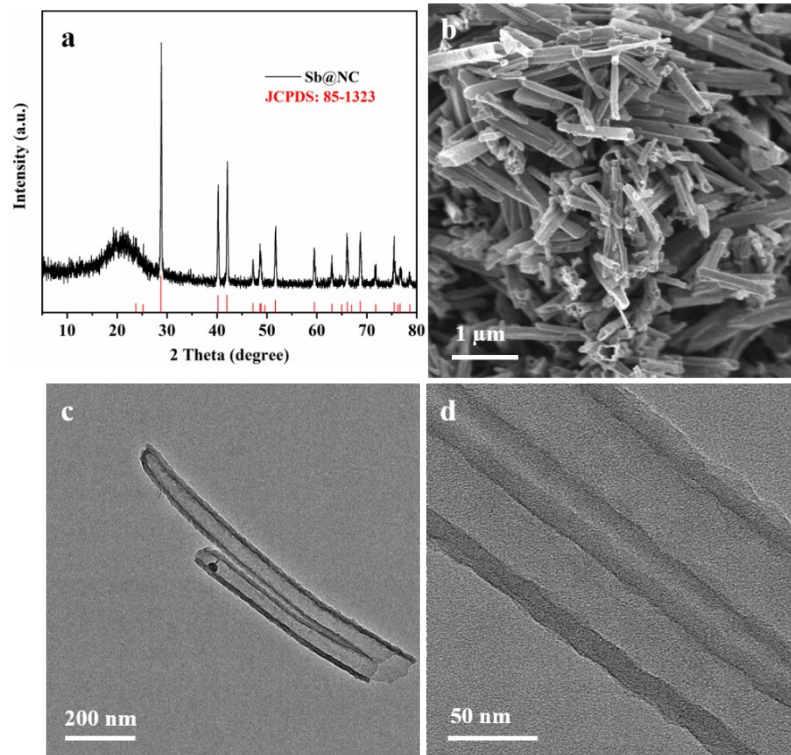


Figure S3. (a) XRD pattern. (b) SEM image. (c, d) TEM images of Sb@NC.

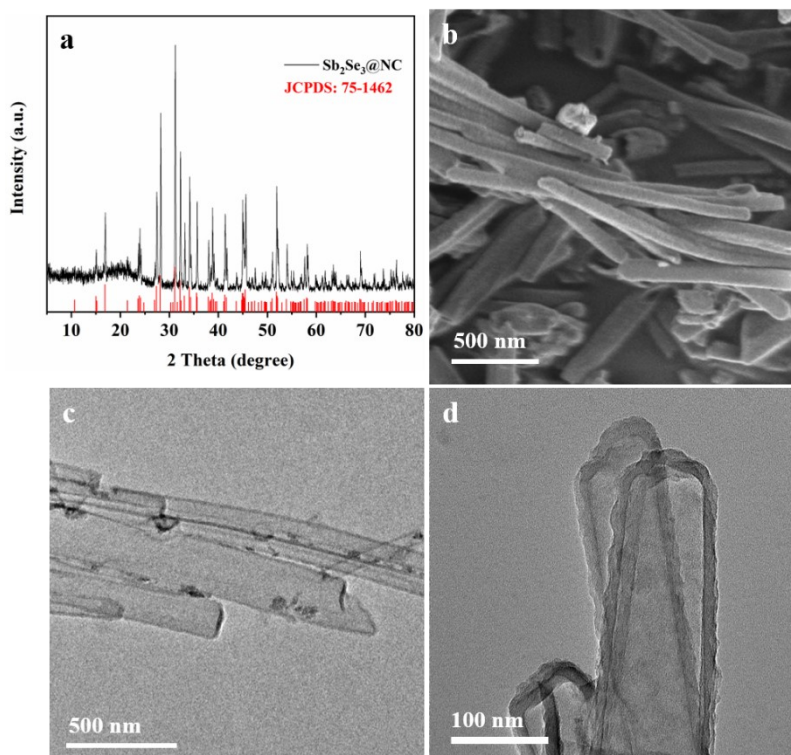


Figure S4. (a) XRD pattern. (b) SEM image. (c, d) TEM images of Sb₂Se₃@NC.

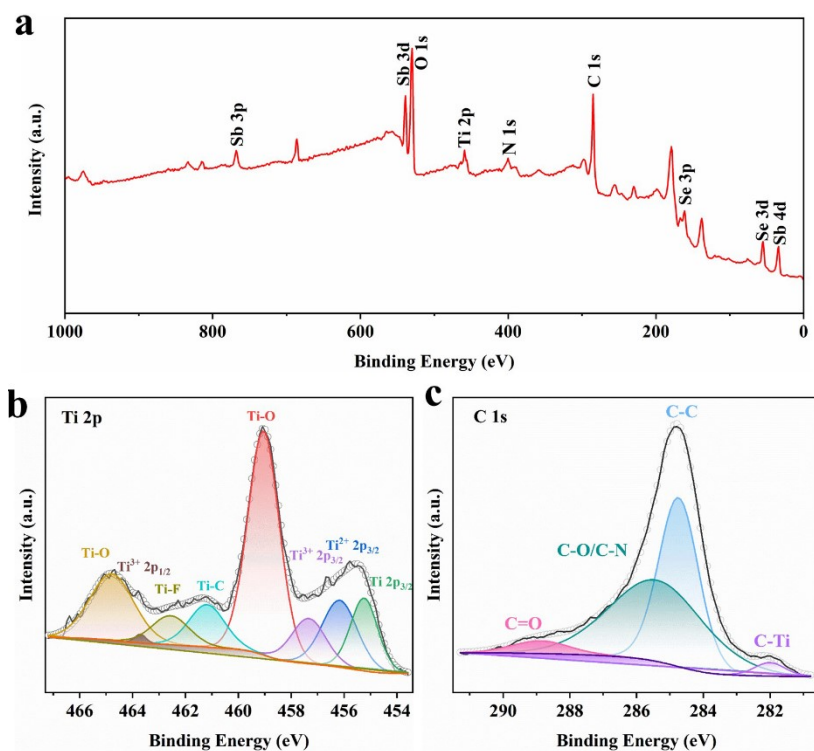


Figure S5. (a) XPS survey spectrum of $\text{Sb}_2\text{Se}_3@\text{NC}/\text{MXene}$. High-resolutions XPS spectra of (b) Ti 2p and (c) C 1s.

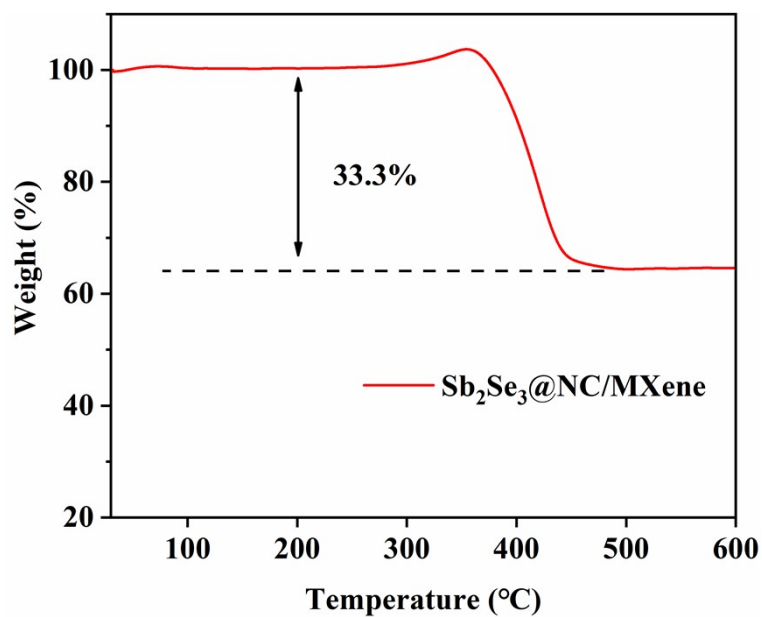


Figure S6. TGA curves of $\text{Sb}_2\text{Se}_3@\text{NC}/\text{MXene}$.

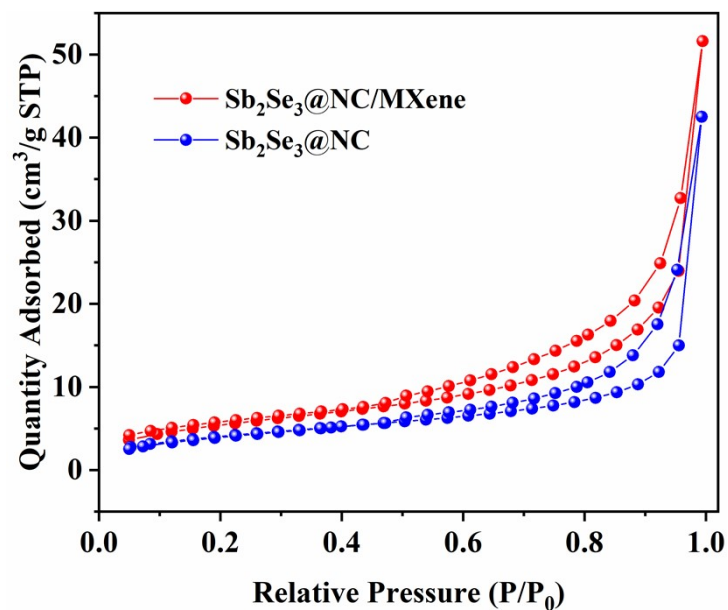


Figure S7. N_2 adsorption–desorption isotherms of $Sb_2Se_3@NC/MXene$ and $Sb_2Se_3@NC$.

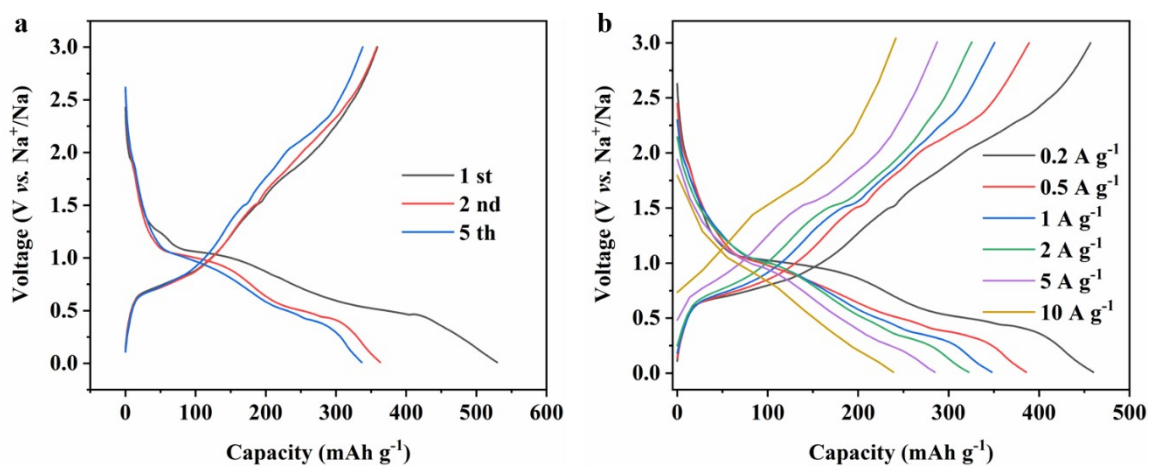


Figure S8. (a) Charge/discharge curves of $Sb_2Se_3@NC$ profiles at 0.2 A g^{-1} . (b) Charge/discharge curves from 0.2 to 10 A g^{-1} of $Sb_2Se_3@NC/MXene$.

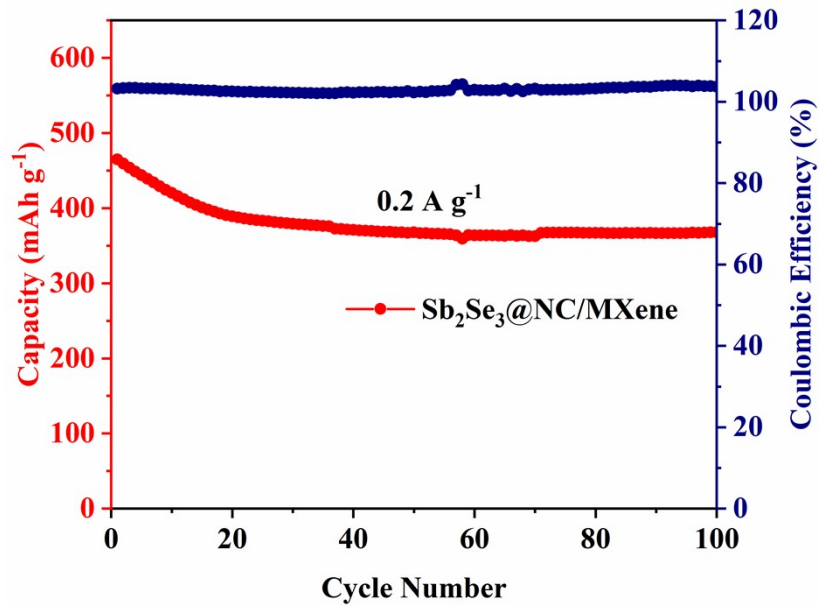


Figure S9. Cycle performance of $\text{Sb}_2\text{Se}_3@\text{NC}/\text{MXene}$ at 0.2 A g^{-1} .

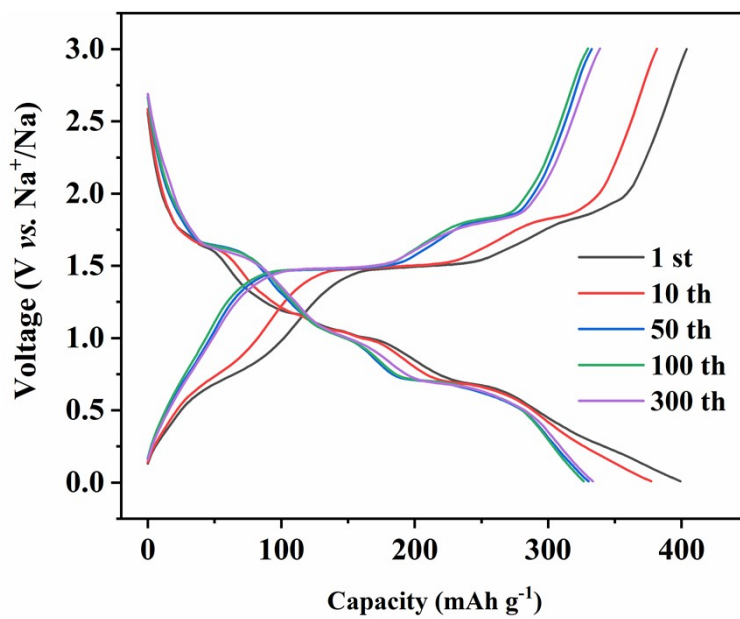


Figure S10. Charge/discharge curves at 1 A g^{-1} of $\text{Sb}_2\text{Se}_3@\text{NC}/\text{MXene}$ for different cycles.

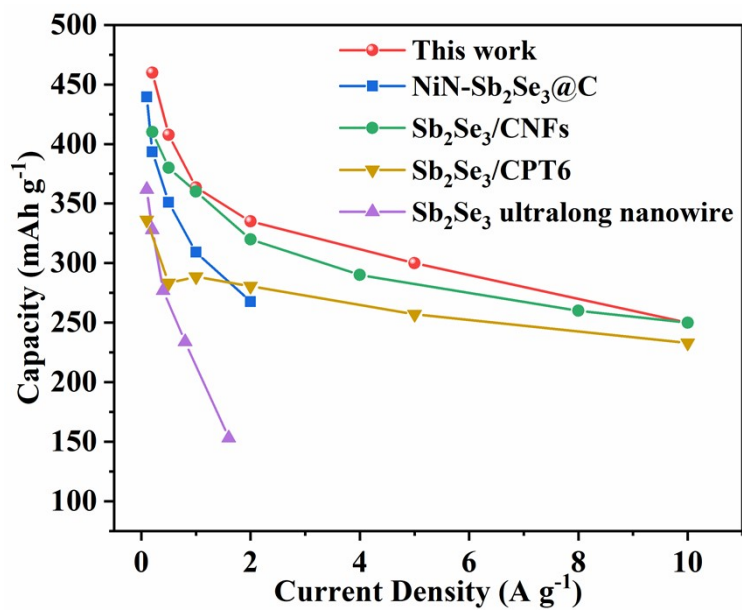


Figure S11. Rate capability comparisons of $\text{Sb}_2\text{Se}_3@\text{NC}/\text{MXene}$ electrode and other Sb_2Se_3 based electrodes as previously reported.¹⁻⁴

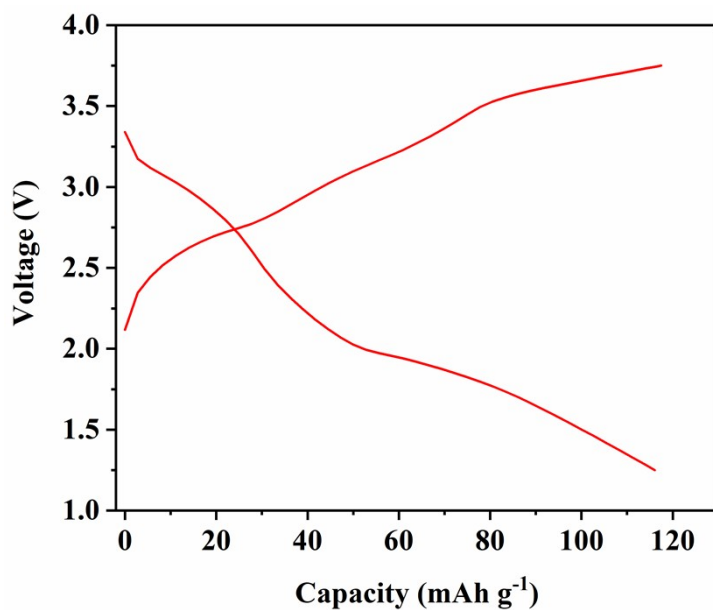


Figure S12. Charge/discharge curves at 1 A g^{-1} of the $\text{Sb}_2\text{Se}_3@\text{NC}/\text{MXene} // \text{NVPOF}$ full cell.

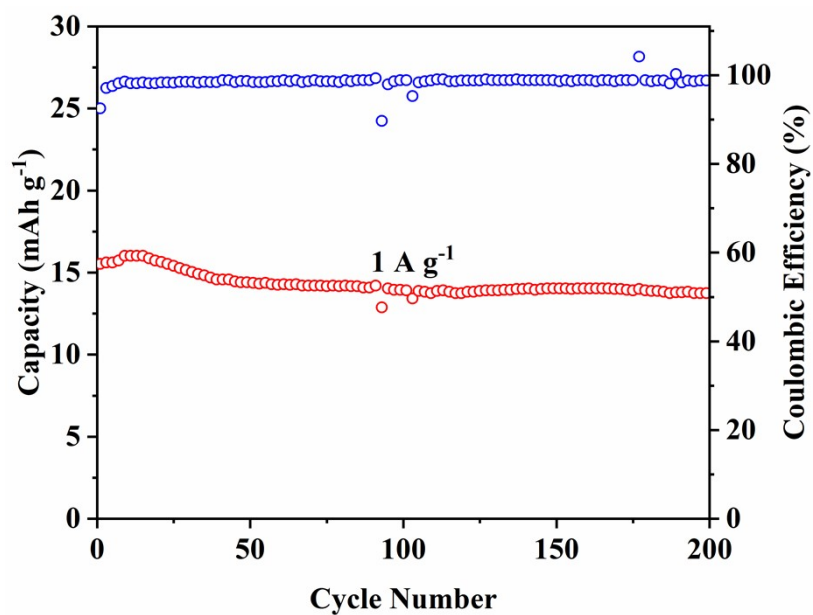


Figure S13. Cycling performance at 1 A g^{-1} of the $\text{Sb}_2\text{Se}_3@\text{NC}/\text{MXene} // \text{NVPOF}$ full cell based on the mass loading of NVPOF.

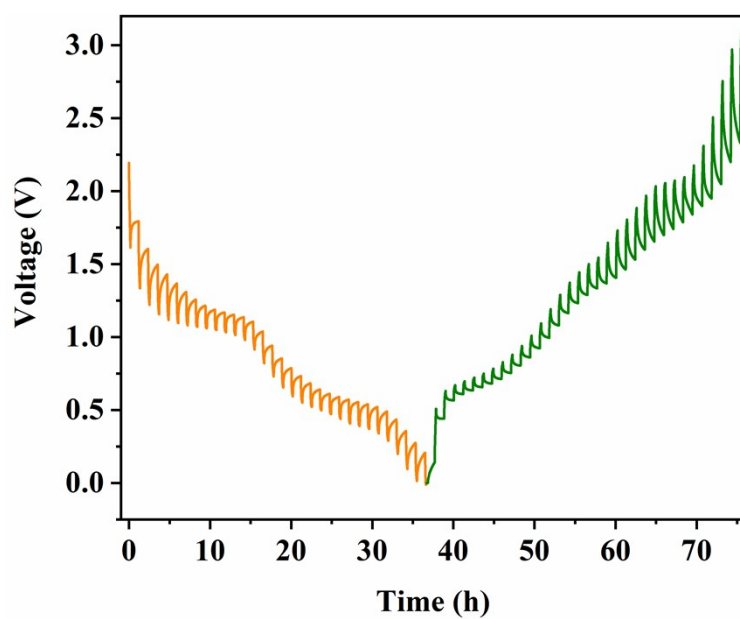


Figure S14. GITT curves of $\text{Sb}_2\text{Se}_3@\text{NC}$.

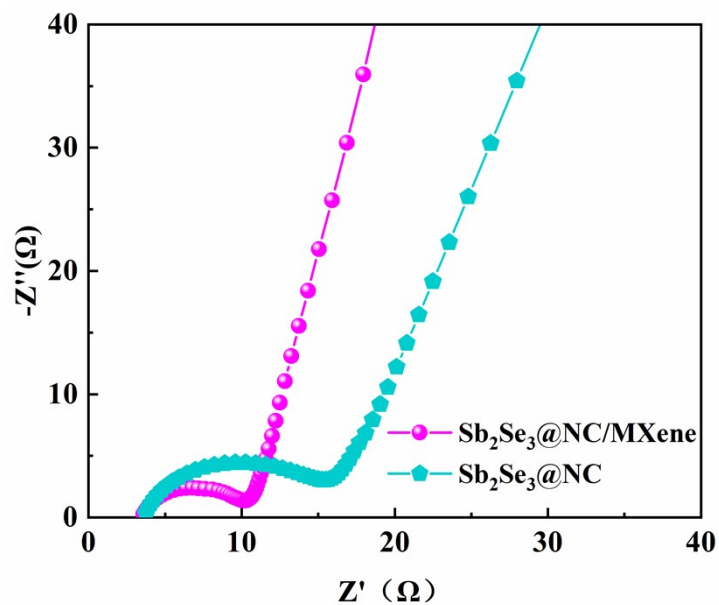


Figure S15. Nyquist plots of the $\text{Sb}_2\text{Se}_3@\text{NC}/\text{MXene}$ and $\text{Sb}_2\text{Se}_3@\text{NC}$

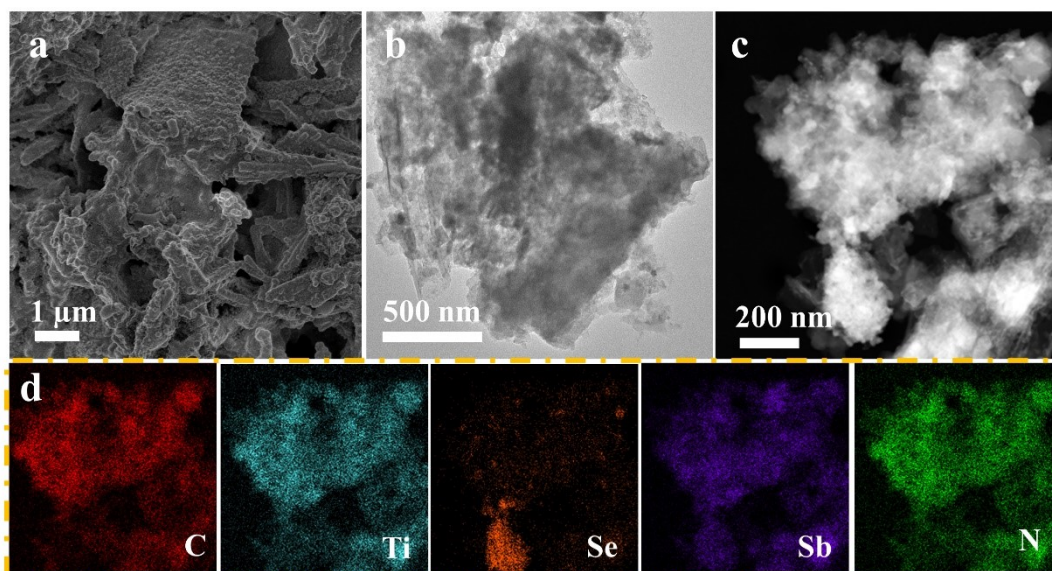


Figure S16. SEM, TEM and EDX mapping images of $\text{Sb}_2\text{Se}_3@\text{NC}/\text{MXene}$ after 20th cycle at 0.5 A g^{-1} .

References

1. X.-Z. Li, S.-H. Tian, Z.-C. Lv, P.-F. Wang and T.-F. Yi, *Ceram. Int.*, 2023, **49**, 7414-7423.
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