Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

3R-NbS_2 as a Highly Stable Anode Material for Sodium-ion Batteries

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Experimental section

Synthesis of Nb-HDA complex

A previously reported procedure was followed to synthesize the Nb–HDA complex.[14] The Nb–HDA complex was prepared using niobium(V) ethoxide ($Nb(OC_2H_5)_5$. 99.95%, Sigma Aldrich), hydrogen peroxide (30%, H_2O_2), and 1-hexadecylamine ($C_{16}H_{33}NH_2$, HDA) in a glass beaker. 25 mL of H_2O_2 solution was added to 1 mL of $Nb(OC_2H_5)_5$ (4×10^{-3} mol) yielding niobium(V) ethoxide dispersion. 3.76 g of HDA (15×10^{-3} mol) was dissolved in 5 mL of ethanol at 70 ^{0}C and poured into the niobium(V) ethoxide dispersion. Nb–HDA complex was formed spontaneously in the form of a yellow foam along with the generation of heat. The Nb–HDA complex was purified and dried at room temperature and was then employed for the preparation of 3R-NbS_2 nanosheets.

Synthesis of 3R-NbS_2 nanosheets

The as-prepared Nb–HDA complex was taken in a quartz boat and placed in a quartz tube far from the heating zone of the furnace. Maintaining a continuous flow of N_2 gas, the temperature was increased to 950 ^{0}C. The quartz boat was then pushed into the hot zone of the furnace using a magnet and the flow rates of H_2S and N_2 gases were programmed to 35 and 65 cm^3 min^{-1}, respectively. The boat remained in the hot zone for 30 min. Afterwards, the boat was pulled away from the hot zone while the flow rates of N_2 and H_2S gases were changed to 85 and 10 cm^3 min^{-1}, respectively, for 30 min.

Characterization

The X-ray diffraction pattern (XRD) was acquired using a Cu Kα source (λ = 0.154178 nm). The Raman spectrum was obtained using a Jobin Yvon LabRam HR spectrometer with a 514 nm Ar laser. X-ray photoelectron spectra (XPS) measurements were carried out using an Omicron nanotechnology spectrometer with Mg Kα as the X-ray source. Field emission scanning electron microscopy (FESEM) was carried out using Tescan and Bruker MIRA 3 and Nova Nano SEM 600. The transmission electron microscope (TEM) images were obtained using JEOL JEM 2100 Plus and TALOS F200S G2. Dynamic light scattering (DLS) measurements were carried out using Malvern Nano ZS.

Electrochemical measurements

2032-type coin cells were fabricated in an Ar-filled glovebox with 3R-NbS_2 as working electrode, Na metal as counter electrode using glass fibre as separator. A slurry was prepared by grinding 3R-NbS_2, conducting carbon and sodium polyacrylate (PAA) binder in 70:15:15 ratio in deionized (DI) water for 30 min. Blade-coating technique is employed to fabricate thin film of 30 μm thickness on a carbon coated Al (Al-C) foil, followed by drying in a vacuum oven at 80 ^{0}C for 24 h. 1 M $NaClO_4$ in ethylene carbonate (EC) and propylene carbonate (PC) mixed solvent in the ratio (1:1) was used as the electrolyte. Neware BTS-4000 battery tester was employed to perform the GCD experiments at different current densities in the voltage range 0.01-2.5 V (vs. Na/Na^+). Metrohm Autolab PGSTAT302N electrochemical workstation was used to carry out electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) measurements. The values of specific capacities and current densities have been determined considering the mass of the active material.

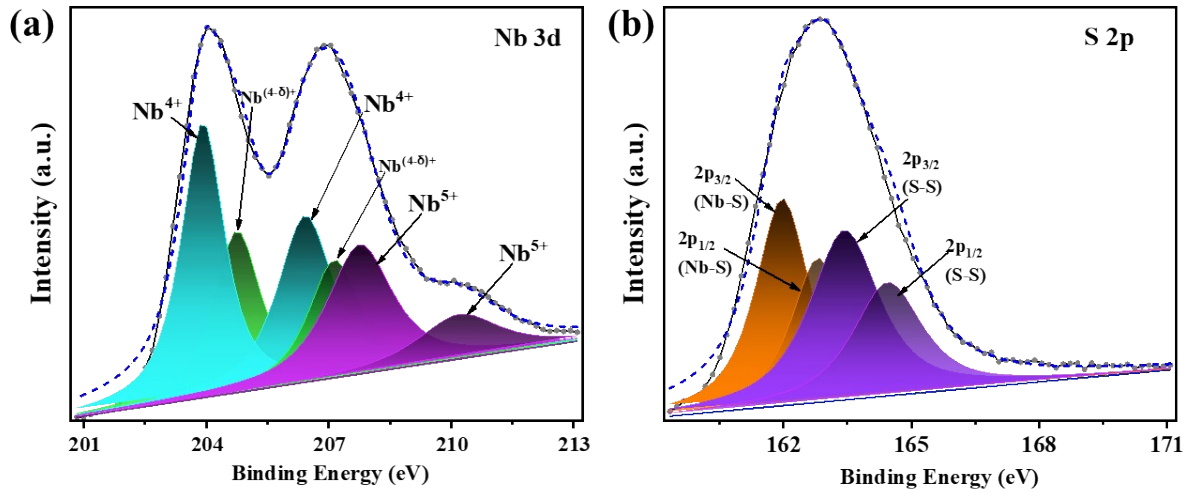

Figure S1 (a) Nb 3d and (b) S 2p XPS spectra of 3R-NbS_2.

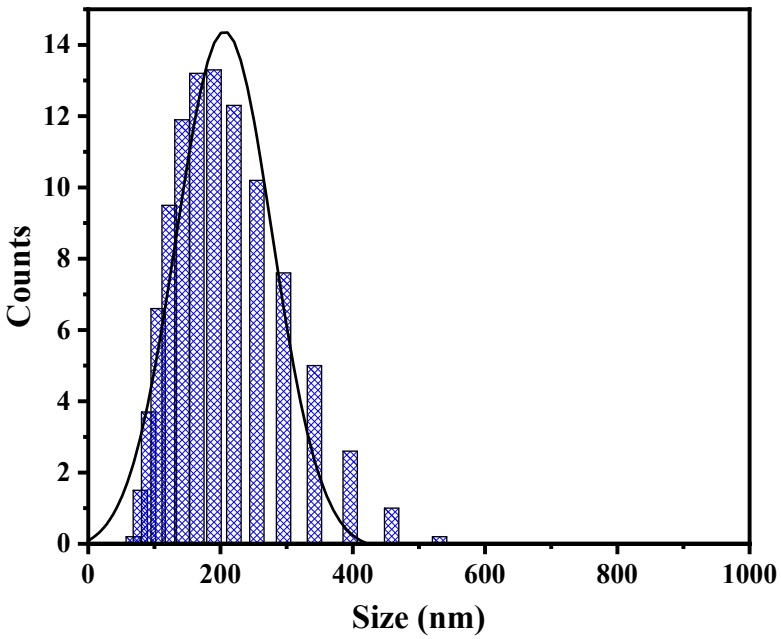

Figure S2. Particle size measurement of 3R-NbS_2 nanosheets by DLS.

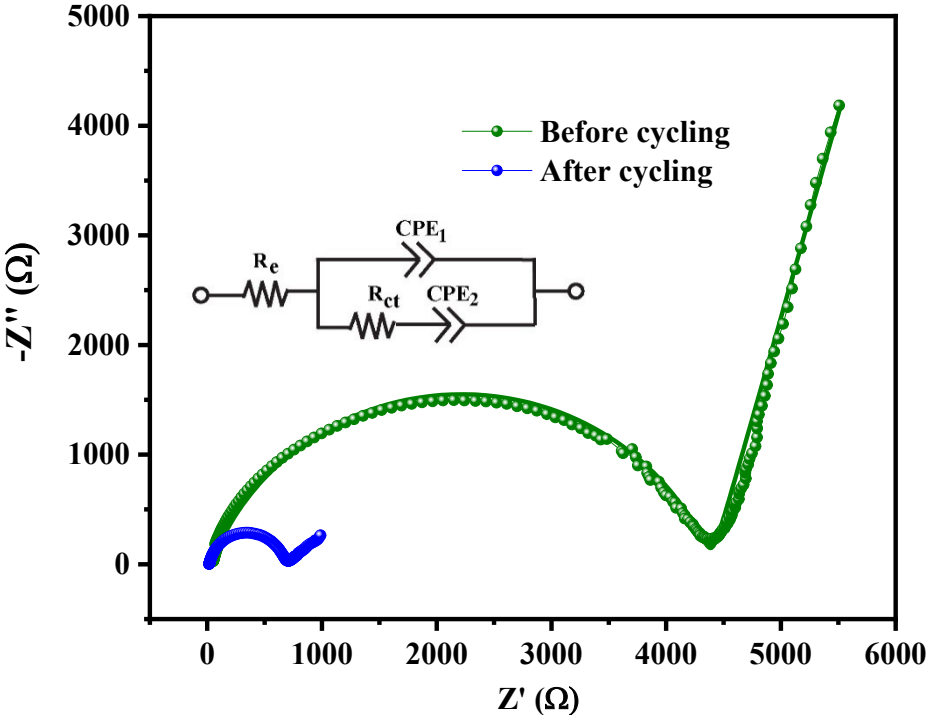

Figure S3. EIS spectra before and after CV cycles at 0.1 mV s^{-1}.

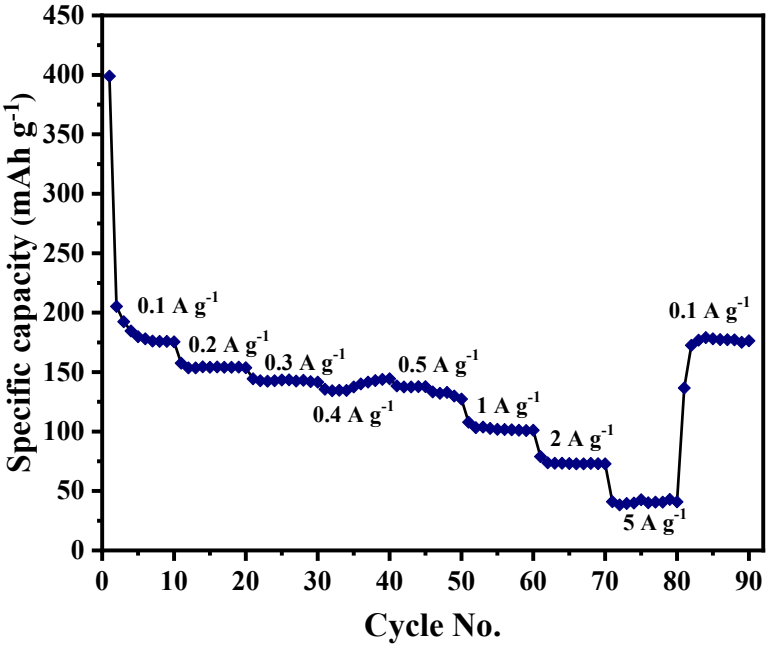

Figure S4. High rate performance of NbS_2 SIB anode.

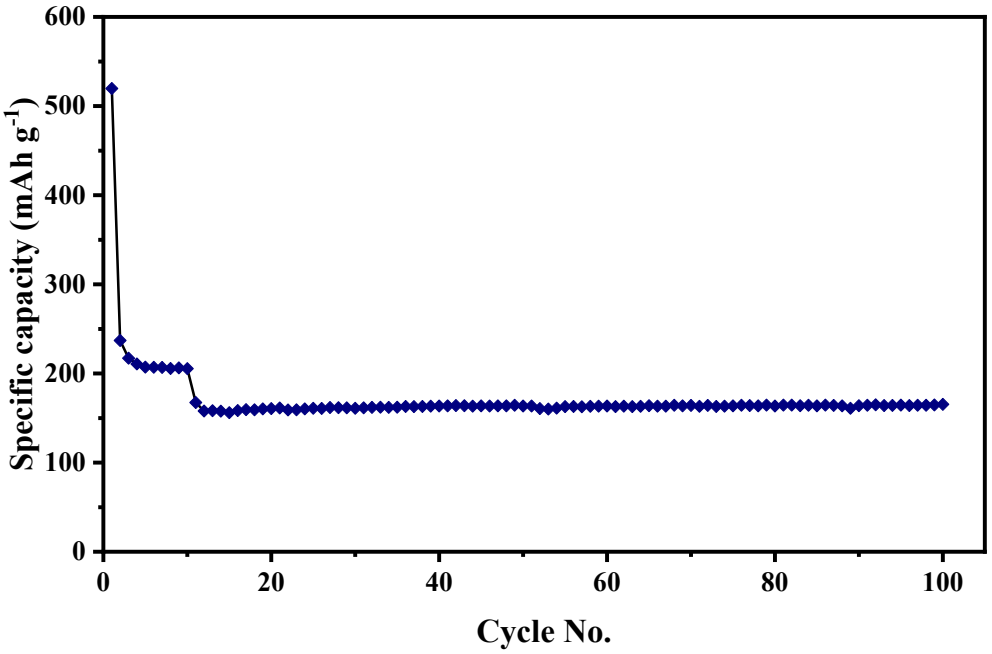

Figure S5. Pre-cycling step at 0.1 A g^{-1} (10 cycles) before cycling at 0.5 A g^{-1}

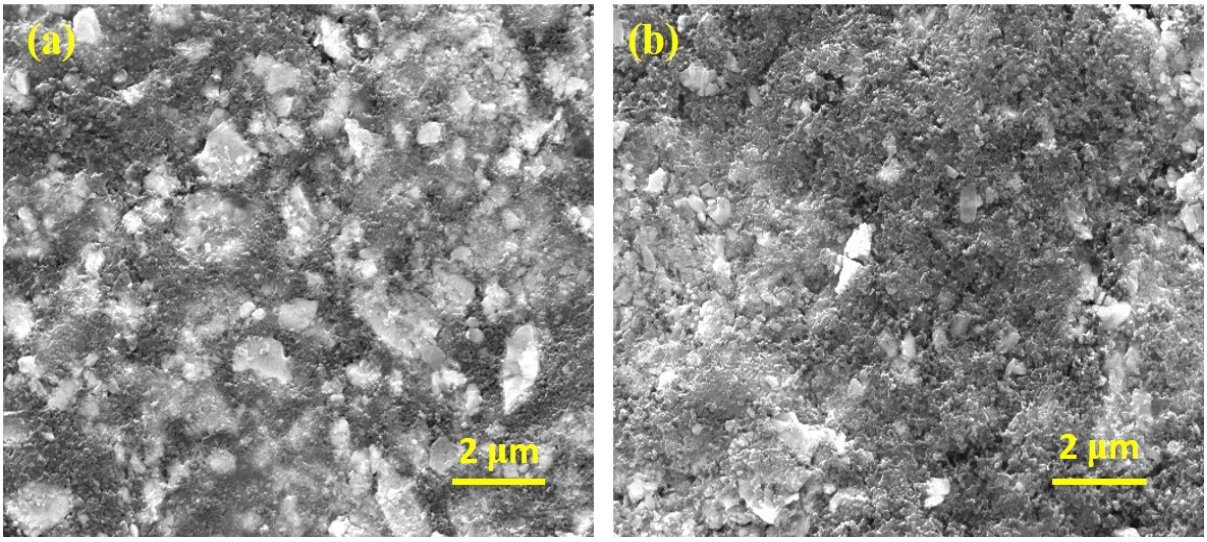

Figure S6. SEM of NbS_2 electrode (a) before cycling and (b) after 2500 cycles.

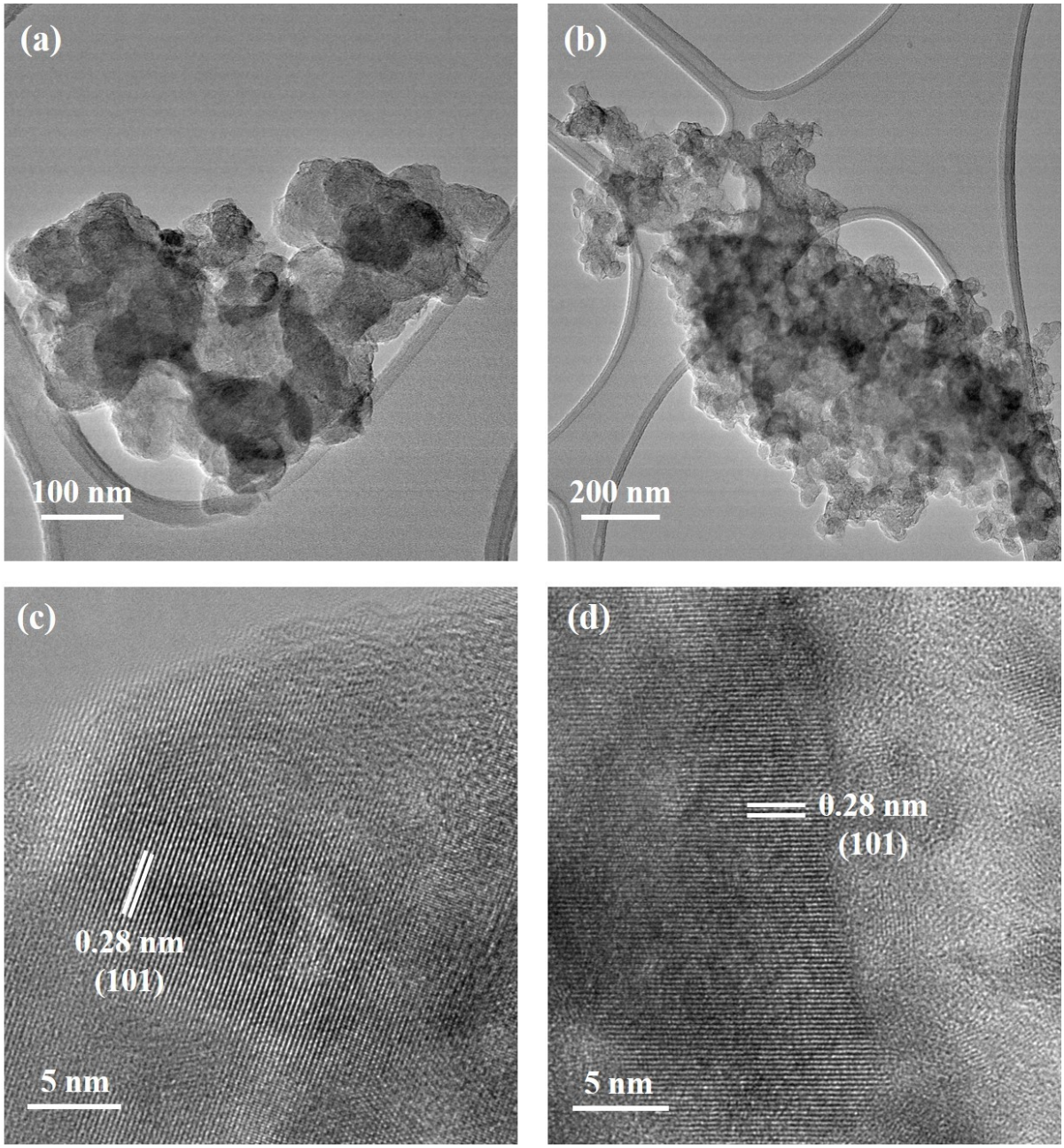

Figure S7. (a,b) TEM images of NbS_2 electrodes before and after 2500 cycles respectively. (c,d) HRTEM images of NbS_2 electrodes before and after 2500 cycles respectively

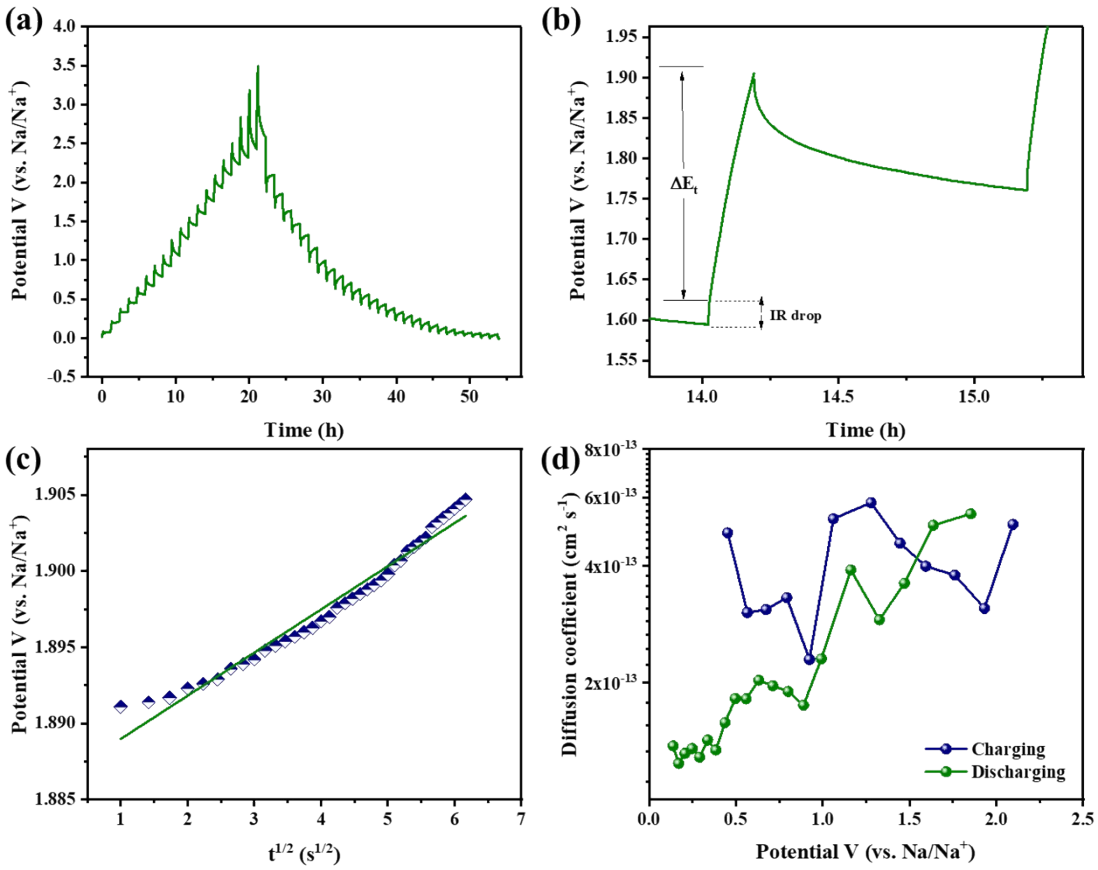

Figure S8. (a) GITT plot of NbS_2 SIB anode, (b) Magnified view of charging step during GITT, (c) ΔE_t-vs-$t^{1/2}$ dependence, (d) diffusion coefficient of anode during charging and discharging.