Electronic Supplementary Information for

Oxygen vacancies boosted the electrochemical kinetics of Nb_2O_{5-x} for superior lithium storage

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1. Experimental section

1.1. Preparation of Nb_2O_{5-x} microflowers

All chemicals were analytical reagents and can be used directly without purification. Firstly, 7.5 mmol of niobium oxalate ($C_{10}H_5NbO_{20}$) and 1.5 mmol of ammonium carbonate ($(NH_4)_2{\cdot}CO_3$) were added to 30 mL of deionized water under sonicating for 30 minutes. Then, the mixed suspension was transferred to a 50 mL of Teflon-lined stainless steel autoclave and kept at 200 °C for 12 hours. After the reaction, the product was collected, washed with deionized water and ethanol, and dried in an oven at 60 °C for 12 hours. Finally, the product was calcined at 450 °C for 30 minutes with a heating rate of 2 °C min^{-1} in Ar/H_2 (95%/5%) to acquire the Nb_2O_{5-x} microflowers.

1.2. Characterization

The composition and phase information of the samples were measured by a powder X-ray diffractometer (XRD, German Bruker D8 diffractometer with Cu radiation). The morphology and microstructure of the samples were tested by the scanning electron microscope (FE-SEM, JEOL, JSM-7800F) and the high-resolution transmission electron microscope (HRTEM, JEOL, JEM-2100F). The surface elemental composition and chemical states of the samples were performed by X-ray photoelectron spectroscopy (XPS, Thermo Scientific ESCALAB 250Xi system with a monochromated Al Kα X-ray source 1486.6 eV). The Raman spectra were obtained on a DXR Raman spectrometer (Thermo Scientific) with a 532 nm laser for illumination. The electron spin resonance (EPR) was performed by using a BRUKER A300-10/12 instrument. The

thermogravimetric analysis (TGA) was characterized on a (Netzsch STA 449 F3, USA) instrument. Nitrogen adsorption–desorption isotherms were measured on a Beishide 3H-2000PS2 type specific surface area and pore size analyzer at 77 K. The Brunauer–Emmett–Teller (BET) specific surface areas (S_{BET}) were calculated using the BET equation, and the pore size distributions were determined using the Barret–Joyner–Halender (BJH) method.

1.3. Electrochemical measurements

The Nb_2O_{5-x} microflowers (80 wt.%), super conductive carbon black (SCCB, Ketjenblack EC-600JD, Lion Corporation) (10 wt.%), and polyvinylidene fluoride (PVDF) (10 wt.%) were mixed in N-methyl-2-pyrrolidone (NMP) and then coated the slurry onto the copper foil. The loading mass was in the range of 0.9 to 1.2 mg cm^{-2}. Using CR2032-type coin cells assembled in an Ar-filled glove box (oxygen/moisture concentrations < 0.01 ppm) carried out the electrochemical test. The celgard 2400 porous polypropylene membrane was used as the separator. The lithium foil was used as the counter electrode. The lithium-ion electrolyte was 1 M $LiPF_6$ EC/DMC/DC solution. Cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) measurements were performed on a Gamry (Interface 1000E Potentiostat) electrochemical workstation. The galvanostatic discharge and charge and galvanostatic intermittent titration technique (GITT) tests were tested with the NEWARE-CT-4008 battery tester between 0.01 V and 3.0 V.

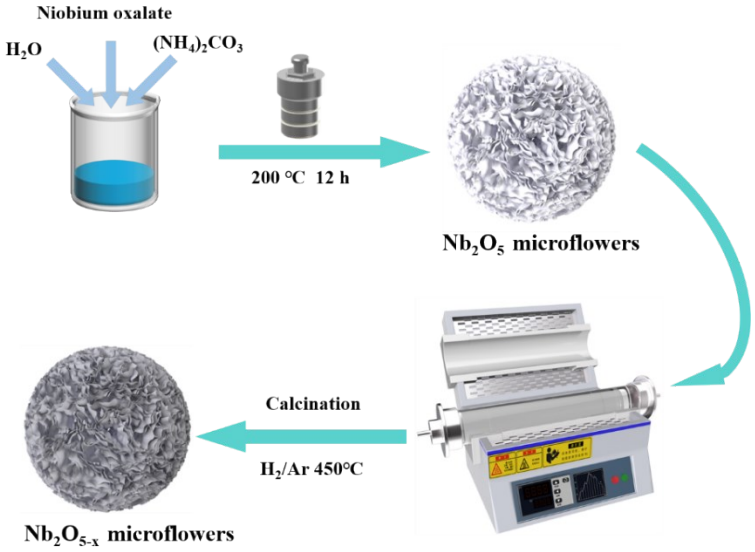

Fig. S1 Illustration of the preparation process of the Nb_2O_{5-x} microflowers.

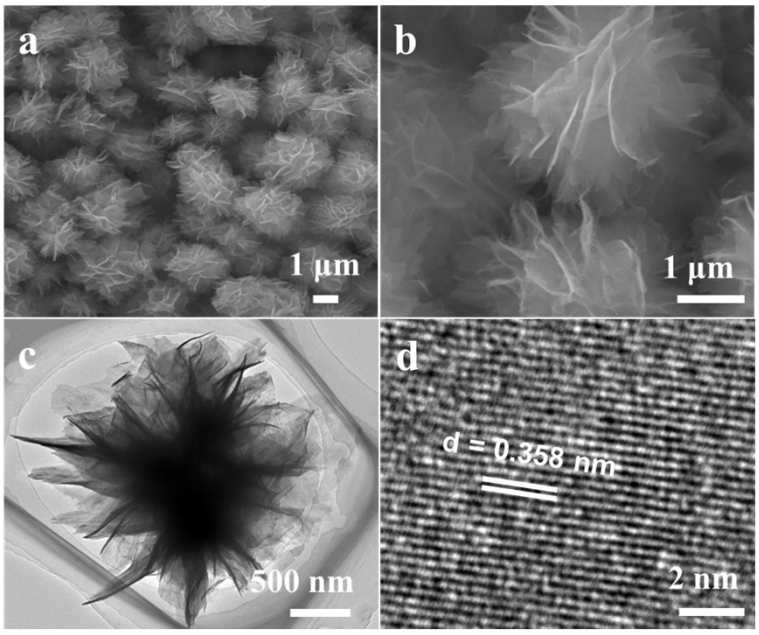

Fig. S2 (a, b) SEM, (c) TEM, and (d) HRTEM images of the Nb_2O_5 microflowers.

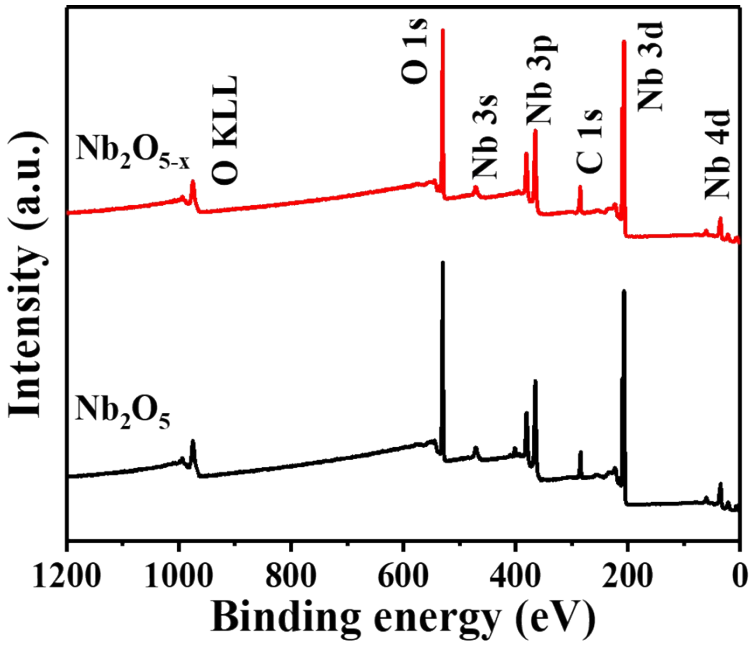

Fig. S3 XPS survey spectra of the Nb_2O_5 and Nb_2O_{5-x}.

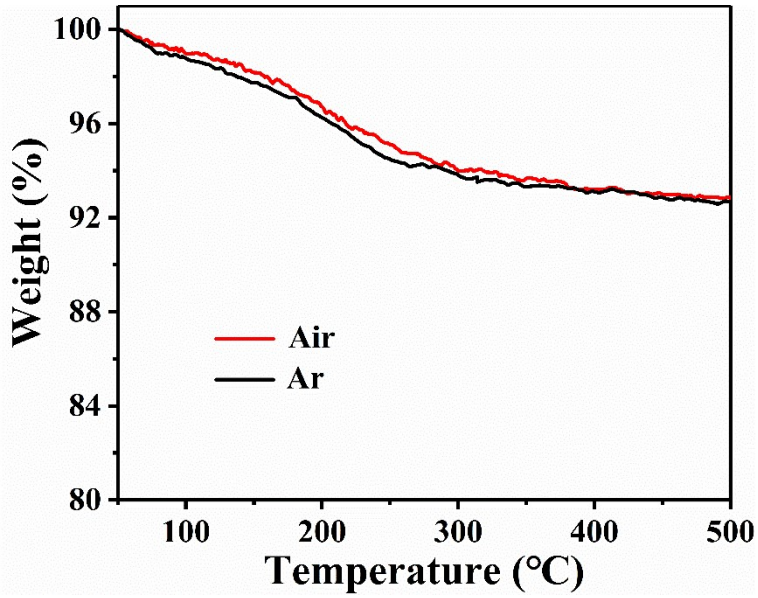

Fig. S4 TGA curves of the Nb_2O_5 sample tested in air and argon, respectively.

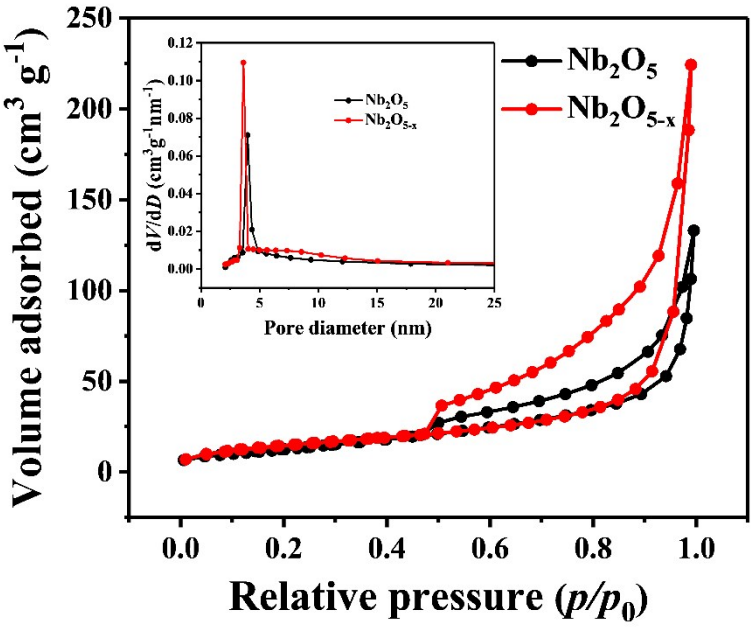

Fig. S5 N_2 adsorption-desorption isotherms and pore size distribution plots (inset) of the Nb_2O_5 and Nb_2O_{5-x} samples.

Table S1 BET specific surface area, BJH pore size, and pore volume of the Nb_2O_5 and Nb_2O_{5-x}.

Samples	BET surface area (m^2 g^{-1})	BJH pore size (nm)	Pore volume (cm^3 g^{-1})
Nb_2O_5	48.61	3.81	0.23
Nb_2O_{5-x}	**53.74**	**3.62**	**0.35**

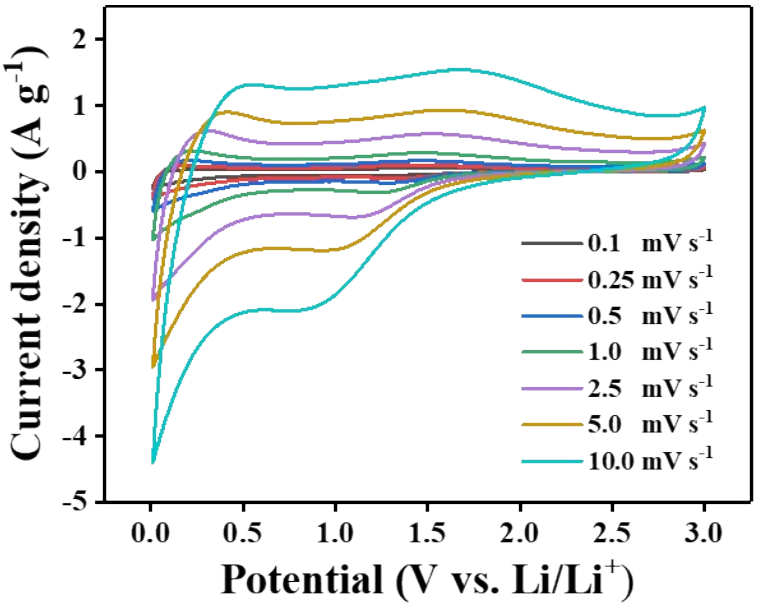

Fig. S6 CV curves of the Nb_2O_5 electrode at different scan rates.

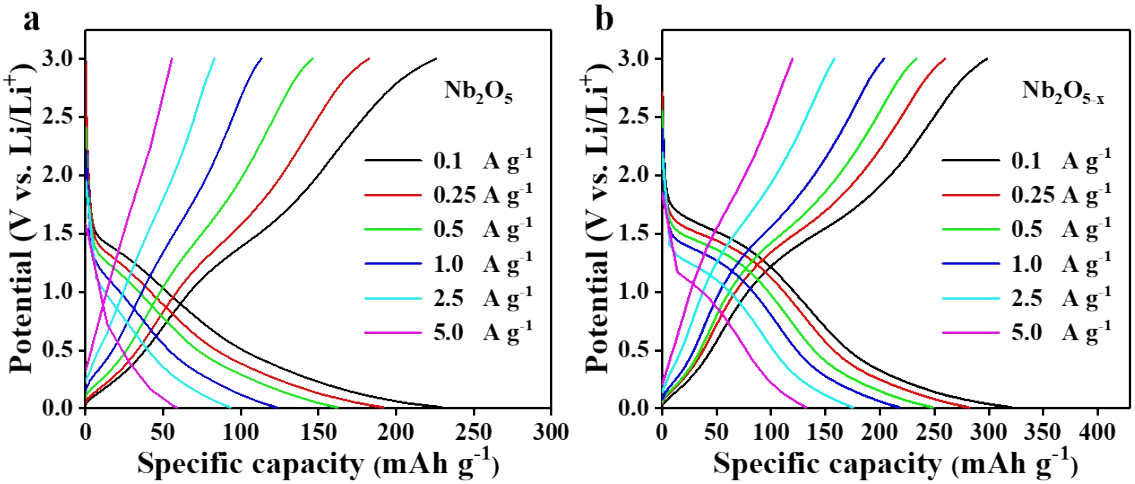

Fig. S7 GCD curves of the (a) pristine Nb_2O_5 and (b) Nb_2O_{5-x} at various current densities, respectively.

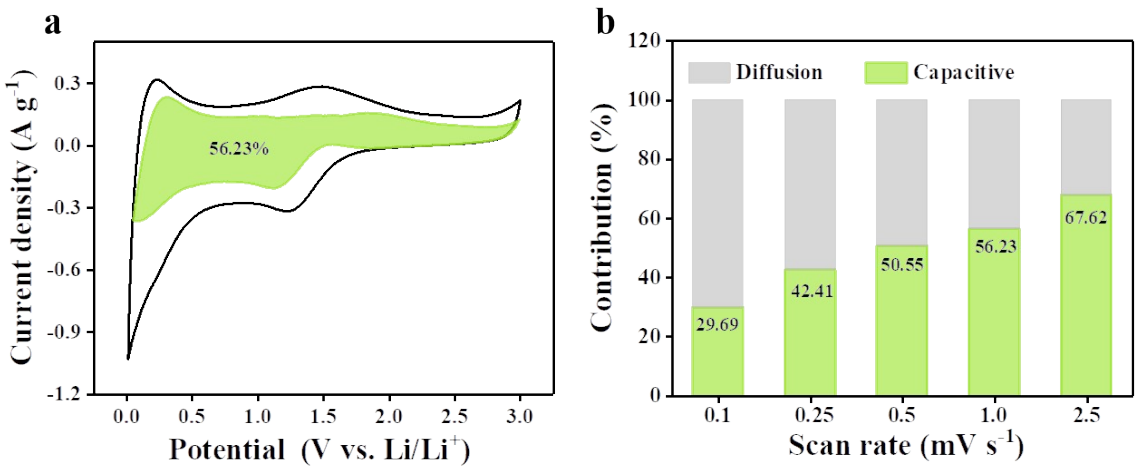

Fig. S8 (a) CV response of the Nb_2O_5 electrode with separation of the capacitive (green region) and diffusion current (blank region) at 1.0 mV s^{-1}. (b) Capacitive contribution ratio of the Nb_2O_5 electrode.

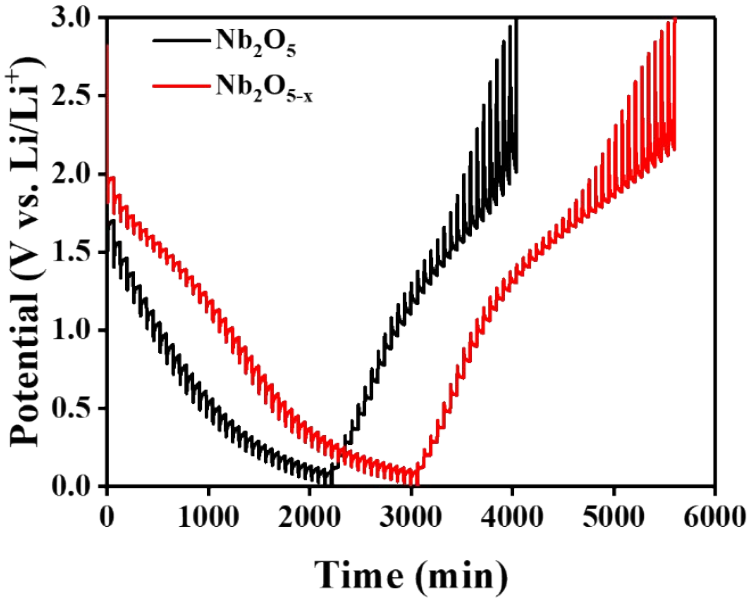

Fig. S9 GITT curves of the Nb_2O_5 and Nb_2O_{5-x}.