Supplementary Material

Amorphous Niobium pentoxide with carbon composite as structurally stable high performance anode material for sodium-ion batteries

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Experimental Methods

1.Synthesis of α-Nb_2O_5@C and α-Nb_2O_5

We prepared a solution by homogeneously blending 10 mL of ethanol (anhydrous) with 30 mL of isopropanol, then 130 mg of niobium pentachloride was added. The resulting mixture was stirred on a magnetic blender for 20 minutes and then subjected to ultrasonication for 20 minutes, after which 2 mL of glycerol was incrementally introduced. The mixture is then sonicated for 5 minutes and stirred for 20 minutes. Following preparation, the mixture was poured into a 100-mL Teflon-coated stainless-steel pressure reactor, and heated to 190°C in an oven and maintained at this temperature for 12 hours. After cooling down, the precipitate was centrifuged using deionized water and anhydrous ethanol. Then it was put in a vacuum drying oven at 80°C for a period of 8 hours. After cooling down, the precipitate was heated in furnace to 450 ℃ at 2℃ min^{-1} for 6 h in argon atmosphere, and the resulting α-Nb_2O_5@C composite material was synthesized. Meanwhile, the α-Nb_2O_5 was produced by subjecting the white precipitate to the same calcination process, but in an air atmosphere.

2.Material characterization

The crystalline phase of α-Nb_2O_5@C and α-Nb_2O_5 were measured by XRD on a Bruker-D8. The morphology was measured by SEM using a Hitachi SU-8010. TEM were conducted on an HT7700 instrument. To investigate the valence states, XPS were provided by an equipment from Thermo Fisher Scientific, EDAX was measured on a FEI Titan Themis Cubed G2 300.

3.Electrochemical measurements

We fabricated the materials into pole pieces and assembled 2032 button cells to test their electrochemical properties. The electrode formulation comprised 70% α-Nb_2O_5, 20% superfine conductive carbon, and 10% PVDF. Some NMP was added to this mixture, which was then thoroughly mixed on a magnetic stirrer for 12 hours to create a homogeneous slurry. Then we coated the slurry onto copper foil and put it in a vacuum at 80°C for 12 hours. Once cooled to ambient temperature, the coated foil was cut into 12 mm diameter circles, with an average mass loading of about 1.2 mg cm^{-2} per anode. Then in a confined, argon-filled glove box, we assembled 2032 button cells. We used sodium metal foils as the counter electrode and glass microfiber separators (Whatman GF/A). The electrolyte solution was formulated by incorporating 1.0 M $NaClO_4$ in EC/DEC (1:1) with 5%FEC. For electrochemical testing, charge and discharge processes were carried out at the voltage of 0.01 to 3 V, utilizing a Land battery testing system. Furthermore, EIS was measured on an AUTOLAB PGSTAT302N electrochemical workstation and CVs were measured on the same instrument over 0.01 to 3 V at scan rates ranging from 0.1 to 5 mV s^{-1}.

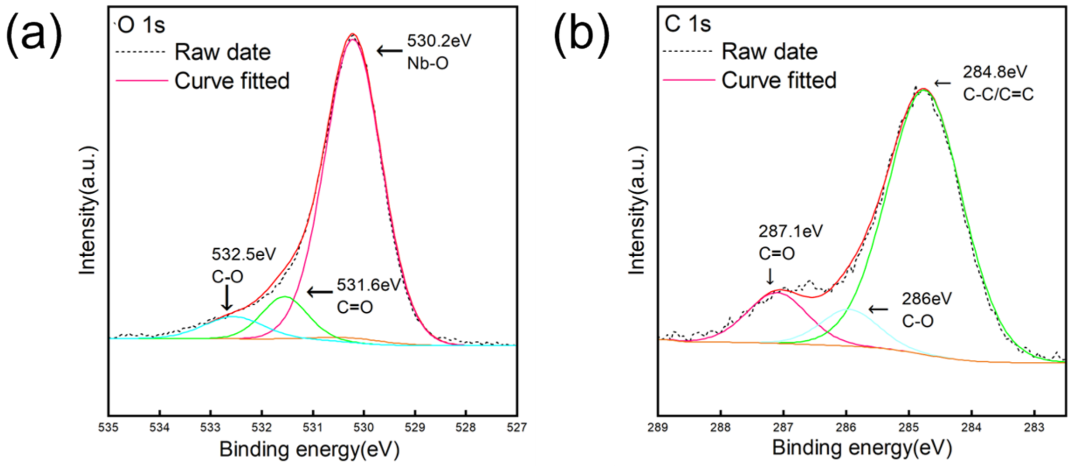

Fig. S1 XPS analysis of (a) O 1s spectra; and (b) C 1s spectra of the α-Nb_2O_5@C.

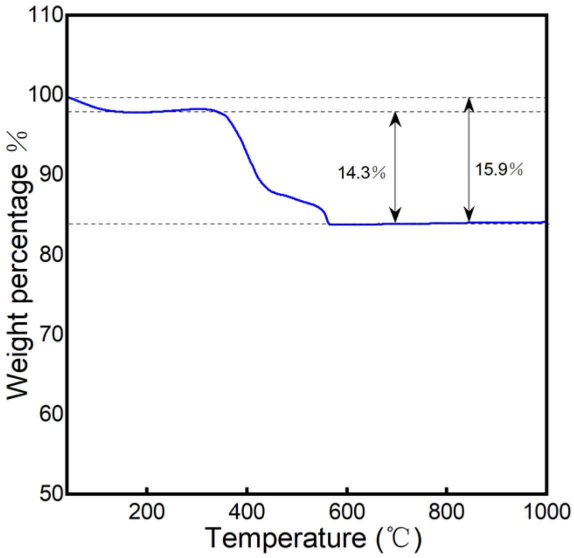

Fig. S2 TGA profile of α-Nb_2O_5@C. The initial weight loss at below 100°C is due to the loss of water in the sample.

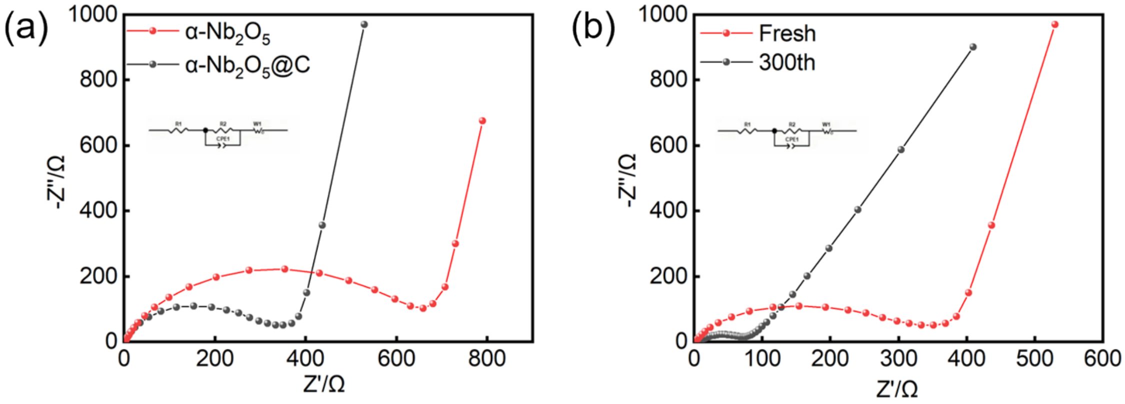

Fig. S3 (a) EIS results of α-Nb_2O_5 and α-Nb_2O_5@C in coin cell; (b) EIS of α-Nb_2O_5@C at fresh and after 300 cycles in coin cell.

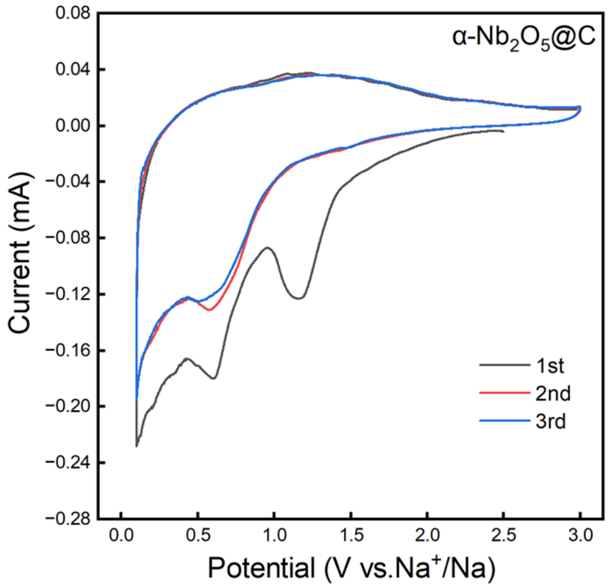

Fig. S4 CV curves of α-Nb_2O_5@C electrode in a 3-electrode cell for the first 3 cycles at a scanning rate of 0.1 mV s^{-1}.

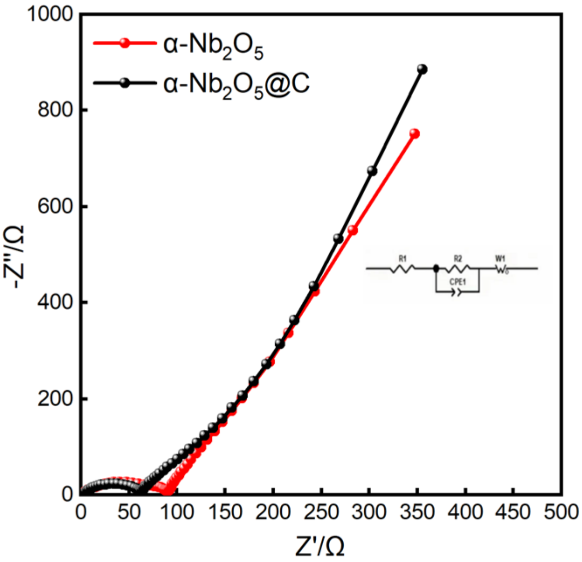

Fig. S5 EIS tests of α-Nb_2O_5 and α-Nb_2O_5@C in a 3-electrode cell.

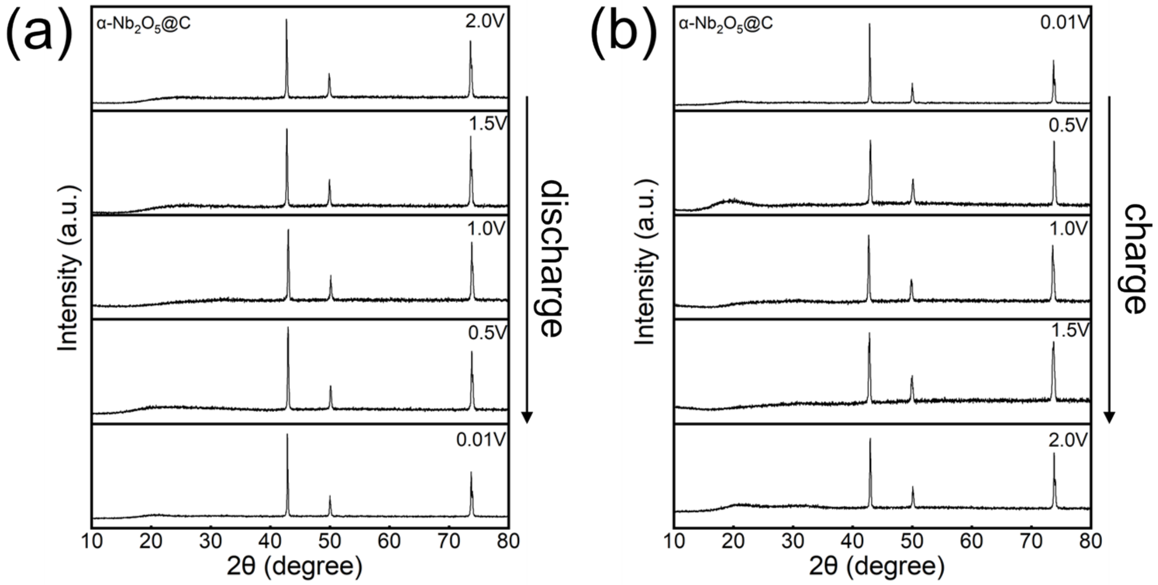

Fig. S6 XRD patterns of α-Nb_2O_5@C at different potentials.

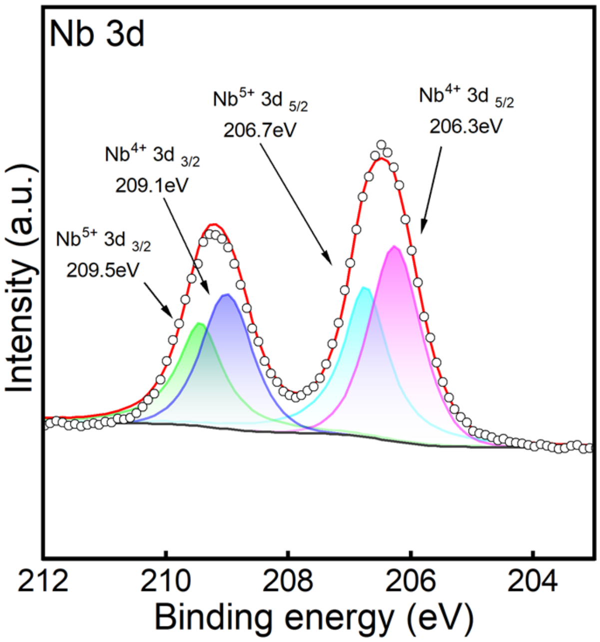

Fig. S7 High-resolution XPS spectra of Nb 3d of α-Nb_2O_5@C at 1.1 V.

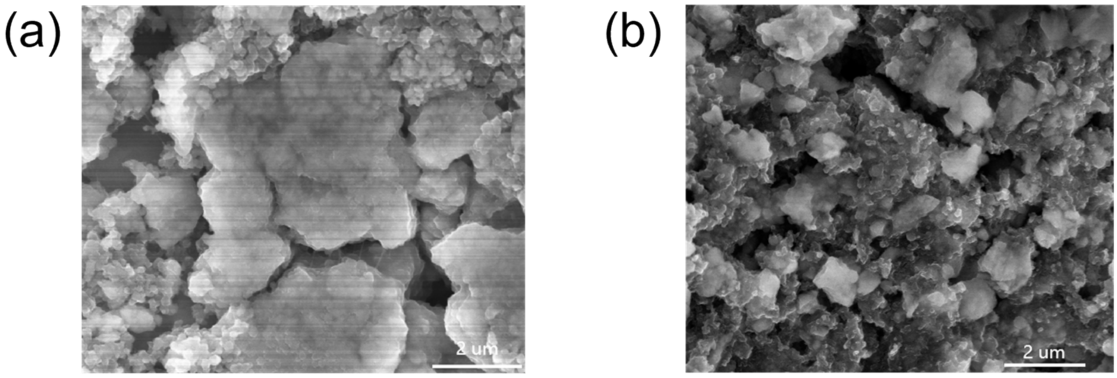

Fig. S8 Post-cycling SEM images of (a) α-Nb_2O_5 and (b) α-Nb_2O_5@C