

SUPPORTING INFORMATION

Nb₂Se₂C: A New Compound as a Combination of Transition Metal Dichalcogenide and MXene for Oxygen Evolution Reaction

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Experimental Section

Preparation of samples. The powder samples of Nb₂Se₂C were synthesized by the traditional high temperature solid-state synthesis method. Starting materials of Nb powder, Se powder, and C powder were mixed stoichiometrically with an excess of C₆Cl₆ (6 wt%) and ground thoroughly in an agate mortar. The mixture was transferred into a silica tube. After flame sealing under vacuum (<10⁻³ torr), the tube was heated to 1473 K at 5 K/min and kept at 1473 K for 2 days and then cool down with the furnace. The synthesis method of NbSe₂ is the same as that of Nb₂Se₂C mentioned above, except that the starting materials were replaced by Nb powder (0.315 g) and Se powder (0.185 g), and the temperature procedure was replaced by holding at 1073 K for 1 day. The Nb₂C samples were purchased from 11 technology Co., Ltd. The NiO (99.9% metals basis, 50 nm) samples were purchased from Aladdin.

Characterization. The power X-ray diffraction data (XRD) were collected using a powder diffractometer (Bruker D8 Advance) at room temperature (filtered Cu K α radiation). The patterns were recorded in a slow-scanning mode with 2θ from 5° to 100° with a scan rate of 1°/min. The transmission electron microscopy (TEM) images were collected on a JEOL-2100F TEM operated at an acceleration voltage of 200 kV. The microtopography and composition of materials were detected by the scanning electron microscopy (SEM, JSM-6510) coupled with energy dispersive X-ray spectroscopy (EDX). The electrical properties of Nb₂Se₂C were studied using a Quantum Design Physical Properties Measurement System (PPMS). The resistivity was measured in the temperature range of 2-300 K. The absorption spectrum of the sample was measured on a Hitachi U4100 UV-VIS-NIR spectrometer at room temperature.

Electrochemical measurement. The electrochemical measurements were carried out using a CHI760E electrochemistry workstation in a traditional three-electrode cell. The ink was obtained after sonication of ethanol (950 μ L) with the catalyst (5 mg) and 5 wt% Nafion solution (50 μ L), and then the dispersion was loaded on the carbon cloth leading to a catalyst loading of ≈ 2.65 mg cm⁻². The ink was dried in air and served as the work electrode. The counter and reference electrodes were carbon rod and Hg/HgO electrode, respectively. The overpotential (η) was calculated from the formula: $\eta = E(\text{RHE}) - 1.23$ V, and $E(\text{RHE}) = E(\text{Hg/HgO}) + 0.059 \times \text{pH} + 0.098$ V in 1.0 M KOH (pH = 13.8). O₂ was poured into the KOH electrolyte for 0.5 h before experiments. O₂-saturated 1.0 M KOH was used as the electrolyte. Polarization curves were obtained using linear sweep voltammetry (LSV) mode at a scan rate of 5 mV s⁻¹ in the range of 1.1 to 1.7 V vs. RHE. To estimate the electrochemical active surface area (ECSA) of the catalyst, cyclic voltammogram (CV) was tested to measure the double-layer capacitance (C_{dl}) under the potential window of 1.31 to 1.41 V vs. RHE with various scan rates of 20-120 mV s⁻¹. Long-term stability tests were performed by chronoamperometric measurement at the overpotential of 350 mV.

Calculation of electronic structure. First-principles calculations were performed by using the Projected Augmented Wave Method (PAW)¹ within the density functional theory (DFT) as implemented in the Vienna Ab Initio Simulation Package (VASP)² with the experimental structures. The exchange correlation functional was treated within the generalized gradient approximation (GGA) and parameterized in the form of Perdew-Burke-Ernzerhof (PBE) version.³ The cutoff energy of plane wave basis was set to 600 eV and the energy convergence criterion was 10⁻⁶ eV. The Gamma-centered 16 \times 16 \times 6 k-mesh was used for Brillouin zone (BZ) sampling.

Calculation of carrier concentration. The calculation of carrier concentration is implemented by the following formula:

$$\text{slope} = \frac{R_{\text{hall}}}{B} = \frac{W_e}{DqW_s n}$$

Table S1. The meaning of symbols in the formula for the calculation of carrier concentration.

R_{hall}	the Hall resistance of the sample
B	the magnetic flux density
W_e	the distance between electrodes
W_s	the width of the sample
D	the height of the sample along the direction of B
q	the charge of the carrier
n	the carrier concentration

The slope is $1.528\text{E-}5 \text{ } \Omega \text{ T}^{-1}$ obtained by the linear fitting of the $R_{\text{hall}}-B$ curves (**Figure 3d**), and then the carrier concentration is calculated to be $6.82 \times 10^{16} \text{ m}^{-3}$.

Accession Codes

Further details of the crystal structure investigations may be obtained from the Fachinformationszentrum Karlsruhe, 76344 Eggenstein-Leopoldshafen (Germany), on quoting the depository number CSD-1982116.

Supplementary figures

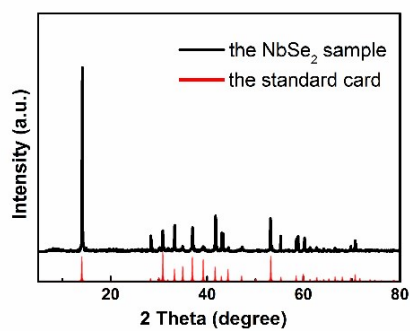


Figure S1. The XRD patterns of the NbSe₂ sample and the standard card (#72-1621).

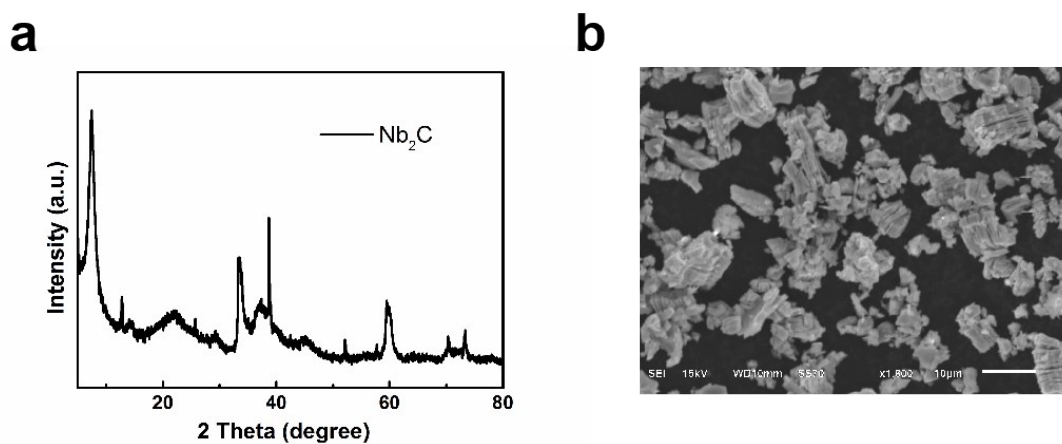


Figure S2. The XRD pattern (a) and the SEM image (b) of the Nb₂C sample.

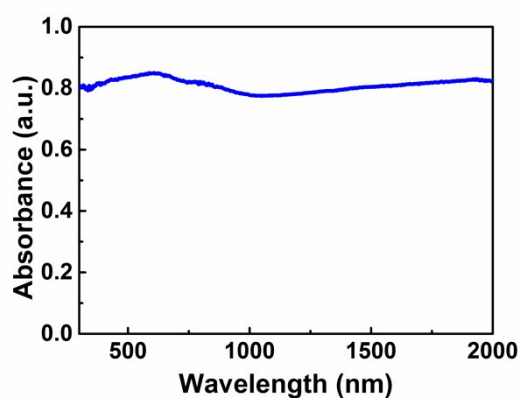


Figure S3. The absorption spectrum of Nb₂Se₂C.

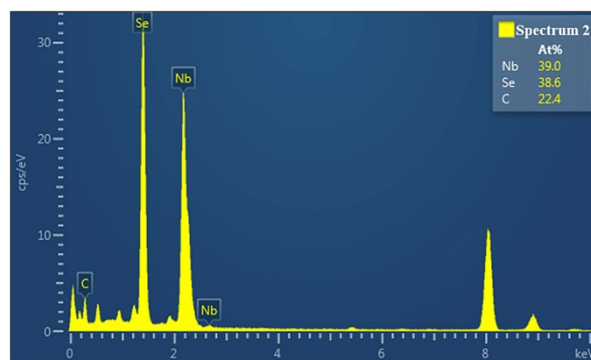


Figure S4. Elemental compositions of Nb, Se, and C in $\text{Nb}_2\text{Se}_2\text{C}$ by EDS. The molar ratio of Nb and Se is close to 1:1.

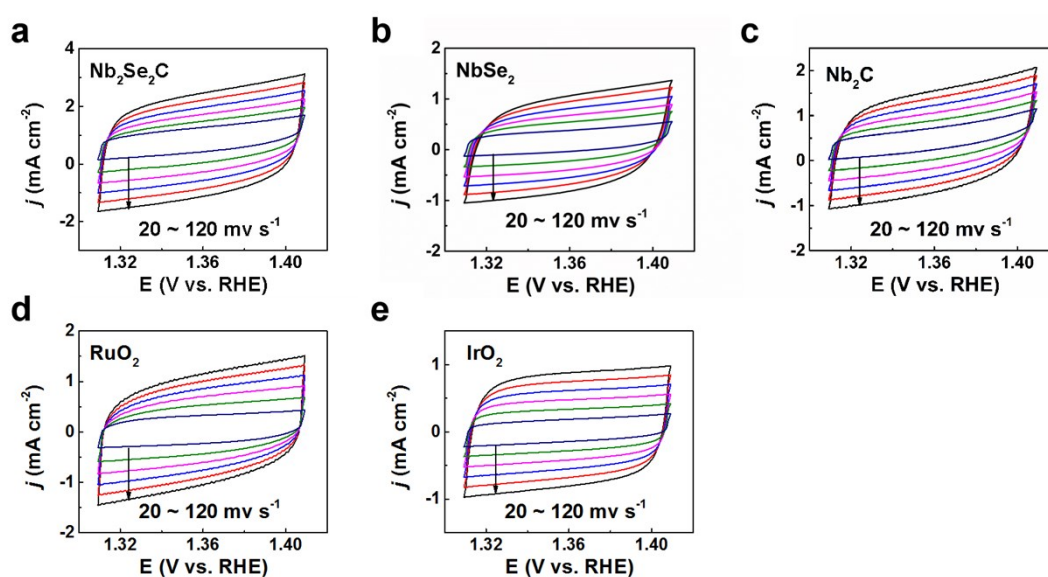


Figure S5. Cyclic voltammogram (CV) curves of (a) $\text{Nb}_2\text{Se}_2\text{C}$, (b) NbSe_2 , (c) Nb_2C , (d) RuO_2 , and (e) IrO_2 tested under the potential window of 1.31 to 1.41 V vs. RHE with various scan rates from 20 to 120 mV s^{-1} .

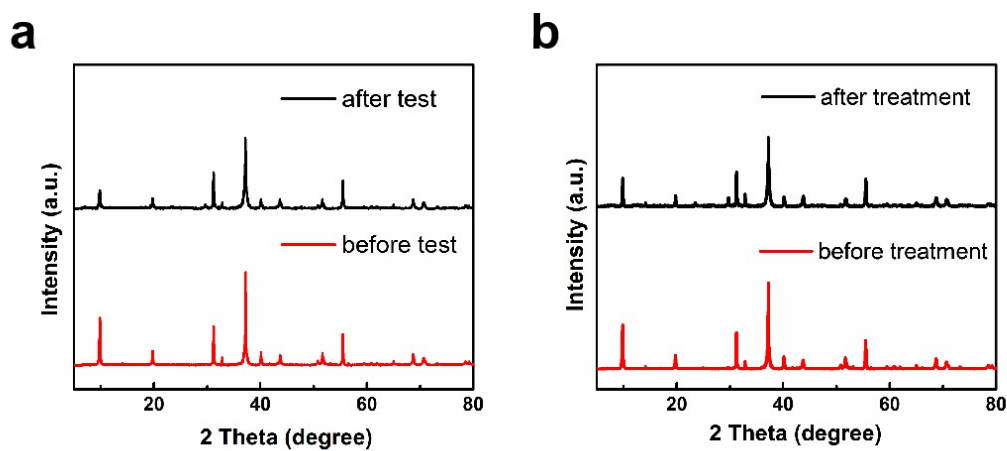


Figure S6. (a) The X-ray diffraction patterns of Nb₂Se₂C samples before (the red lines) and after (the black lines) the long-term durability test. (b) The X-ray diffraction patterns of Nb₂Se₂C samples before (the red lines) and after (the black lines) the heat treatment in air at 573 K for 3 h.

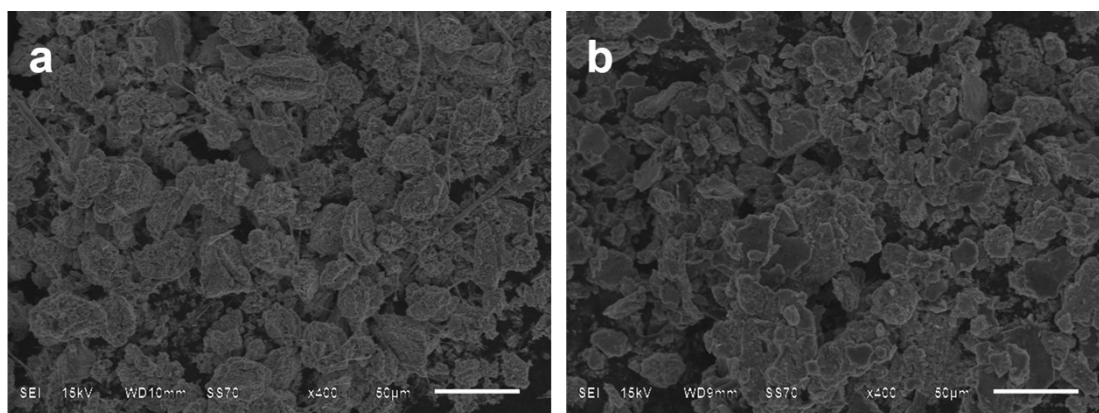


Figure S7. The SEM images of Nb₂Se₂C samples before (a) and after (b) the long-term durability test.

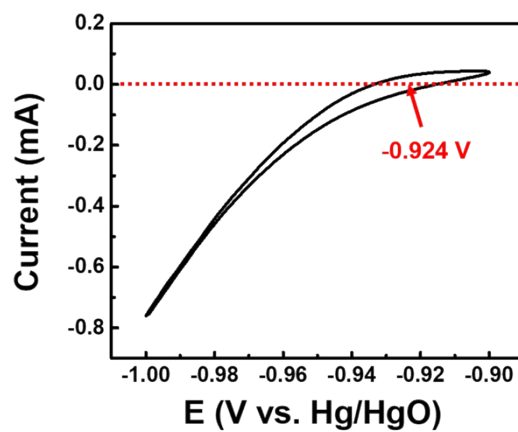


Figure S8. The calibration curves for reference electrode in 1 M KOH supporting electrolyte.

Reference

1. P. E. Blöchl, *Phys. Rev. B*, 1994, **50**, 17953.
2. G. Kresse and J. Furthmüller, *Phys. Rev. B*, 1996, **54**, 11169.
3. J. P. Perdew, K. Burke and M. Ernzerhof, *Phys. Rev. Lett.*, 1996, **77**, 3865.