Supporting Information

Chemoresistive Properties of NiO-Surface-Modified Nb-doped TiO_2 Mesoporous Thin Films

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Author Contributions

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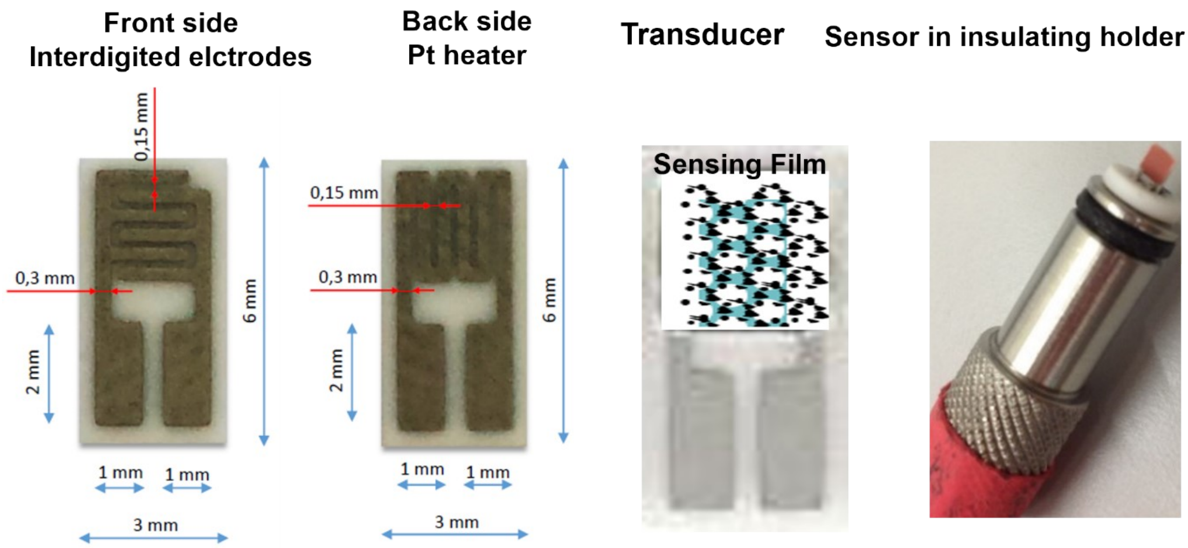

Figure S1. Schematic of the transducer used to record the chemoresistive properties of NiO–$NbTiO_x$ thin films in a resistive-type gas sensor. It consists of a film of sensing material electrically connected *via* two metal terminals and positioned on a heatable support. The substrate (6 × 3 × 0.4 mm) has a rectangular planar geometry, onto which interdigitated electrical contacts and a heater, both made of platinum, are deposited by screen printing.[1-3] All thin films, including mesoporous TiO_2 and $NbTiO_x$, were directly deposited on top of the electrodes in the area highlighted as the "Sensing Film."

Measurements were carried out in a stainless-steel chamber comprising of a support body with radial holes for sample holders, a splitter, and a bottom cover with gas inlet and outlet manifolds. The chamber was designed to ensure test reproducibility over time, minimize internal volume for fast evacuation, prevent recirculating or stagnant flows that could distort sensor responses, and maintain quasi-steady flow conditions by avoiding velocity gradients. All tests were conducted in a reference atmosphere simulating air (80% nitrogen and 20% oxygen). Initial conditioning involved exposing the sensors to this atmosphere at 400°C for one hour to eliminate impurities. Then, under the same conditions, the sensors were tested at progressively lower temperatures to assess their electrical properties and baseline resistance values. Based on these results, the optimal operating temperature for further testing was selected. Six sensors underwent the following test cycles: Reference atmosphere tests at 400°C and decreasing temperatures, sensitivity tests using acetone and ethanol at the selected temperature and varying concentrations, the $5NiONbTixO_2$ sensor showed the highest sensitivity at a temperature where it maintained good stability and low resistance, stability tests were conducted by exposing the selected sensor to five pulses of the target gas (5 minutes each) at constant temperature and concentration, and selectivity tests were performed using a gas mixture at the operating temperature, with similar concentrations, to evaluate cross-sensitivity.[1,2]

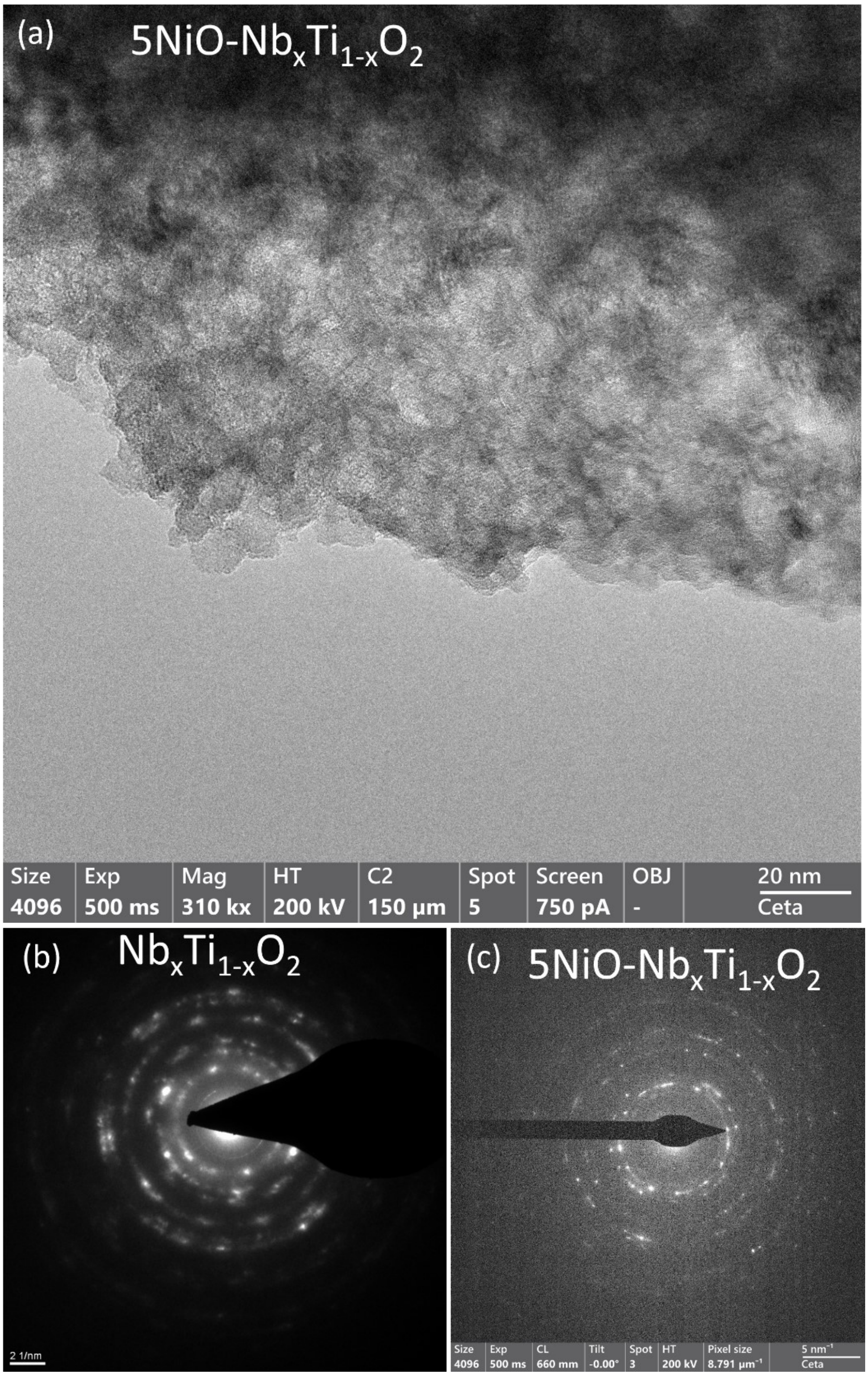

Figure S2. (a) BF-HRTEM image for *5*NiO-$Nb_xTi_{1-x}O_2$, selected-area electron diffraction patterns (SAED) of (b) $Nb_xTi_{1-x}O_2$, and (c) for *5*NiO-$Nb_xTi_{1-x}O_2$ samples.

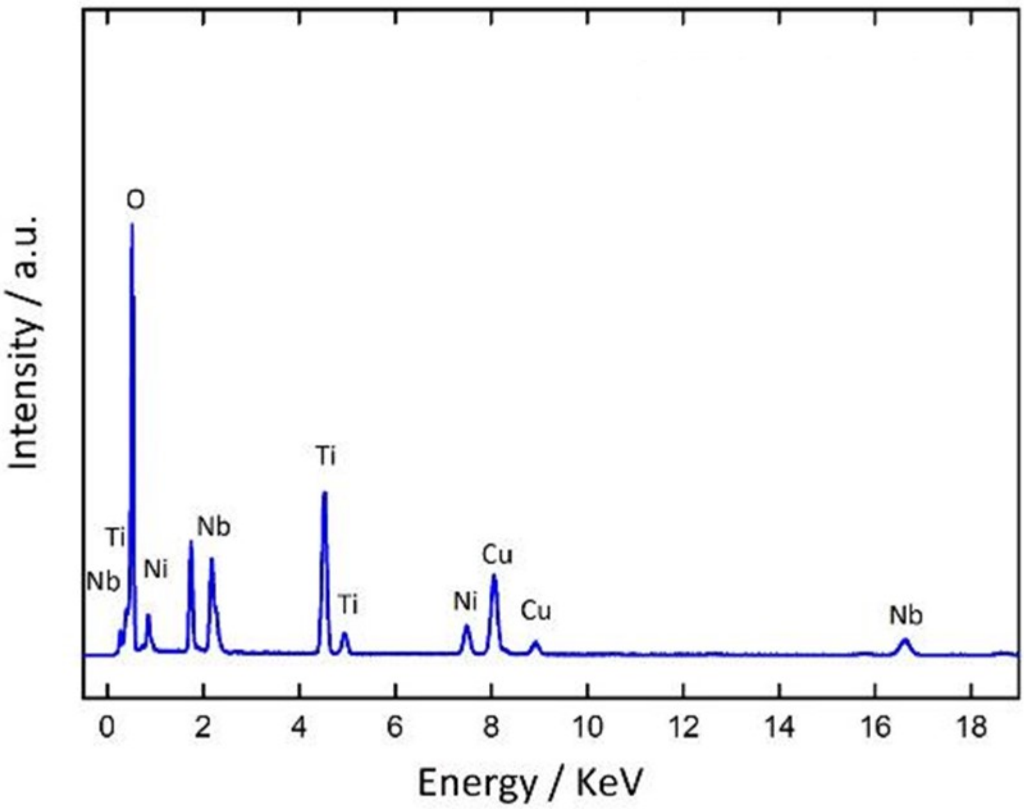

Figure S3. EDX spectra corresponding to the HAADF-STEM EDX elemental mapping shown in **Figure 3**. The copper signal arises because the EDX spectra and HAADF-STEM EDX was recorded by loading the sample onto a carbon-coated TEM copper grid.

References

(1) Dhahri, R.; Hjiri, M.; Mir, L. E.; Bonavita, A.; Iannazzo, D.; Leonardi, S. G.; Neri, G. CO sensing properties under UV radiation of Ga-doped ZnO nanopowders. *Applied Surface Science* **2015**, *355*, 1321-1326. DOI: https://doi.org/10.1016/j.apsusc.2015.08.198.

(2) Hjiri, M.; Neri, G. Photo-Activated Ga-ZnO Gas Sensor for NO2 Detection at Near Ambient Temperature. *Journal of Inorganic and Organometallic Polymers and Materials* **2024**, *34* (8), 3374-3383. DOI: 10.1007/s10904-023-02934-z.

(3) Neri, G.; Bonavita, A.; Rizzo, G.; Galvagno, S.; Pinna, N.; Niederberger, M.; Capone, S.; Siciliano, P. Towards enhanced performances in gas sensing: SnO2 based nanocrystalline oxides application. *Sensors and Actuators B: Chemical* **2007**, *122* (2), 564-571. DOI: https://doi.org/10.1016/j.snb.2006.07.006.