

Aliovalent Fe (III)-doped NiO microspheres for enhanced butanol gas sensing properties

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Characterization

The crystal structure and phase are conducted on a Scintag XDS-2000 X-ray diffractometer with Cu K α radiation ($\lambda=1.5418 \text{ \AA}$). Scanning electron microscopy (SEM) images are performed on a S-4800 (Japan) instrument. Transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) observations are conducted on a TF20 (Japan) instrument. The specific surface area is studied through N₂ adsorption-desorption isotherms are measured at 77 K with a Micromeritics ASAP 2020 HD88 system. The chemical component is characterized by X-ray photoelectron spectroscopy (XPS) is measured on a (VG, Britain) with Mg K α excitation. The doping concentration of Fe in various Fe-doped NiO materials is determined by inductively coupled plasma-atomic emission spectroscopy (ICP-AES, NexION 300 X).

Fabrication and measurement of gas sensor

A pair of Au electrodes is mounted at each end of the ceramic tube. Each electrode is connected with two Pt wires. The samples are dispersed in deionized water to form slurry. Each sample powder is then coated onto a ceramic tube to form a thick sensing film. The Ni-Cr alloy coil heater is inserted into an alumina tube. We can control the operating temperature by regulating the heating current that through the heater.

Typically, 50 mg·mL⁻¹ the pure and Fe-doped NiO multi-shelled microspheres-butanol solution are deposited on the surface of the device and calcined at 120 °C for 3 h. In this work, the response of the sensor is defined as R_a/R_g (n-type) or R_g/R_a (p-type), where R_a is the resistance of the gas sensor in the air and R_g is the resistance of the target gases. τ_{res} and τ_{rec} are defined as the time required for the sensor to achieve 90 % of the total resistance change in the case of response and recovery, respectively.

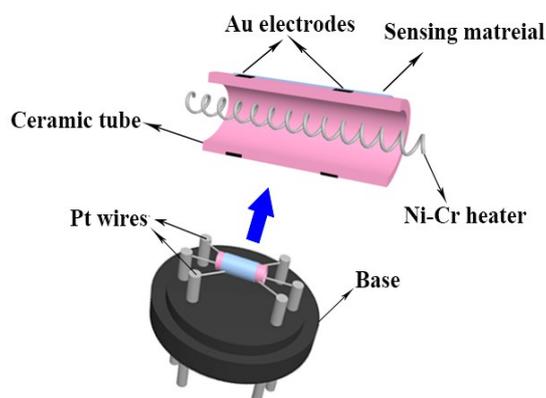


Fig. S1 Schematic diagram of the gas sensor.

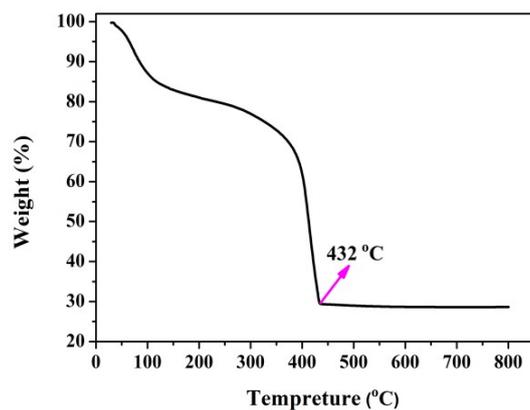


Fig. S2 TG curves of NiO microspheres precursor

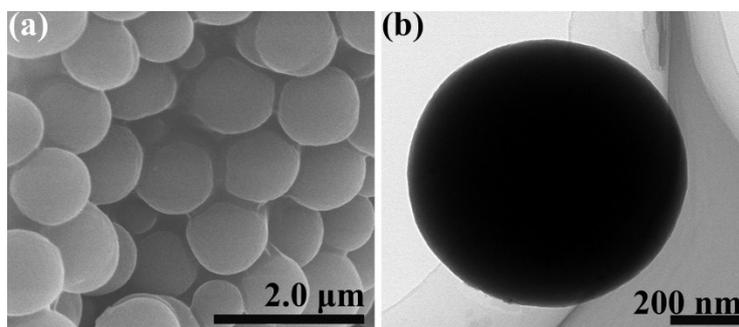


Fig. S3 (a) SEM images, (b) TEM image of the pure NiO microspheres precursor.

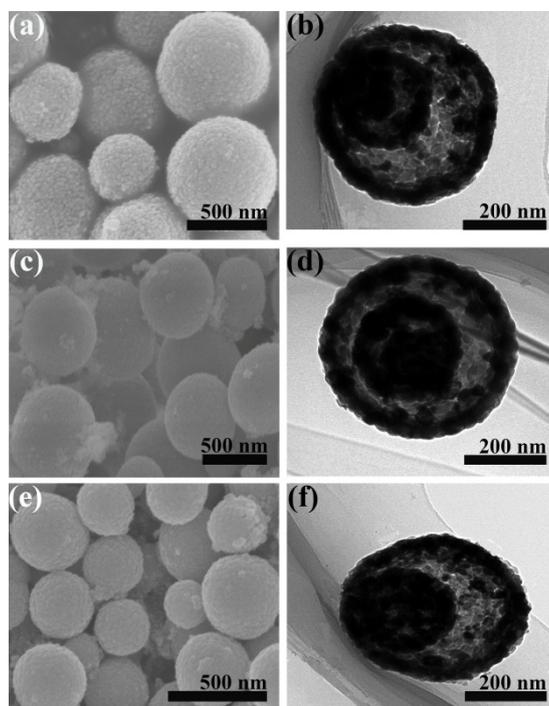


Fig. S4 (a, c, e) SEM images, (b, d, f) TEM image of the 0.91, 3.74 and 4.64Fe-NiO multi-shelled microspheres, respectively.

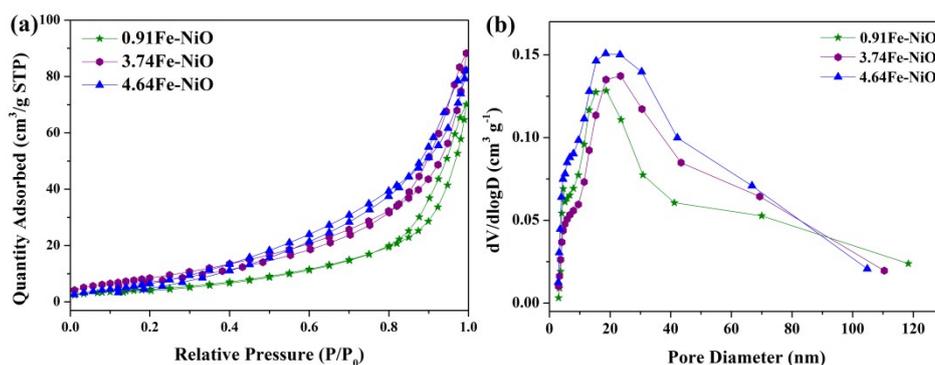


Fig. S5 (a) N₂ adsorption–desorption isotherms and (b) pore size distributions of 0.91Fe-NiO, 3.74Fe-NiO and 4.64Fe-NiO multi-shelled microspheres.

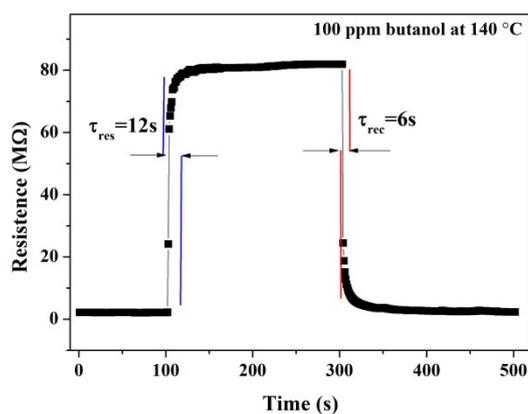


Fig. S6 The response and recovery times of the sensor based on the 1.92 Fe-NiO to 100 ppm butanol at 140 °C.

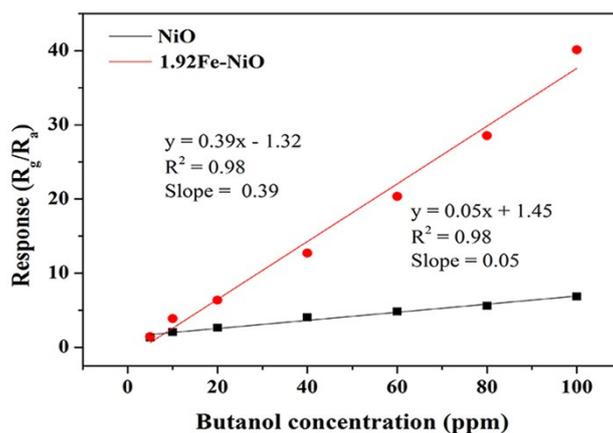


Fig. S7 Responses of the pure NiO and 1.92Fe-NiO sensors as a function of low butanol concentration (5–100 ppm).

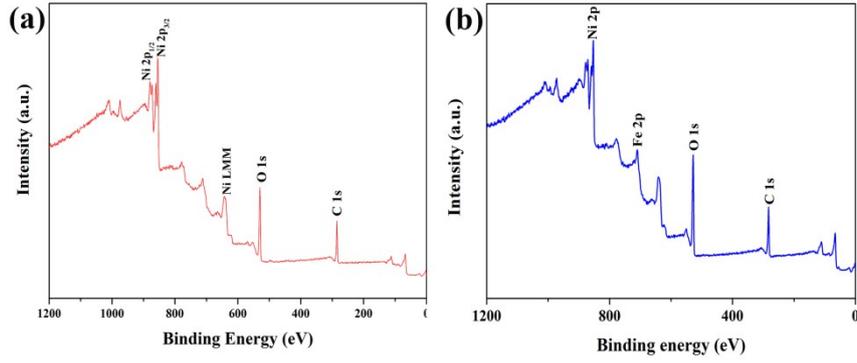


Figure. S8 The full-range XPS spectrum of the pure NiO (a) and 1.92Fe-NiO (b) multi-shelled microspheres.

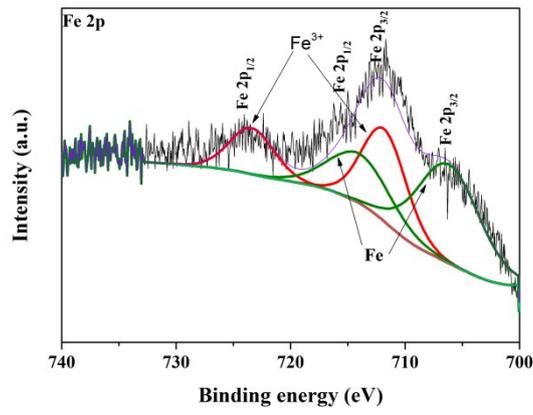


Figure. S9 The XPS spectra of the 1.92Fe-NiO multi-shelled microspheres: Fe 2p

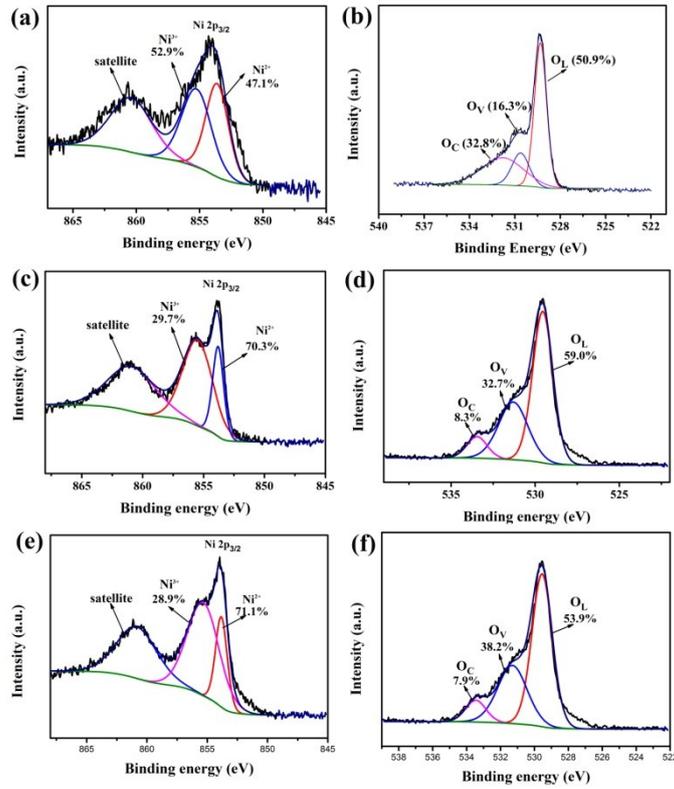


Fig. S10 (a, c, e) Ni 2p_{3/2} XPS spectra and (b, d, f) O 1s XPS spectra of the 0.91Fe-NiO, 3.74Fe-NiO and 4.64Fe-NiO multi-shelled microspheres, respectively.