

Supplementary Materials – KIT-5-assisted Synthesis of Mesoporous SnO₂ for High-Performance Humidity Sensors with a Swift Response/Recovery Speed

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Transmission electron microscopy

Table S1 Comparison between theoretical (JCPDF 01 077-0452) and observed *d*-values of SnO₂-KIT-5 and SnO₂-R powders.

(hkl)	SnO ₂ -KIT-5 <i>d</i> _{hkl} /Å	SnO ₂ -R <i>d</i> _{hkl} /Å	SnO ₂ (JCPDF 01 077-0452) <i>d</i> _{hkl} /Å
(110)	3.421	3.374	3.362
(101)	2.715	2.664	2.654
(200)	2.432	2.364	2.378
(111)	2.356	2.325	2.318
(210)	2.170	2.132	2.127
(211)	1.821	1.776	1.771

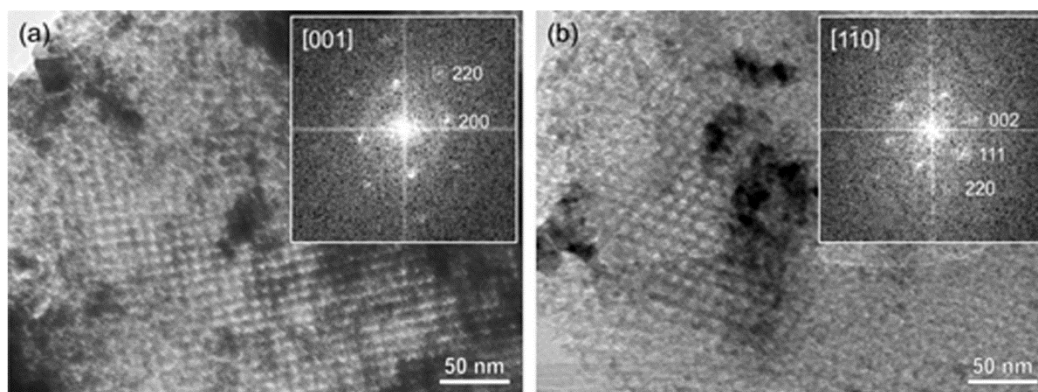


Figure S1. TEM analysis of KIT-5 pore distribution within SnO₂-KIT-5: (a) [001] projection, (b) [110] projection.

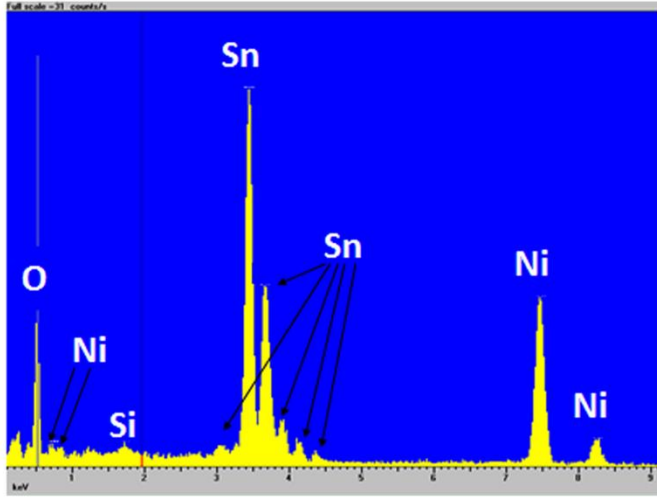


Figure S2. EDXS analysis.

X-ray diffraction analysis

The XRD patterns of SnO₂-R and SnO₂-SG were shown in Figure S3. The average crystallite size of the SnO₂-R and SnO₂-SG particles was calculated by applying the Debye-Scherrer equation, Equation (S1):

$$L = K\lambda / \Delta 2\theta \cos \theta \quad (S1)$$

where L is the mean size of the ordered (crystalline) domains, K is a dimensionless shape factor with a value close to unity, λ wavelength (0.154 nm⁻¹), $\Delta 2\theta$ is the line broadening at half of the maximum intensity (FWHM), and θ is the Bragg angle. It appeared to be 10 nm for SnO₂-R and 43 nm for SnO₂-SG.

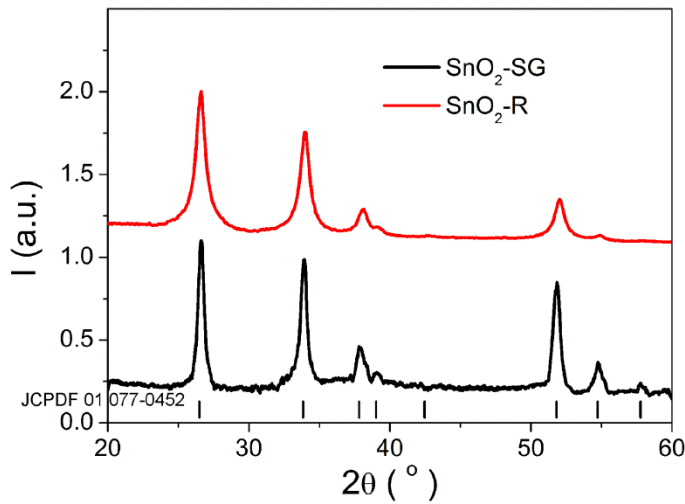


Figure S3. XRD patterns of SnO₂-SG and SnO₂-R powders.

BET

Using the S_{BET} values of the SnO₂-R and SnO₂-SG and the formula for the BET equivalent particle diameter, Equation (S2):

$$L_{\text{BET}} = (\Psi_A \cdot \Psi_V^{-1}) / (S_{\text{BET}} \cdot \rho_t) \quad (S2)$$

where $(\Psi_A \cdot \Psi_V^{-1})$ is the shape factor ratio (≈ 6), S_{BET} is the specific surface area of the powder and ρ_t is the theoretical density of SnO₂ (6.95 g/cm³) [45], the primary particle size values were calculated to be 12.9 and 42.3 nm for the SnO₂-R and SnO₂-SG, respectively, and they are in agreement with WAXS-XRD/TEM findings.

SAXS Parameters Analytics

The spectra of the same capillary (=the background) were subtracted after merging the spectra and the background spectra at the same transmission line. The radius of gyration R_g and $I(0)$ were determined *via* the Guinier approximation for globular particles (see Equation S3).

$$I(q) = I(0) \cdot \exp\left(-q^2 \cdot \frac{R_g^2}{3}\right) \quad (S3)$$

The surface S to volume V ratio was determined using the approximation derived from Porod's law (see Equation S4) for line-collimated experiments.

$$S/V = 4 \cdot \frac{K_l}{Q_l} \cdot \varphi \cdot (1 - \varphi) \quad (S4)$$

Here φ is the volume fraction, which was set to $\varphi = 0.5\%$ assuming the general solid state/air ratio for solid samples after transmission normalisation [43,44]. Q_l (the invariant of the first moment of the smeared intensity) was determined by:

$$Q_l = \int_0^\infty q \cdot I(q) dq \quad (S5)$$

and K_l (the invariant of the first moment of the smeared intensity) was determined by fitting:

$$I(q) = K_l/q^3 \quad (S6)$$

All data were evaluated using SAXS quant 3.100 (Anton Paar GmbH, Austria).

Complex impedance analysis

The change in complex impedance over 30–90% RH range for TF(SnO₂-R) and TF(SnO₂-SG), measured at 50 °C is shown in Figure S4(a). A 1.9 order of change in the impedance magnitude in the 30-90% RH range is observed for the TF(SnO₂-R) sensor, while these values for the and TF(SnO₂-SG) is found to be 0.35 order.

Figure S4(b) presents the frequency dependence of the complex impedance of TF(SnO₂-R) sensor measured in the relative humidity 30–90% RH range at 50 °C. With increase in RH the impedance decreases more slowly with frequency, especially at high RH (above 70% RH). In addition, an increase in RH from 30% to 90% more noticeable influences a decrease of complex impedance in the lower frequency range. For example, at 42 Hz the impedance of 17.3 MΩ is measured at 30% RH and it lowers to 102 kΩ at 90% RH. This operating temperature (50 °C) resulted in similar behavior as observed at 25 °C for TF(SnO₂-R) sensor, but with overall lower measured impedances.

Hysteresis curve is measured at 100 Hz and 50 °C for TF(SnO₂-R) sensor, and it is presented in Figure S4(c). The adsorption and desorption curves for the TF(SnO₂-R) sensor nearly overlay each other showing a small hysteresis error of 3.2%, which represents a good reliability of this sensor.

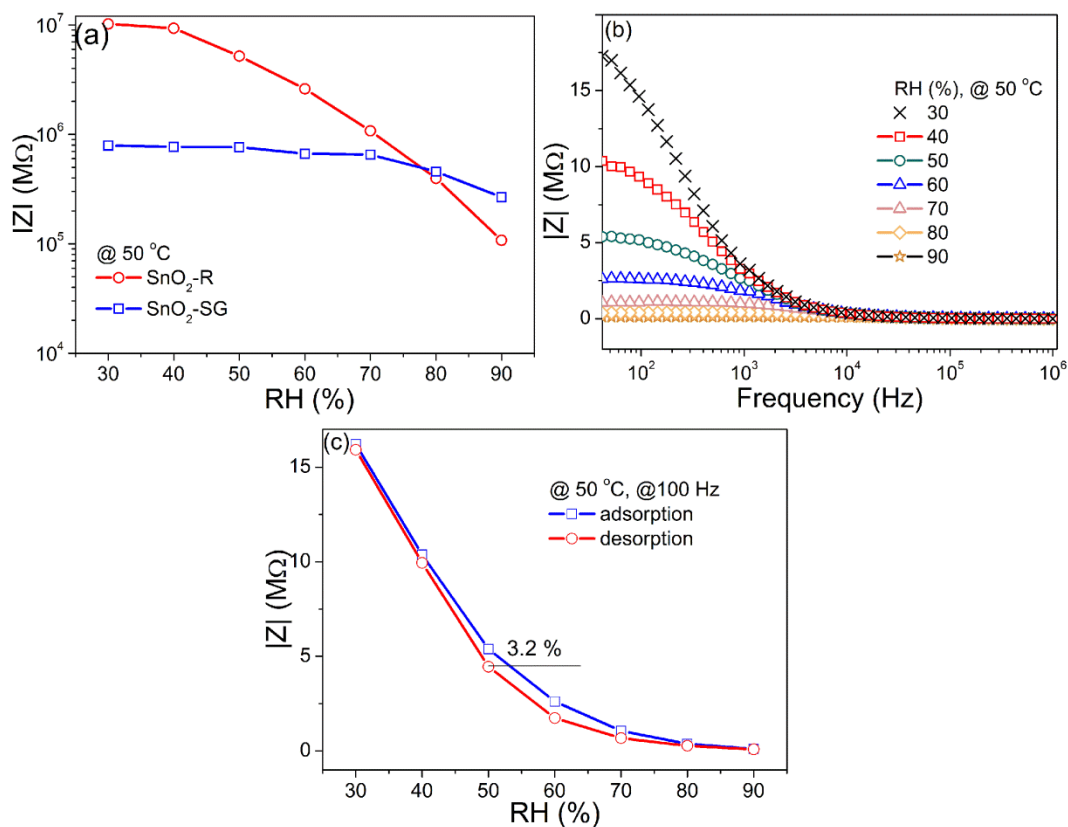


Figure S4. (a) The humidity sensing curves of fabricated, SnO₂-R and SnO₂-SG sensors presented as the change of complex impedance with a relative humidity change in the 30–90% RH range; (b) the change of the complex impedance with frequency of the TF(SnO₂-R) sensor at 50 °C exposed to the certain RH values; (c) hysteresis behavior of the TF(SnO₂-R) over 30-90% RH range at 50 °C.

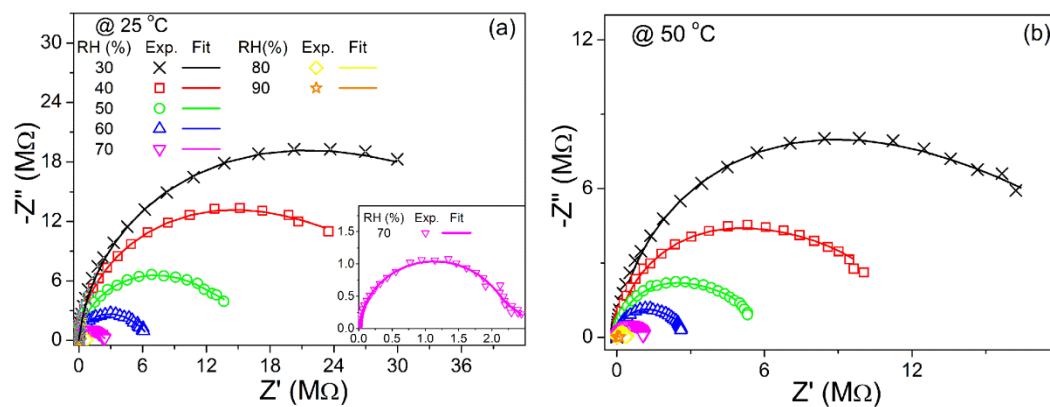


Figure S5. Cole-Cole plots of the TF(SnO₂-R) sensor measured over 30-90% RH range at the temperature (a) 25 °C and (b) 50 °C; experimental data are presented by symbols, simulated by lines.

Table S2 The area under deconvoluted O₁-O₃ and O_{tot.} peaks from the high-resolution O 1s spectra of different SnO₂.

Sample	Area under the O ₁ -O ₃ and O _{tot.} peaks						
	O ₁	O ₂	O ₃	O _{tot.}	O _{latt.} O ₁ /O _{tot.}	O _{deff.} [O ₂ +O ₃ /O _{tot.}]	[O _{deff.} /O _{latt.}]
TF(SnO ₂ -SG)	37025	6405	1417	44849	0.8255	0.1744	0.2112
TF(SnO ₂ -R)	34030	8549	2020	44599	0.7630	0.2370	0.3106

References

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