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## **Supplementary Information**

## Single-Step Growth of p-type 1D Se/2D GeSe<sub>x</sub>O<sub>y</sub> Heterostructures for Optoelectronic NO<sub>2</sub> Gas Sensing at Room Temperature

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Fig. S1 (a) The crystal structure of layered GeSe, in which grey balls present Ge and yellow balls present Se atoms. (b) The XRD pattern FESEM image (insert) of bulk

GeSe.



Fig. S2 The FE-SEM images of the drop-casted dispersion dried in the silicon substrate after leaving in a sealed environment for (a) 0, (b) 1, (c) 12, (d) 24 h. It is observed that the belt-like morphology only appears after 24 h.



Fig. S3 The FE-SEM images of the drop-casted dispersion sonicated for a) 1, (b) 2, (c) 3, and (d) 4 h dried in the silicon substrate after leaving in a sealed environment for 24 h. It is observed that the belt-like morphology appears in all samples.



Fig. S4 (a) UV-vis absorption spectra of dispersion sonicated in the solvent containing 0, 25, 50, 75, 100 %v/v ethanol. (b) The corresponding optical images.



Fig. S5 The statistical distribution of (a) the height of 2D  $GeSe_xO_y$  nanosheets and (b) the diameter of Se belts.



Fig. S6 TEM-EDS mapping elements of (a) Ge, (b) Se, and (c) O for the Se/GeSe<sub>x</sub>O<sub>y</sub> heterostructure.



Fig. S7  $N_2$  adsorption-desorption isotherms of Se/GeSe<sub>x</sub>O<sub>y</sub>, (b) Pore size distribution of this sample.



Fig. S8 The FE-SEM image of the 1D/2D heterostructures drop-casted onto a transducing substrate.

Table S1 Comparison of sensing performances of the heterostructure towards 10 ppm NO<sub>2</sub> under irradiation of different visible light.

Light source	Wavelength (nm)	Response	Recovery time (min)
No light	-	12.5%	41
Blue	455	17.5%	19
Green	525	23.0%	27
Red	625	27.3%	38



Fig. S9 The FE-SEM images of the sensors based on (a) Se, (c)  $GeSe_xO_y$  and (c) Se/ GeSe<sub>x</sub>O<sub>y</sub>.

Table S2 Comparison of sensing performances of Se belts, GeSexOy nanosheets and Se/GeSexOy heterostructures towards 10 ppm NO2 under irradiation of different visible light.

Materials	Response (%)	Response time	Recovery time
		(min)	(min)
Se	17.6	28	46
GeSe <sub>x</sub> O <sub>y</sub>	20.4	18	31
Se/GeSe <sub>x</sub> O <sub>y</sub>	27.3	19	38



Fig. S10 The optical images of the sensors with (a) 20, (c) 40 and (e) 60  $\mu$ L of the Se and GeSe<sub>x</sub>O<sub>y</sub> dispersion on the transducing substrate and the images of those sensor (inset). (b), (d), and (f) their corresponding dynamic gas response towards the 10 ppm NO<sub>2</sub> gas under red light irradiation.

Volume (µL)	Response time (min)	Recovery time (min)
20	11	32
40	19	38
60	8	29

Table S3 The response and recovery time for the sensor based on different volume dispersions.



Fig. S11 The gas response of 1D Se/2D  $GeSe_xO_y$  heterostructure gas sensor towards 5ppm NO<sub>2</sub> at red light irradiation under different humidity.



Fig. S12 Dynamic resistance changes of 1D Se/2D  $GeSe_xO_y$  heterostructure gas sensor towards 10 ppm NO<sub>2</sub> gas with nitrogen and air under red light irradiation at room temperature.



Fig. S13 (a) SEM, (b) XRD pattern, (c) Raman spectrum, (d) XPS survey spectrum, (e) Ge 3d spectrum, and (f) Se 3d spectrum of Se/GeSe<sub>x</sub>O<sub>y</sub> heterostructure after long-term stability test.