

## Supplementary Information

### Ultrathin oxysulfide semiconductors from liquid metal: a wet chemical approach

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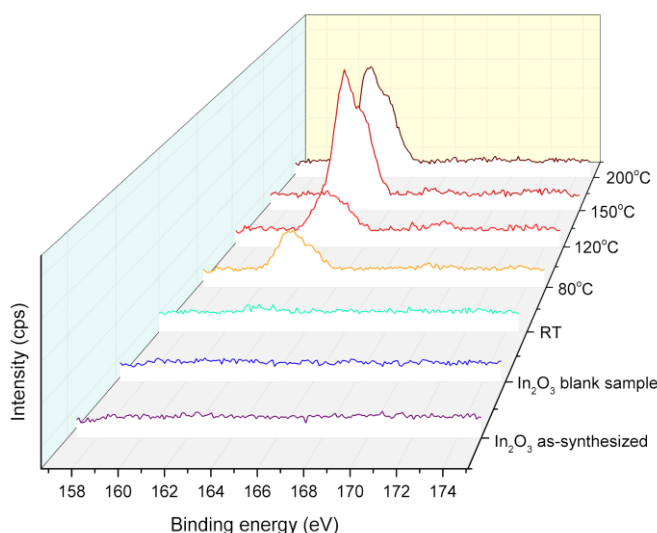
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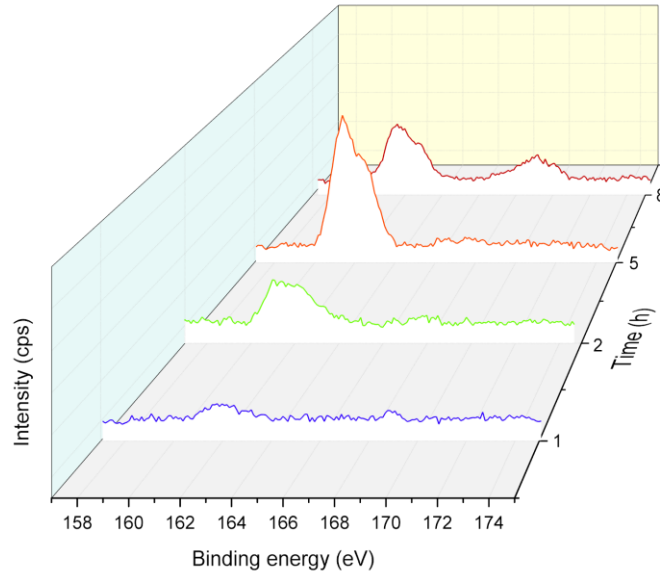
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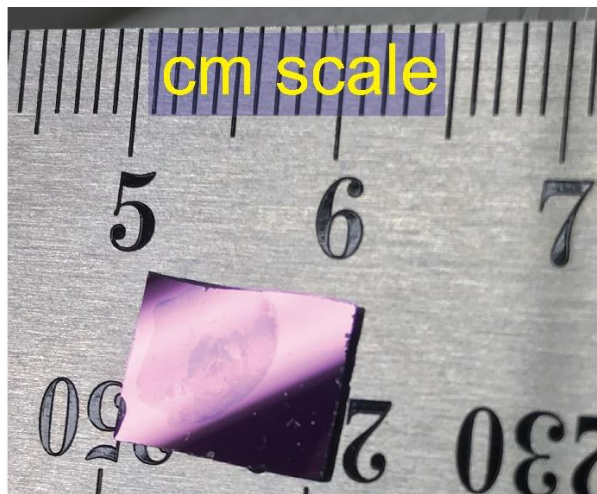
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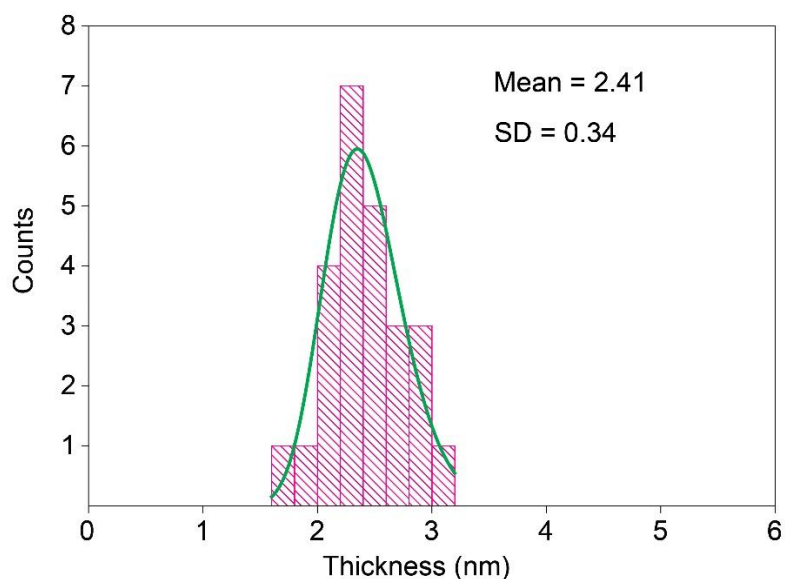
**Figure S1.** XPS spectra of S<sub>2p</sub> taken from samples treated at different temperatures in 5 hours. No signal was detected in the S<sub>2p</sub> regions of In<sub>2</sub>O<sub>3</sub> as-synthesized, blank sample, and the sample treated at room temperature. The S<sub>2p</sub> peaks of S<sup>2-</sup> are located at ~162 eV.<sup>1</sup>



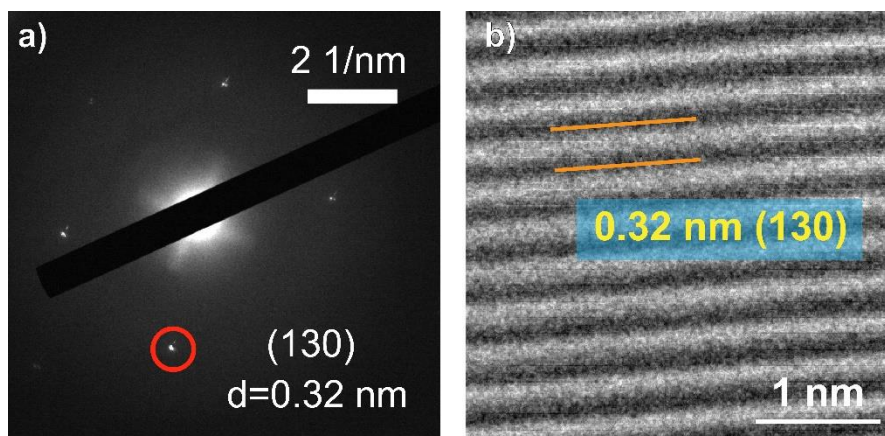
**Figure S2.** XPS spectra of  $S2p$  taken from samples treated at  $150^{\circ}\text{C}$  after different reaction times. The  $S2p$  peaks of  $\text{S}^{2-}$  are located at  $\sim 162$  eV, while the broaden peak at  $169.1$  eV detected in the sample after 8 hours is associated with sulfite/sulfate compounds ( $\text{SO}_4^{2-}$ ).<sup>1, 2</sup>



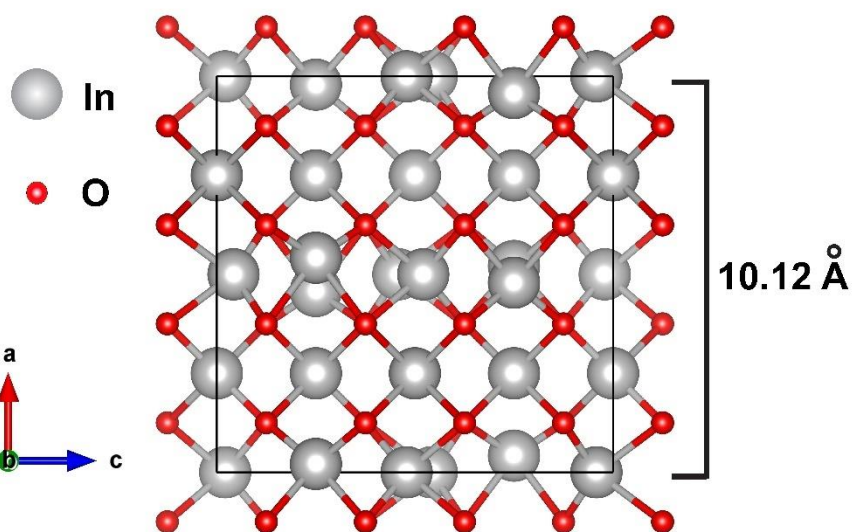
**Figure S3.** A representative  $2\text{D In}_2\text{O}_{3-x}\text{S}_x$  sheet printed on a  $\text{SiO}_2/\text{Si}$  substrate, approaching centimetre scale. It can be observed that the 2D sheet covers a homogeneous area of several millimetres with some inevitable folded regions at the edge and where the molten droplet was placed.



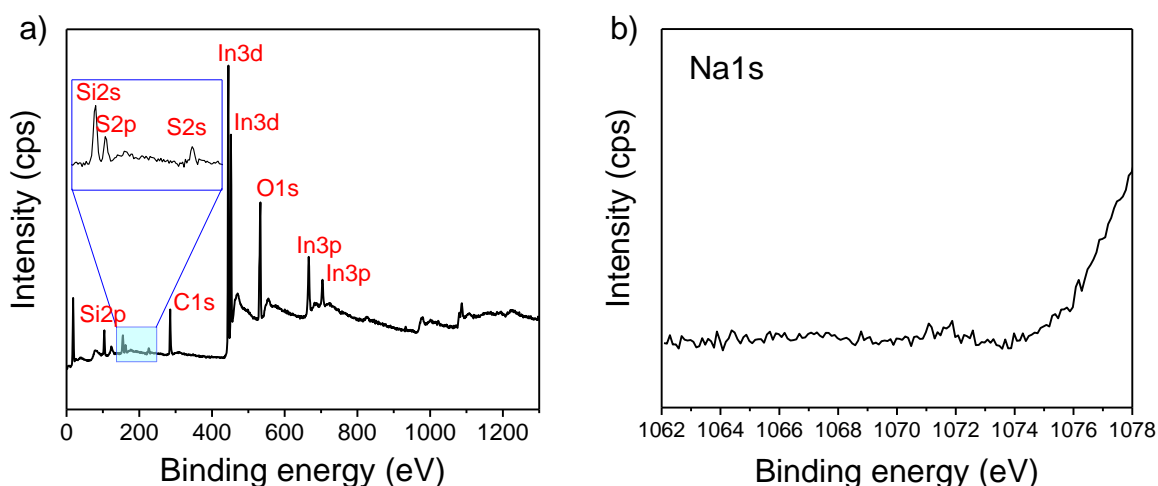
**Figure S4.** Statistical distribution of 2D  $\text{In}_2\text{O}_{3-x}\text{S}_x$  sheets thickness. The mean and standard deviation (SD) were found to be 2.41 nm and 0.34 nm, respectively. The difference between measurements is due to AFM tip-surface interactions, noises from environment, and substrate effects.<sup>3</sup>



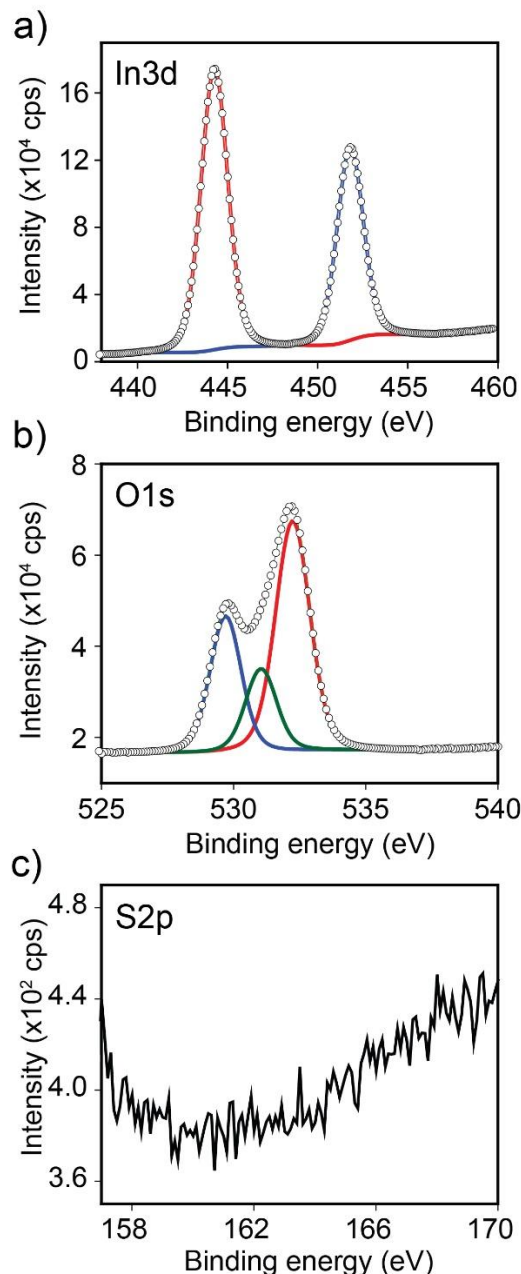
**Figure S5.** SAED pattern and HRTEM image of 2D  $\text{In}_2\text{O}_3$  directly exfoliated from molten indium metal. The diffraction spot in red circle and a lattice spacing of 0.32 nm can be indexed to (130) plane of *c*- $\text{In}_2\text{O}_3$ .<sup>4</sup> The crystal structure of cubic 2D  $\text{In}_2\text{O}_3$  derived from liquid metal has been reported in the previous study.<sup>5</sup>



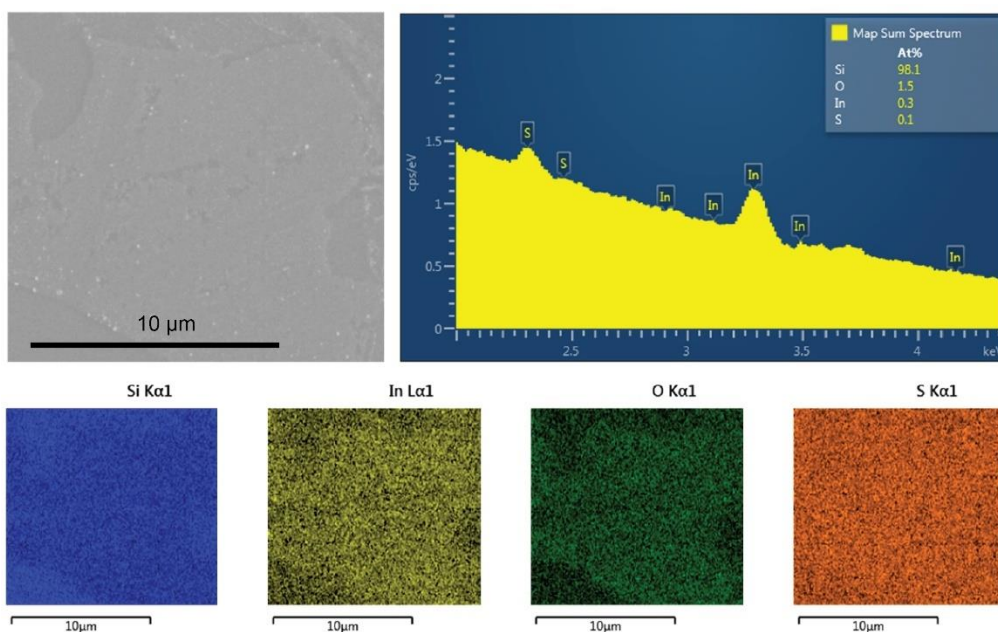
**Figure S6.** Crystal structure of  $c$ - $\text{In}_2\text{O}_3$  with the unit cell thickness of  $10.12 \text{ \AA}$ .<sup>4</sup>



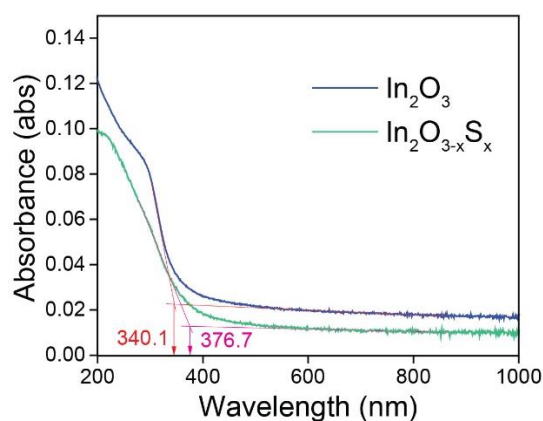
**Figure S7.** (a) XPS survey spectrum of 2D  $\text{In}_2\text{O}_{3-x}\text{S}_x$  nanosheets.  $\text{Si}2p$  and  $\text{Si}2s$  peaks arising from the silicon substrate have been detected at  $103.2 \text{ eV}$  and  $155.5 \text{ eV}$  respectively. (b) High resolution XPS spectrum of  $\text{Na } 1s$  for 2D  $\text{In}_2\text{O}_{3-x}\text{S}_x$  with no peaks being detected, indicating the absence of surface adsorbed  $\text{Na}_2\text{S}$ .



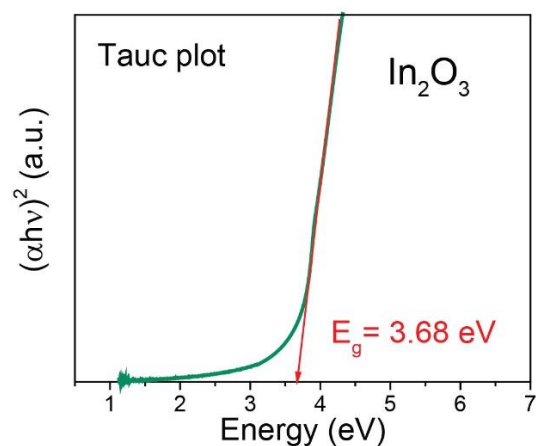
**Figure S8.** XPS spectra of 2D  $\text{In}_2\text{O}_3$  nanosheets. (a) XPS scanning for In 3d: the doublet signal at 444.3 eV and 451.77 eV is associated with In  $3d_{5/2}$  and In  $3d_{3/2}$ , respectively, no signals of indium metal have been recorded. (b) XPS scanning for O 1s: the dominant peak at 532.3 eV is related to Si-O-Si bonds in the substrate. The peak at 529.7 eV is attributed to  $\text{O}^{2-}$  ions in indium oxide lattice. The peak at 531.2 eV may be assigned to C=O bonds originating from surface contamination as well as oxygen atoms near defect sites.<sup>6-11</sup> (c) No peaks were detected in the S 2p region.



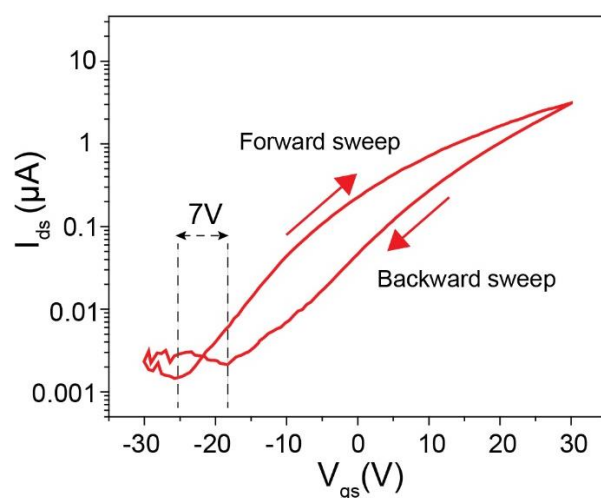
**Figure S9.** SEM-EDX spectrum of a 2D  $\text{In}_2\text{O}_{3-x}\text{S}_x$  nanosheet prepared on 300 nm  $\text{SiO}_2/\text{Si}$  wafer with corresponding maps for the elements Si, In, O, and S. The analysis was conducted at a folded edge region that was thicker and hence provided better signal. The map sum spectrum reveals a In:S ratio of 3:1, with the signal being close to the detection limit. The excess oxygen is due to surface  $\text{SiO}_2$  from the Si substrate. Because of the limitations of SEM-EDX, XPS elemental analysis is better suited and should be referred to when determining the composition of the synthesised nanosheets.



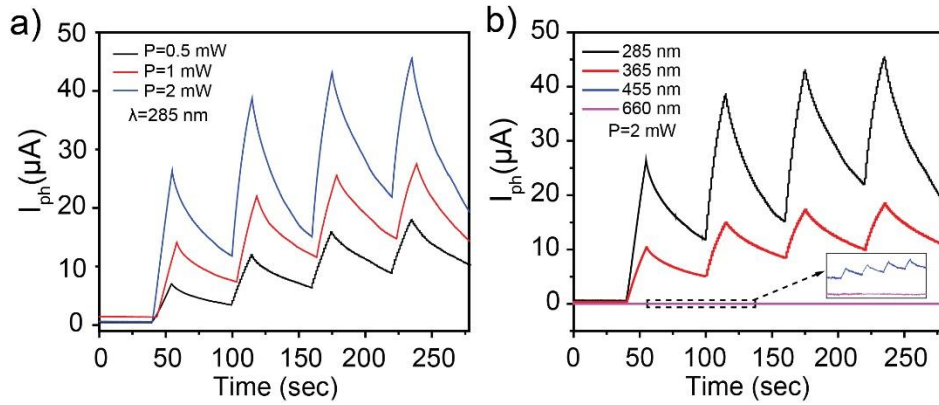
**Figure S10.** UV-vis spectra of 2D  $\text{In}_2\text{O}_3$  and 2D  $\text{In}_2\text{O}_{3-x}\text{S}_x$  ( $x=0.41$ ) indicates the shift in light absorption from 340.1 nm to 376.7 nm.



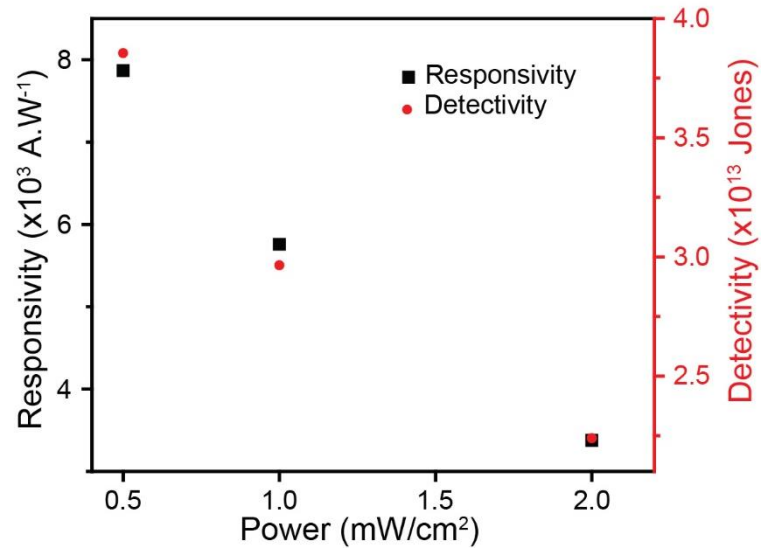
**Figure S11.** UV-vis Tauc plot of 2D  $\text{In}_2\text{O}_3$  sample reveals a direct bandgap of 3.68 eV.



**Figure S12.** The transfer ( $I_{ds}$ - $V_{gs}$ ) curve with 1V source drain bias ( $V_{ds}$ ) shown in a semi-logarithmic scale. The dashed black lines indicate the change in the position of the charge neutrality point. Red arrows indicate the back-gate sweep direction. The hysteresis can be assigned to the trapping and de-trapping of charge carriers by adsorbed water molecules or by trap states on the dielectric substrate.<sup>12, 13</sup>

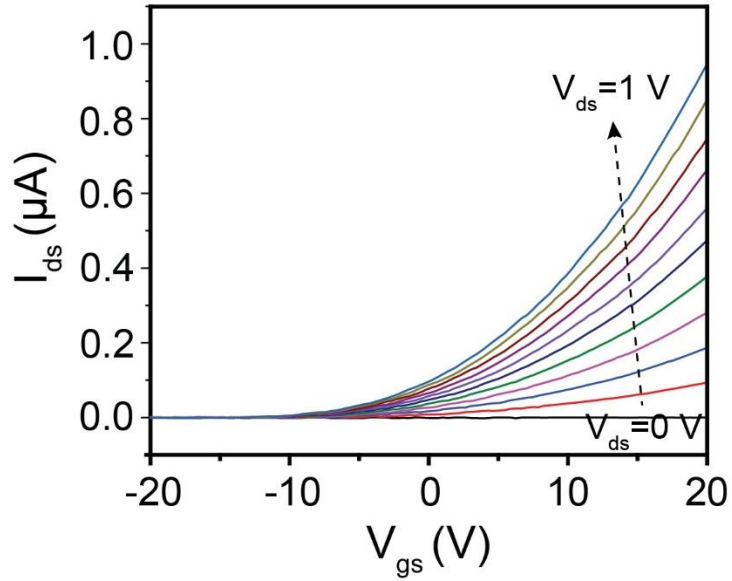


**Figure S13.** The photocurrent response of 2D  $\text{In}_2\text{O}_{3-x}\text{S}_x$  based device as a function of time under (a) different applied powers of the incident light and (b) different wavelengths. The  $V_{ds}$  was set at 0.5 V.

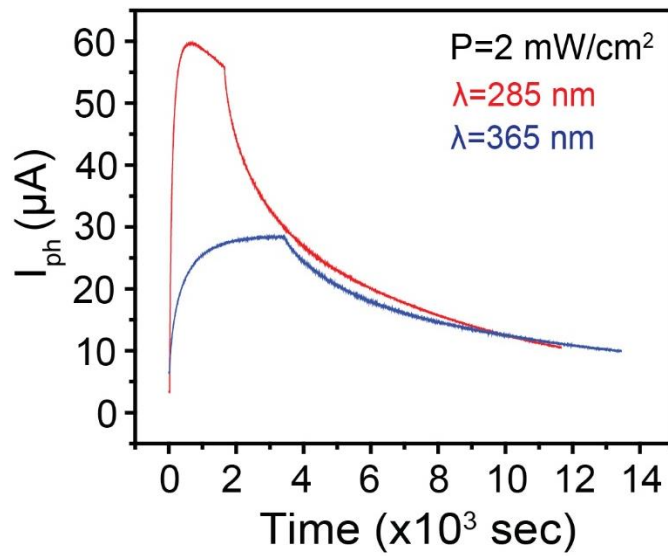


**Figure S14.** Responsivity and detectivity values of 2D  $\text{In}_2\text{O}_{3-x}\text{S}_x$  based device at different power intensities, with the wavelength of 285 nm and 0.5 V bias voltage.

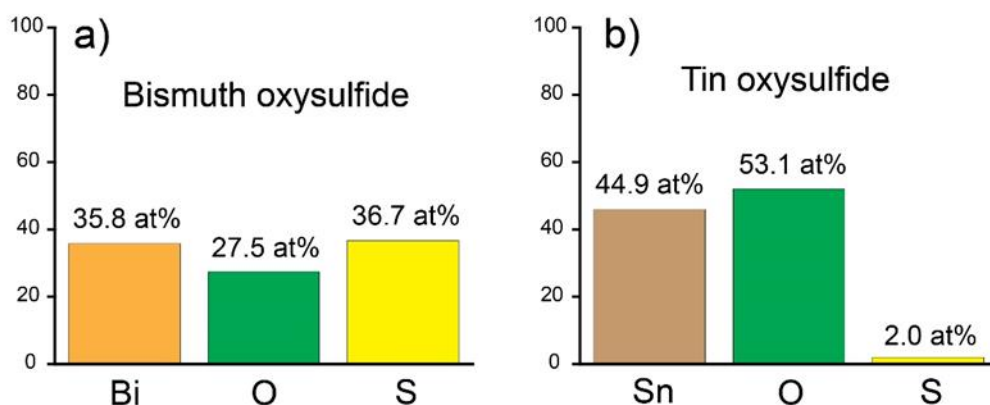




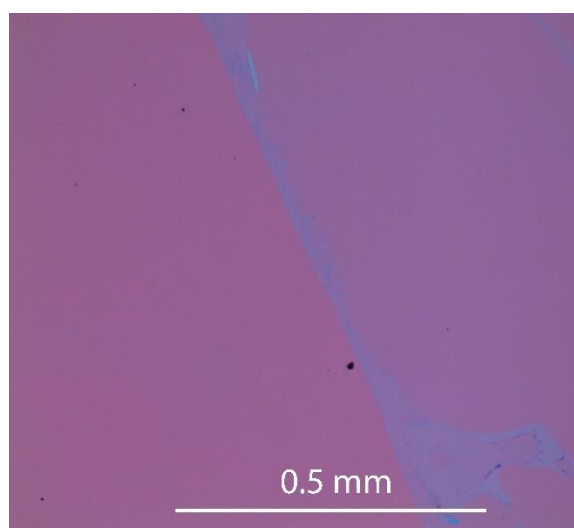
**Figure S15.** Transfer characteristic  $I_{ds}$ - $V_{gs}$  of 2D  $\text{In}_2\text{O}_3$  based device with  $V_{ds}$  being varied from 0 to 1 V.



**Figure S16.** The photocurrent response of 2D  $\text{In}_2\text{O}_3$  based photodetector under 285 nm and 365 nm UV exposure at  $P = 2 \text{ mW cm}^{-2}$  and  $V_{ds} = 0.5 \text{ V}$ . The device is more sensitive to shorter wavelengths and features a persistent photocurrent for several hours.



**Figure S17.** Elemental composition derived from XPS results of (a) 2D bismuth oxysulfide and (b) 2D tin oxysulfide.



**Figure S18.** Optical image of an as-grown 2D In<sub>2</sub>O<sub>3</sub> nanosheet on silicon substrate with a folded region being observed at the edge of the 2D sheet.

**Table S1.** Screening the reaction parameters for the transformation of 2D In<sub>2</sub>O<sub>3</sub> into 2D In<sub>2</sub>O<sub>3-x</sub>S<sub>x</sub> (calculated from XPS data).

Sample	Temp. (°C)	Time (h)	S content (At%)	x
In <sub>2</sub> O <sub>3</sub> as-synthesized *	-	-	0	0
In <sub>2</sub> O <sub>3</sub> blank sample **	150	5	0	0
In <sub>2</sub> O <sub>3-x</sub> S <sub>x</sub>	RT	5	<1	-
In <sub>2</sub> O <sub>3-x</sub> S <sub>x</sub>	80	5	4.2	0.21
In <sub>2</sub> O <sub>3-x</sub> S <sub>x</sub>	120	5	4.6	0.23
In <sub>2</sub> O <sub>3-x</sub> S <sub>x</sub> ***	150	5	8.3±0.5	0.41±0.02
In <sub>2</sub> O <sub>3-x</sub> S <sub>x</sub>	200	5	7.8	0.39
In <sub>2</sub> O <sub>3-x</sub> S <sub>x</sub>	150	1	<1	-
In <sub>2</sub> O <sub>3-x</sub> S <sub>x</sub>	150	2	1.8	0.09
In <sub>2</sub> O <sub>3-x</sub> S <sub>x</sub>	150	8	6.4	0.32

\* 2D In<sub>2</sub>O<sub>3</sub> was analysed after squeeze printing process

\*\* 2D In<sub>2</sub>O<sub>3</sub> was treated at optimized conditions, but without sulfur sources

\*\*\* Experiments have been repeated 4 times for standard error calculation

Note: x is calculated from the relation  $x=2/(\text{In:S ratio})$  with In:S ratio being obtained from XPS data of 2D In<sub>2</sub>O<sub>3-x</sub>S<sub>x</sub>. Sulfur content is calculated from the relation %S=100x/5

**Table S2.** Comparison of FETs based on 2D In<sub>2</sub>O<sub>3-x</sub>S<sub>x</sub> nanosheets with some reported 2D oxides and chalcogenides over the past few years.

Materials	Thickness (nm)	Method	Mobility (cm <sup>2</sup> V <sup>-1</sup> s <sup>-1</sup> )	$I_{on}/I_{off}$	Ref.
In <sub>2</sub> O <sub>3-x</sub> S <sub>x</sub>	2.4	vdW Exfoliation and wet chemical process	44 * 20.4±6.3 **	0.6×10 <sup>2</sup>	This work
In <sub>2</sub> O <sub>3</sub>	2.0	vdW Exfoliation	5.5±1.5 **	4×10 <sup>2</sup>	This work
In <sub>2</sub> O <sub>3</sub>	1.5	vdW Exfoliation	4	10 <sup>5</sup>	14
In <sub>2</sub> O <sub>3</sub>	12-20	Inkjet-printing	3.98	10 <sup>8</sup>	15
SnO	0.4	vdW Exfoliation	0.7	20	16
SnO <sub>2</sub>	50	Solution process and annealing	0.23	10 <sup>6</sup>	17
ZnO	5	ALD	2.8	10 <sup>5</sup>	18
IGZO	10	Solution process and annealing	9.1	10 <sup>6</sup>	19
In <sub>2</sub> S <sub>3</sub>	3.7	vdW Exfoliation and CVD	56	10 <sup>4</sup>	1
InSe	12	Mechanical exfoliation	0.1	10 <sup>4</sup>	20
InSe	41	Solvent exfoliation	19	10 <sup>6</sup>	21
α-Ga <sub>2</sub> S <sub>3</sub>	2	vdW Exfoliation and CVD	3.5	10 <sup>2</sup>	22
γ-Ga <sub>2</sub> S <sub>3</sub>	9	CVD	1.1	10 <sup>5</sup>	23
GaS	1.5	vdW Exfoliation and CVD	0.2	150	24
GaSe	1.1	Mechanical exfoliation	0.6	10 <sup>5</sup>	25
MoS <sub>2</sub>	3	CVD	12.24	10 <sup>6</sup>	26
MoS <sub>2</sub>	0.8	CVD	17	10 <sup>3</sup>	27
MoSe <sub>2</sub>	3-80	Mechanical exfoliation	50	10 <sup>6</sup>	28
MoTe <sub>2</sub>	1.6	Mechanical exfoliation	10	10 <sup>6</sup>	29
WS <sub>2</sub>	0.7	CVD	0.91	10 <sup>6</sup>	30

* Highest value
** Average value with standard error

**Table S3.** Comparison of 2D  $\text{In}_2\text{O}_{3-x}\text{S}_x$  nanosheets with other recently reported 2D nanosheets and commercial materials for photodetector performance. NM: not mentioned.

Materials	Wavelength (nm)	Thickness (nm)	R ( $\text{A W}^{-1}$ )	D* (Jones)	EQE (%)	Response time (ms)	Ref.
$\text{In}_2\text{O}_{3-x}\text{S}_x$	285	2.4	$3.4 \times 10^3$	$2.18 \times 10^{13}$	$1.47 \times 10^6$	$t_{\text{rise}} = 42 \times 10^3$ $t_{\text{fall}} = 87 \times 10^3$	This work
SnO-In <sub>2</sub> O <sub>3</sub>	280	5.5	$10^3$	$5 \times 10^9$	NM	$\leq 1$	5
Bi <sub>2</sub> O <sub>3</sub>	365	0.75	$4 \times 10^2$	$1.1 \times 10^{13}$	NM	0.07	31
Graphene- $\beta$ -Ga <sub>2</sub> O <sub>3</sub>	254	2	1.48	$2.24 \times 10^{12}$	$7.27 \times 10^2$	$3.1 \times 10^5$	32
$\gamma$ -Ga <sub>2</sub> S <sub>3</sub>	350	9	61.3	$1.52 \times 10^{10}$	$2.17 \times 10^4$	10-15	23
GaS	254	2.3	4.2	$10^{13}$	$2 \times 10^3$	$< 30$	33
WS <sub>2</sub>	365	25.2	53.3	$1.22 \times 10^{11}$	NM	NM	34
MoS <sub>2</sub>	405	1.3	$6.3 \times 10^{-5}$	$4.2 \times 10^8$	NM	20	35
InSe	254	30	$5.6 \times 10^4$	$2 \times 10^{13}$	NM	5	36
SnSe	370	9	5.5	$6 \times 10^{10}$	$1.83 \times 10^3$	NM	37
In <sub>2</sub> Se <sub>3</sub>	300	3.9	$3.9 \times 10^2$	$2.26 \times 10^{12}$	$16.3 \times 10^4$	18	38
Commercial Si	400-1100	NM	0.5	$3 \times 10^{12}$	NM	$10^{-6}$	39
Commercial GaP	150-550	NM	0.1	$2 \times 10^{13}$	NM	$1.1 \times 10^{-4}$	39

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