

Supplementary Information

Three-dimensional $\text{CoS}_{1.097}/\text{Al}_2\text{O}_3$ composites assembled with ultra-thin nanosheets for high-performance NO_x gas sensing at room temperature

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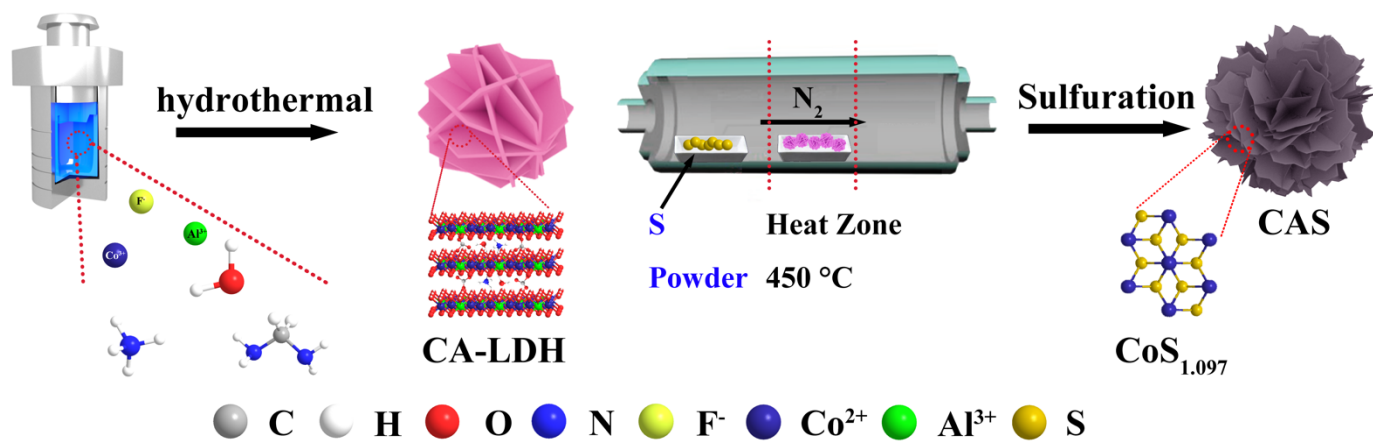
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1. **Scheme 1** Schematic diagram of preparation of the 3D flower-like $\text{CoS}_{1.097}/\text{Al}_2\text{O}_3$ composites.
2. **Fig. S1** Raman spectra of CAS-6 MFs.
3. **Fig. S2** AFM images and height of CA-LDH MFs.
4. **Fig. S3** AFM images and height of CAS-6 MFs.
5. **Fig. S4** SEM images (a), elemental mapping images (b–e), EDS analysis (f) of CAS-6.
6. **Fig. S5** TEM images of (a) CA, (b) CAS-6 and (c-d) HRTEM of CAS-6.
7. **Fig. S6** Dynamic response-recovery curve of (a) CA, (b) CAS-3 and (c) CAS-9 sensors.
8. **Fig. S7** The recovery times (a) and the response times (b) of CA, CAS-3, CAS-6 and CAS-9 sensors.
9. **Table S1** Comparison of the gas sensing characteristics of sulfide based materials reported in literature.
10. **Table S2** The binding energy of the different oxygen species in CA, CAS-6 before and after NO_x adsorption.
11. **Table S3** The contents of the different oxygen species in CA, CAS-6 before and after NO_x adsorption.
12. **Table S4** Responses, response times and recovery times of CA, CAS-3, CAS-6 and CAS-9 sensors.
13. **Table S5** The quantitative results of the of the CAS-6 on successive exposure (12 cycles) to 100 ppm NO_x .
14. **Table S6** The quantitative results of the stability for the CAS-6 to 100 ppm NO_x for 9 weeks.
15. **Table S7** The contents and binding energy of different sulfur species in CAS-6 before and after NO_x adsorption.



Scheme 1 Schematic diagram of preparation of the 3D flower-like $\text{CoS}_{1.097}/\text{Al}_2\text{O}_3$ composites.

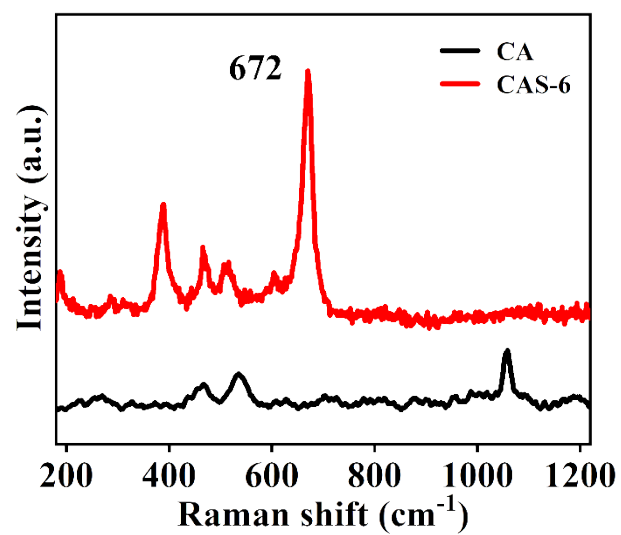


Fig. S1. Raman spectra of CAS-6 MFs.

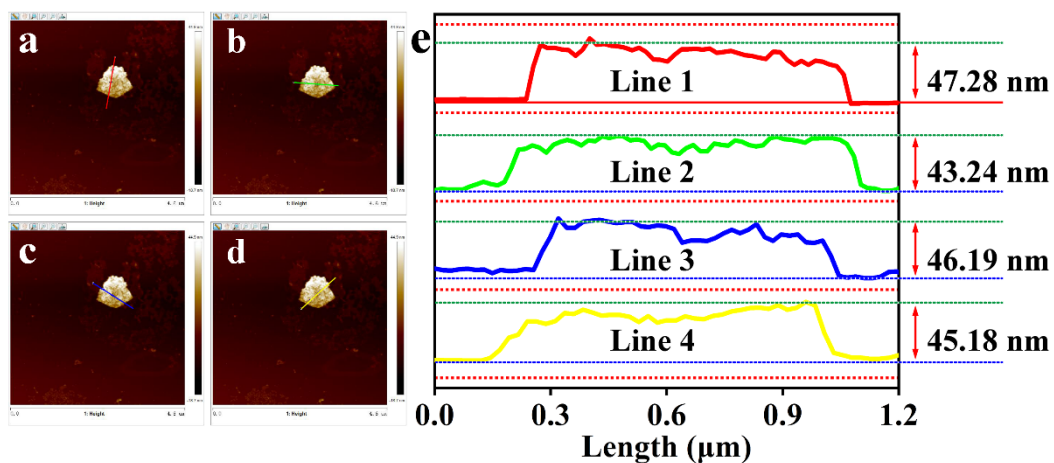


Fig. S2. AFM images and height of CA-LDH MFs.

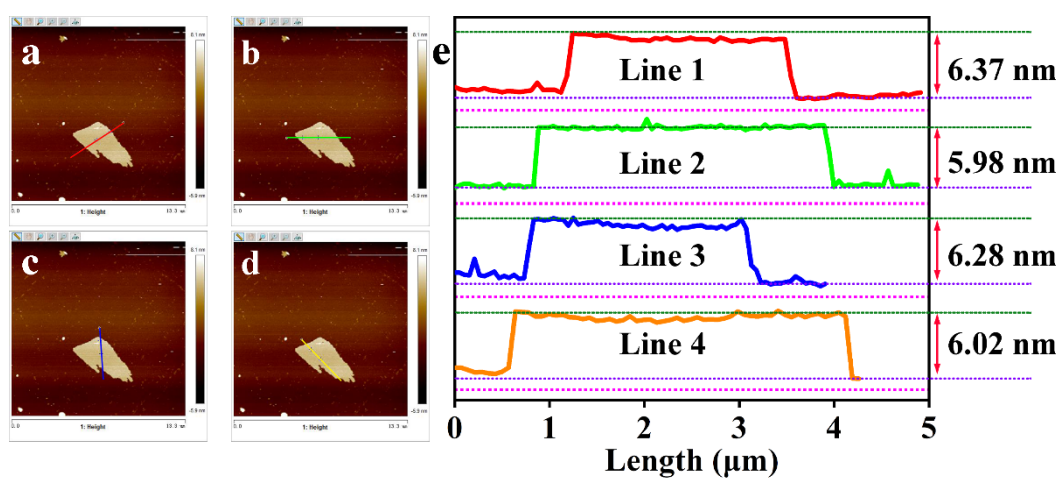


Fig. S3. AFM images and height of CAS-6 MFs.

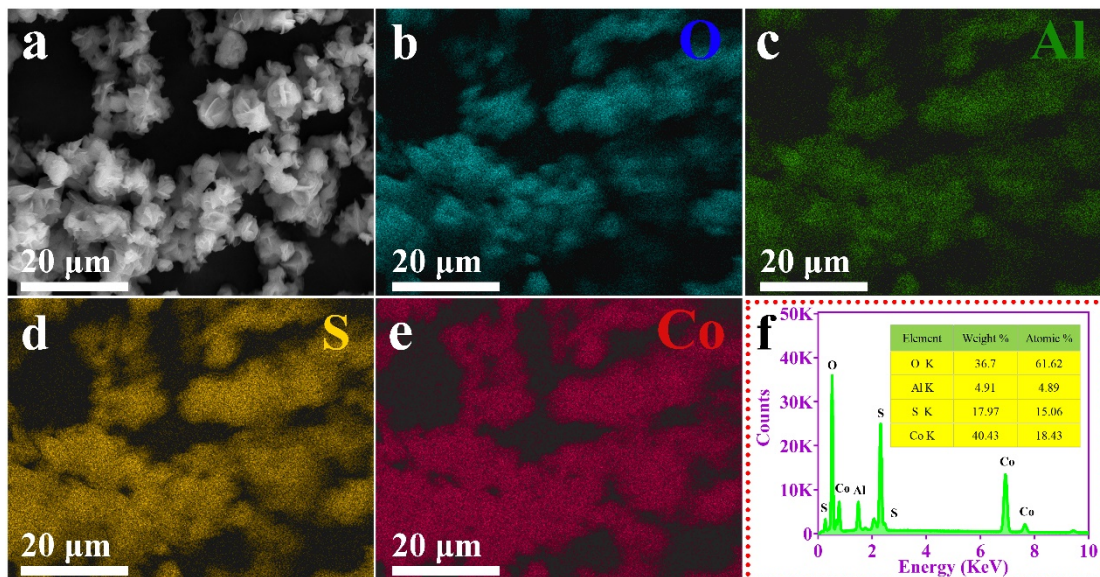


Fig. S4. SEM images (a), elemental mapping images (b–e), EDS analysis (f) of CAS-6.

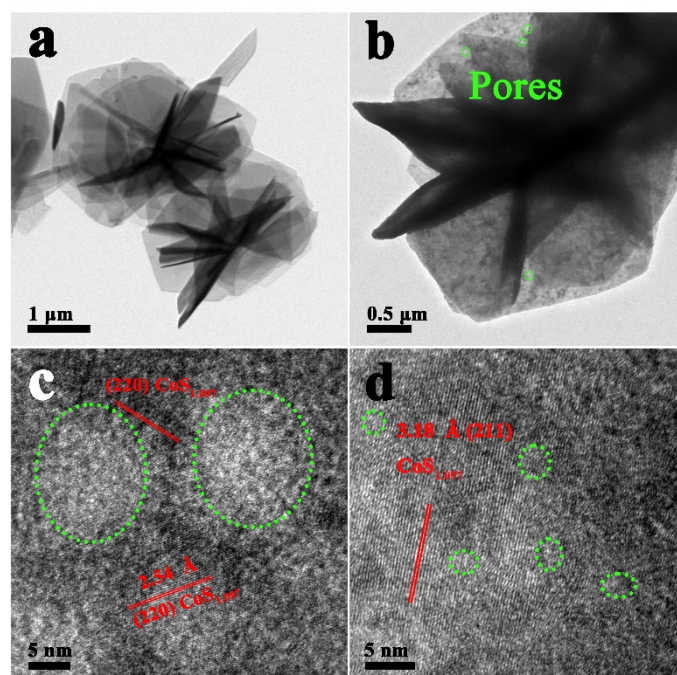


Fig. S5. TEM images of (a) CA, (b) CAS-6 and (c-d) HRTEM of CAS-6.

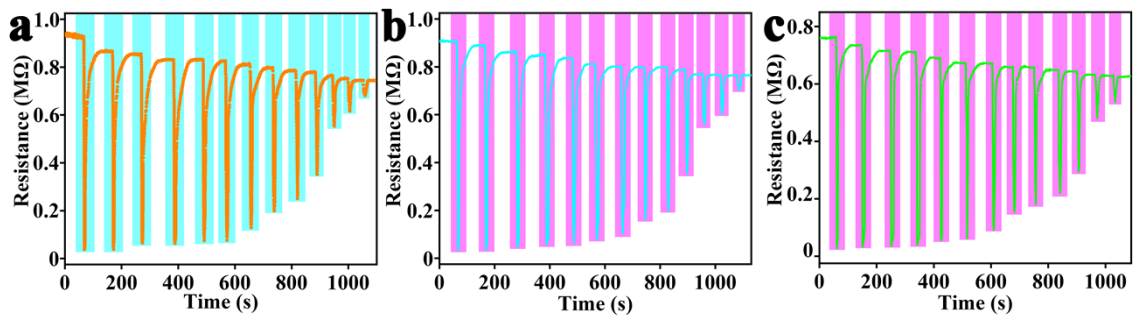


Fig. S6. Dynamic response-recovery curve of (a) CA, (b) CAS-3 and (c) CAS-9 sensors.

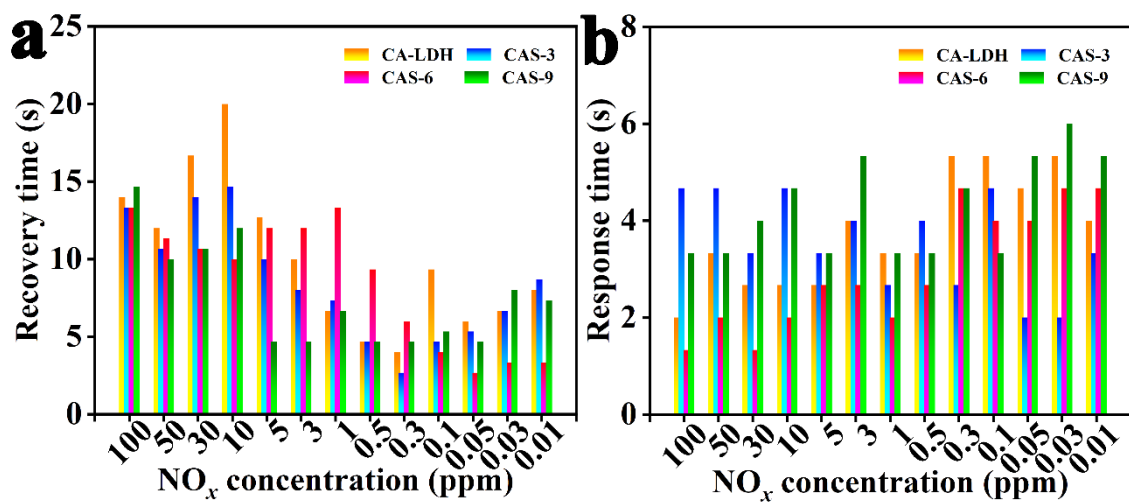


Fig. S7. The recovery times (a) and the response times (b) of CA, CAS-3, CAS-6 and CAS-9 sensors.

Table S1. Comparison of the gas sensing characteristics of Sulfide based materials reported in literature.

Sensor material	Operation temperature	Gas	Response/ concentration	Response/ recovery time (s)	limit of detection	References
p-NiS/n-In ₂ O ₃	300°C	ethanol	10.3/ 100 ppm	8 s/ 20 s	5 ppm	12
MoS ₂ -Bi ₂ O ₃ -Bi ₂ S ₃ NMs	RT	NO _x	10.7/ 50 ppm	1 s/--	50 ppb	16
MoS ₂ @WS ₂	RT	NO ₂	26.12/ 50 ppm	1.6 s/ 27.7 s	10 ppb	17
Mn-SnS	RT	acetone	1.107/ 500 ppb	3 s/ 4 s	500 ppb	18
MoS ₂ @SnO ₂ heterostructure	RT	NO ₂	34.67/ 100 ppm	2.2 s/ 10.54 s	10 ppb	51
n-MoS ₂ / p-CuO	RT	NH ₃	47%/ 500 ppm	17 s/26 s	5 ppm	S1
Au-decorated WS ₂	RT	CO	1.48/ 50 ppm	--/--	0.2 ppm	S2
CdS QDs /Co ₃ O ₄	RT	H ₂ S	12.7/ 100 ppm	0.6 s/ 1.0s	1-5 ppm	S3
CNFs/CoS ₂ /MoS ₂	RT	NO	32%/ 60 ppm	--/--	1 ppm	S4
Co ₉ S ₈ sensor	160°C	ethanol	7.326/ 100 ppm	1 s/ 15 s	--	S5
CAS-6	RT	NO_x	30.05/ 100 ppm	1.3s/ 13.3s	10 ppb	This work

Table S2. The binding energy position of the different oxygen species in CA and CAS-6 before and after NO_x adsorption.

Oxygen species	CA	CAS-6	CAS-NO_x
Lattice oxygens	531.9	531.8	531.8
Oxygen defects	532.7	532.5	532.6
Chemisorbed oxygens	534.2	533.4	533.5

Table S3. The contents of the different oxygen species in CA and CAS-6 before and after NO_x adsorption.

Oxygen species	CA	CAS-6	CAS-NO_x
Lattice oxygens	54.7%	44.4%	46.4%
Oxygen defects	26.3%	31.9%	25.7%
Chemisorbed oxygens	19.0%	23.7%	27.9%

Table S4. Response, response time and recovery time of CA, CAS-3, CAS-6 and CAS-9 sensors.

Sensors	CA			CAS-3			CAS-6			CAS-9		
	NO_x (ppm)	R	Ts	Tr	R	Ts	Tr	R	Ts	Tr	R	Ts
100	25.04	2.00	14.00	27.21	4.67	13.33	30.05	1.33	13.33	27.22	3.33	14.67
50	22.53	3.33	12.00	25.71	4.67	10.67	27.25	2.00	11.33	21.85	3.33	10.00
30	13.15	2.67	16.67	17.98	3.33	14.00	22.54	1.33	10.67	19.57	4.00	10.67
10	13.09	2.67	20.00	15.03	4.67	14.67	18.81	2.00	10.00	17.69	4.67	12.00
5	11.09	2.67	12.67	13.13	3.33	10.00	15.46	2.67	12.00	12.46	3.33	4.67
3	10.75	4.00	10.00	10.49	4.00	8.00	11.32	2.67	12.00	10.63	5.33	4.67
1	6.28	3.33	6.67	7.72	2.67	7.33	8.28	2.00	13.33	7.25	3.33	6.67
0.5	4.01	3.33	4.67	4.91	4.00	4.67	6.83	2.67	9.33	4.26	3.33	4.67
0.3	3.12	5.33	4.00	4.03	2.67	2.67	5.13	4.67	6.00	3.59	4.67	4.67
0.1	2.21	5.33	9.33	2.22	4.67	4.67	3.80	4.00	4.00	2.95	3.33	5.33
0.05	1.38	4.67	6.00	1.38	2.00	5.33	2.27	4.00	2.67	2.19	5.33	4.67
0.03	1.22	5.33	6.67	1.27	2.00	6.67	1.46	4.67	3.33	1.31	6.00	8.00
0.01	1.09	4.00	8.00	1.08	3.33	8.67	1.22	4.67	3.33	1.17	5.33	7.33

***R:** Response **Ts:** Response time **Tr:** Recovery time

Table S5. The quantitative results of the of the CAS-6 on successive exposure (12 cycles) to 100 ppm NO_x.

Cycles	1	2	3	4	5	6	7	8	9	10	11	12
Gas response (R ₀ /R _g)	30.06	30.01	29.46	29.98	29.89	28.87	29.46	28.32	28.56	28.12	28.52	28.41

*RSD value = 2.6% (RSD: Relative Standard Deviation).

Table S6. The quantitative results of the stability for the CAS-6 to 100 ppm NO_x for 9 weeks.

Weeks	1	2	3	4	5	6	7	8	9
Gas response (R ₀ /R _g)	30.02	30.05	30.00	29.83	29.95	29.76	30.02	30.01	29.99

Table S7. The contents and binding energy positions of different sulfur species in CAS-6 before and after NO_x adsorption.

Sulfur species	CAS-6		CAS-NO_x	
	Position (eV)	Content (%)	Position (eV)	Content (%)
S-Co	163.0	14.4	161.8	6.4
S2p_{3/2}	169.0	49.2	168.9	34.1
S2p_{1/2}	170.1	28.3	170.1	37.6
Satellite peak	176.1	8.1	174.1	21.9

References

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