

**Fabrication of three-dimensional flower-ball heterostructures
MoS₂/LDHs based on interface coupling strategy and their
application in high-performance NO_x gas sensing**

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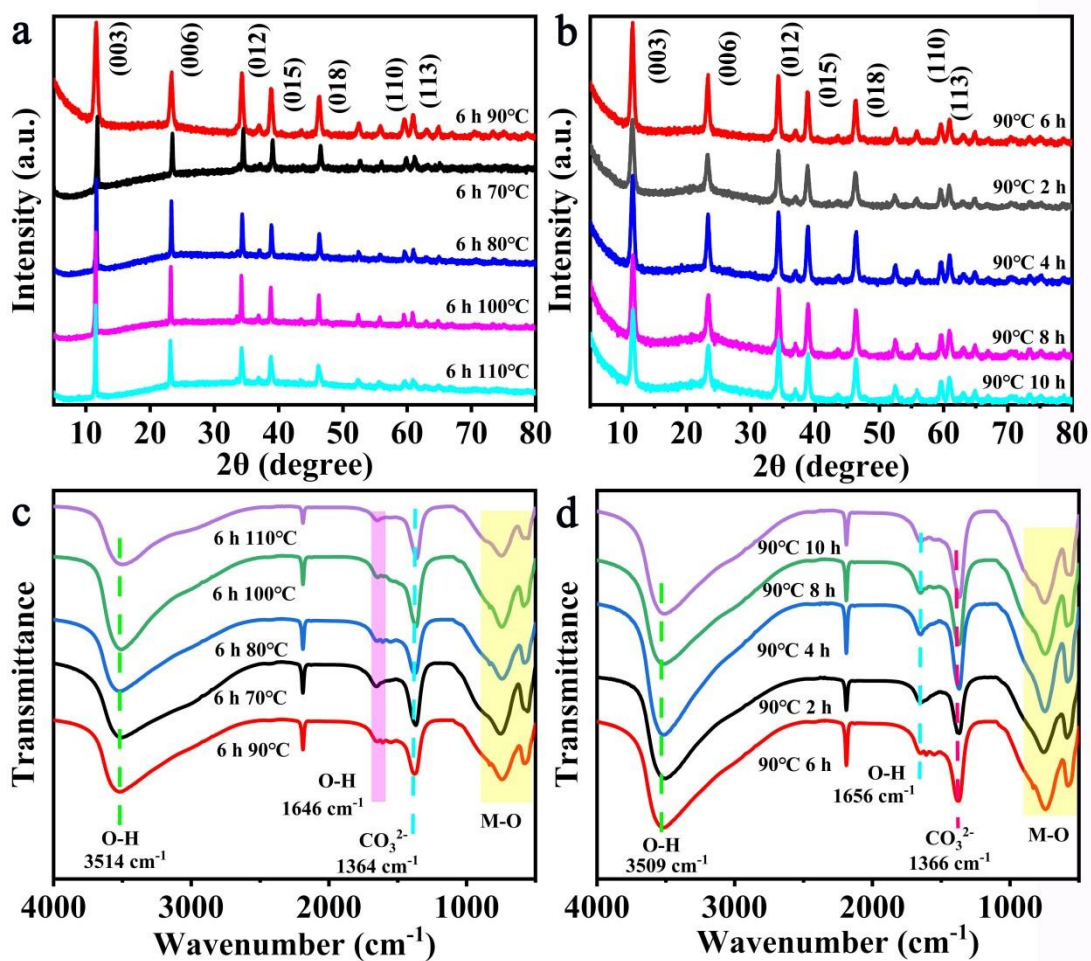


Fig. S1 XRD spectra and FT-IR spectrum of CoAl-LDHs for (a,c) different temperatures and (b,d) different times.[1,2]

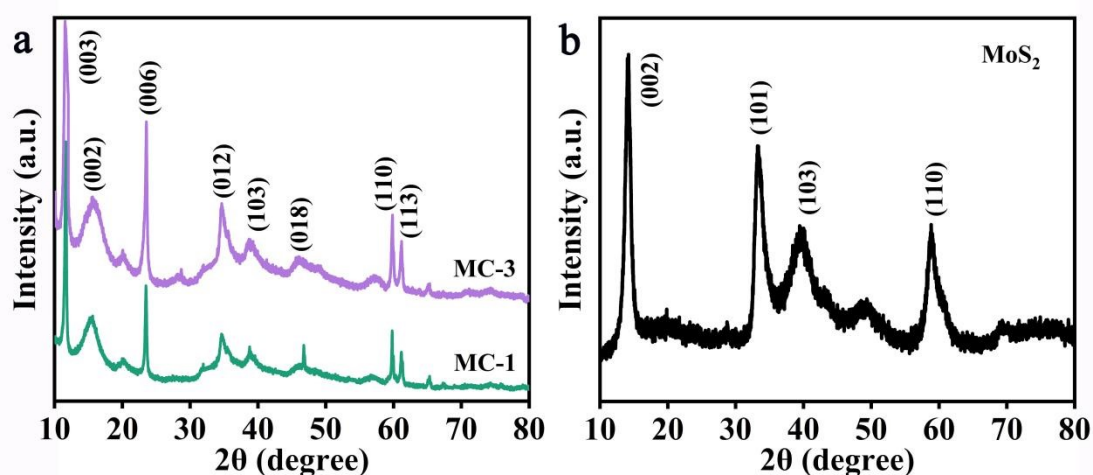


Fig. S2 XRD spectra for (a) MC-1, MC-3 and (b) MoS₂. [3]

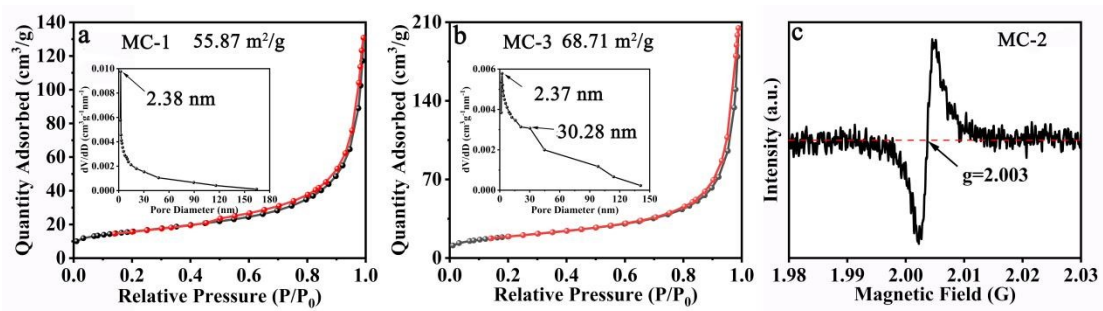


Fig. S3 (a, b) N₂ adsorption-desorption curves and pore size distributions for MC-1 and MC-3; (c) EPR spectrum for CM-2.

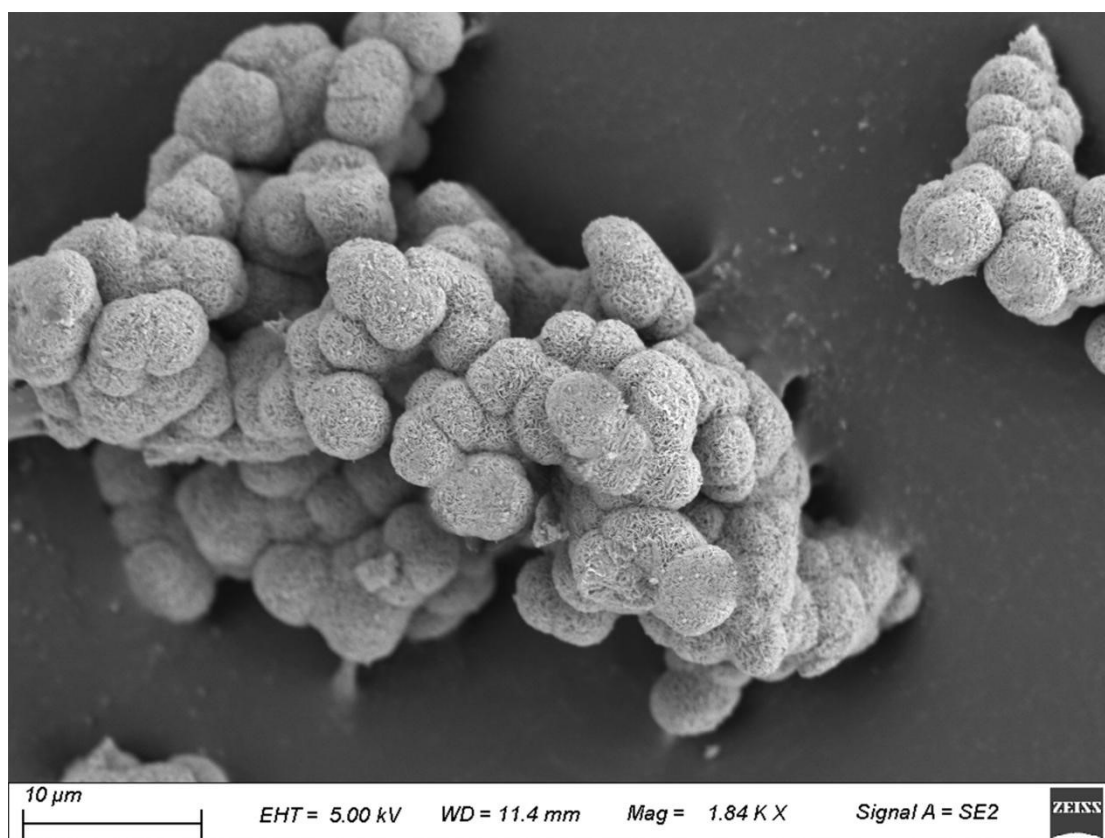


Fig. S4 SEM images of MoS₂.

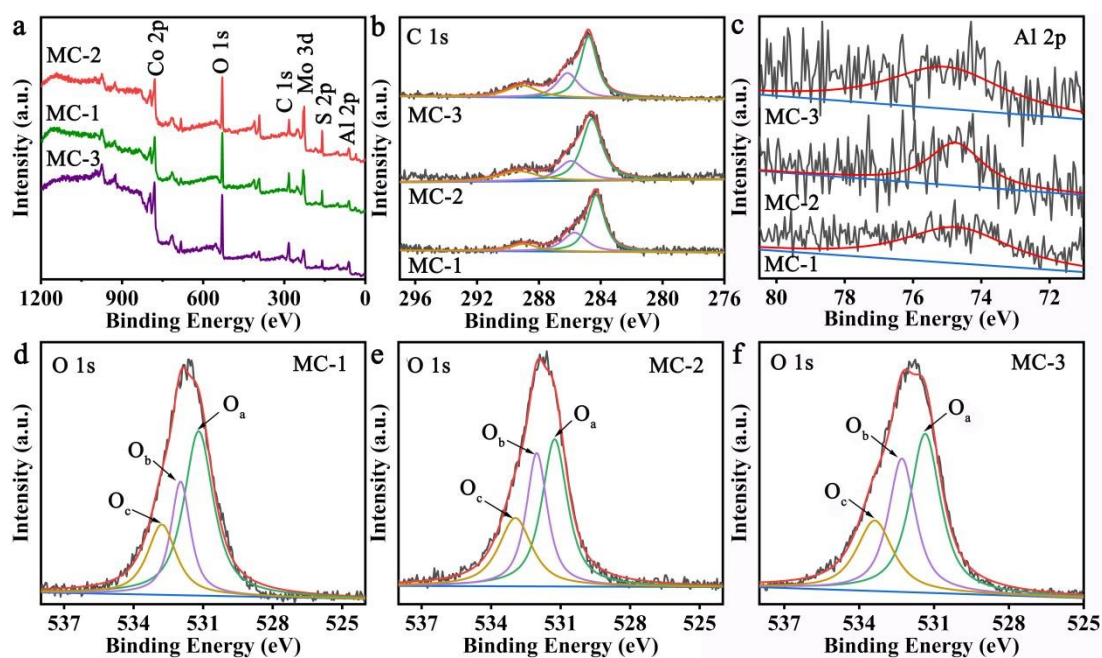


Fig. S5 XPS analysis of MC-1, MC-2, MC-3 (a) full spectrum analysis; (b) C 1s analysis; (c) Al 2p analysis; (d, e, f) O 1s analysis.[4]

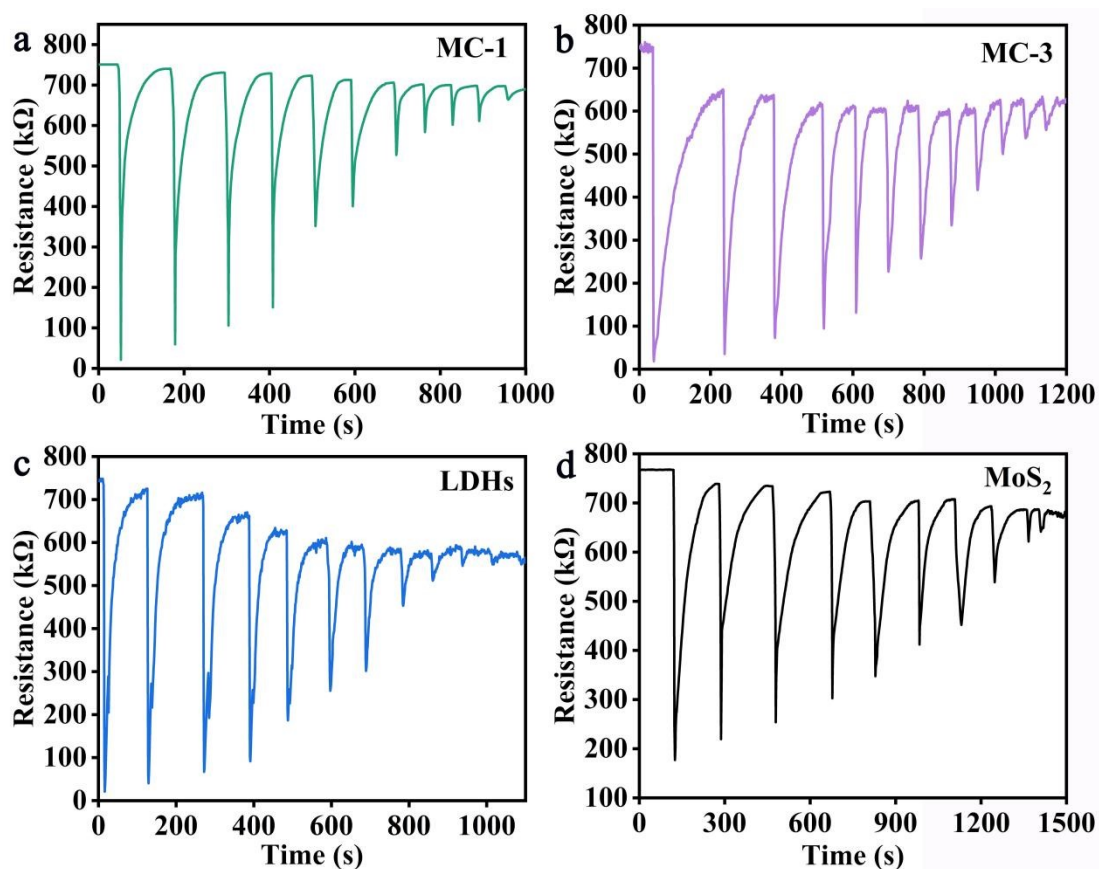


Fig. S6 Dynamic response-recovery time curves for (a) MC-1, (b) MC-3, (c) LDHs, and (d) MoS₂.

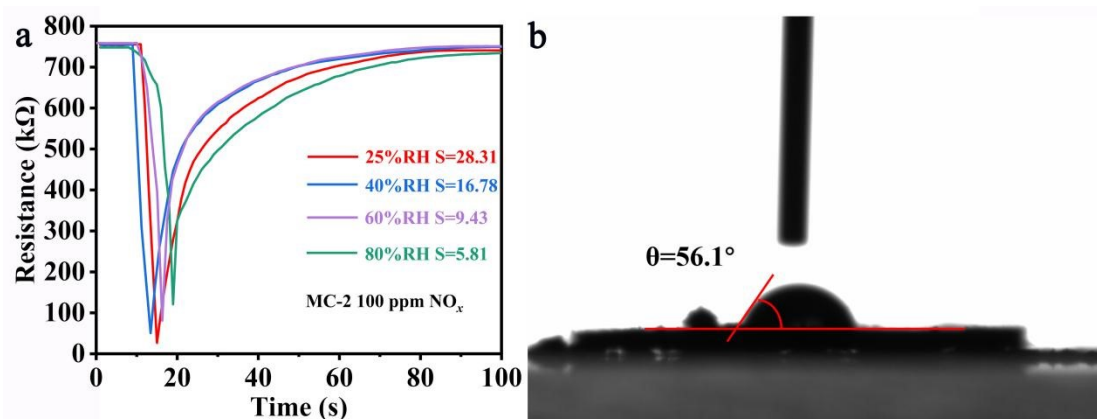


Fig. S7 MC-2 of (a) Gas sensing test at room temperature for 100 ppm NO_x at different humidity conditions; (b) Contact angle images.

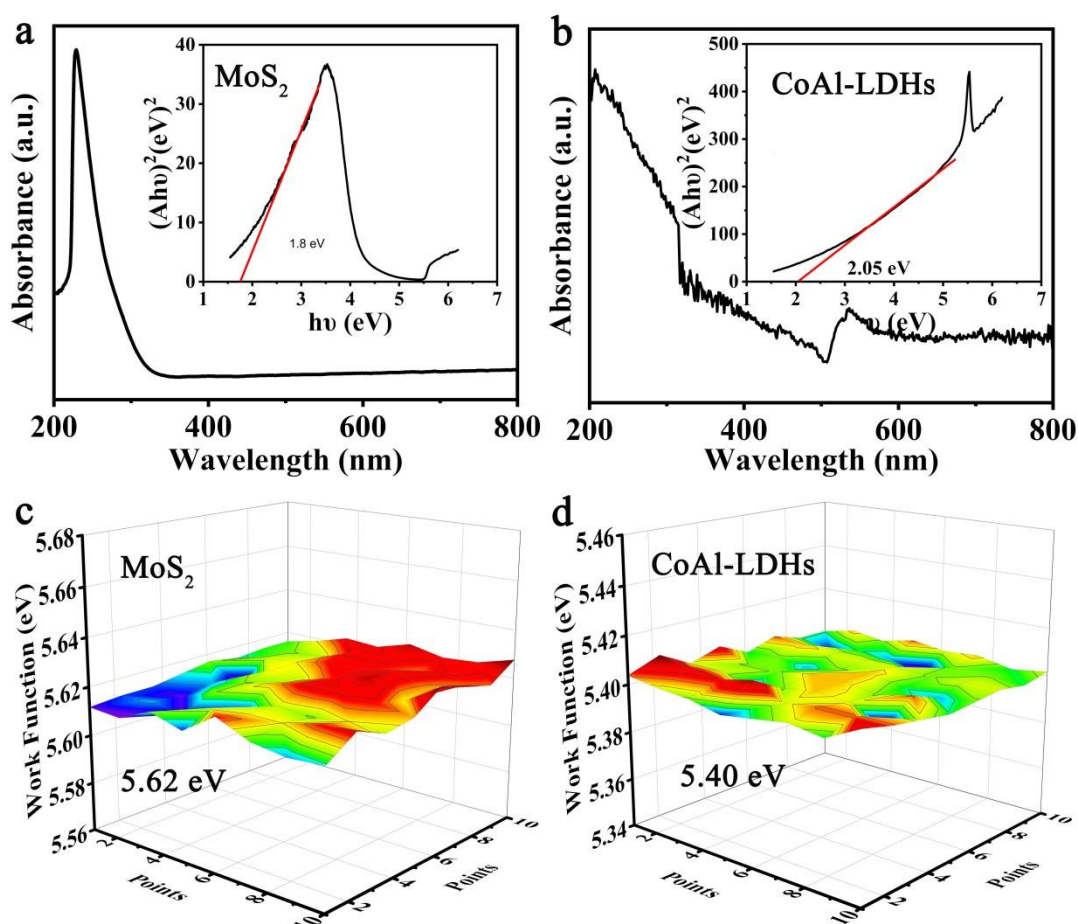


Fig. S8 (a, b) UV-Vis diffuse reflectance spectra of MoS₂ and LDHs; (c, d) Kelvin probe test results for MoS₂ and LDHs.

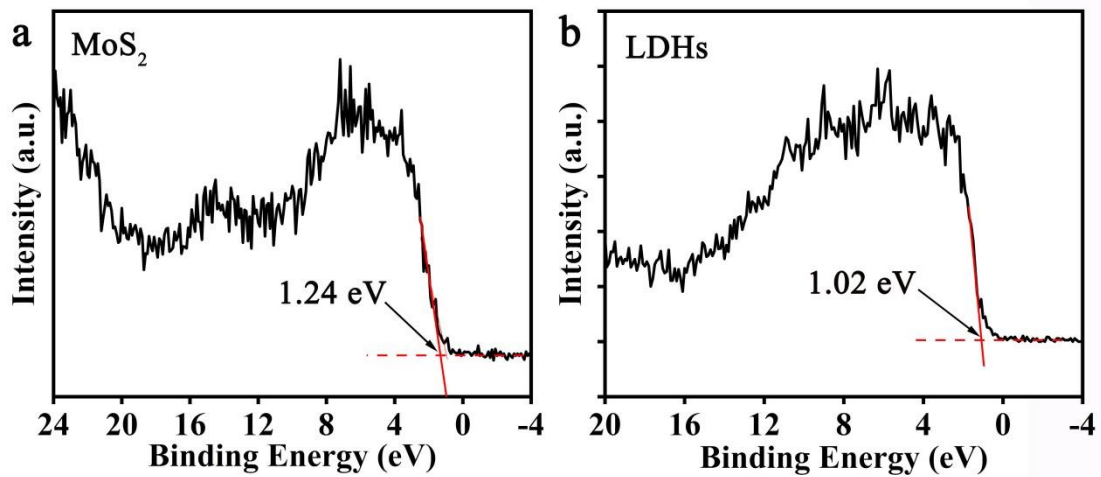


Fig. S9 XPS analysis of valence band (a)MoS₂; (b) LDHs.

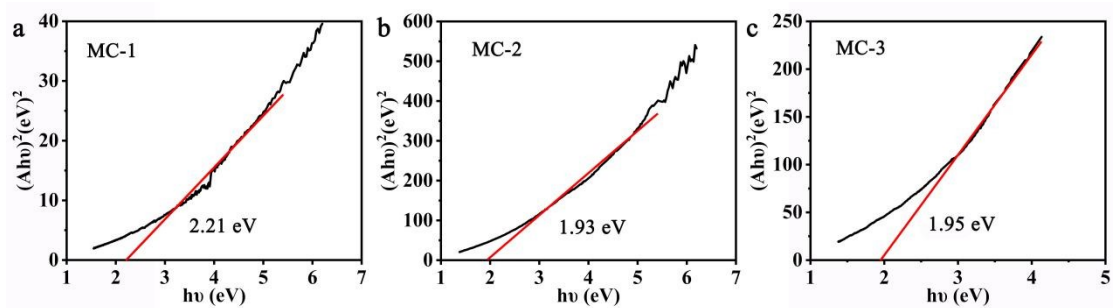


Fig. S10 Band gap diagram of (a) MC-1 , (b) MC-2 and (c) MC-3.

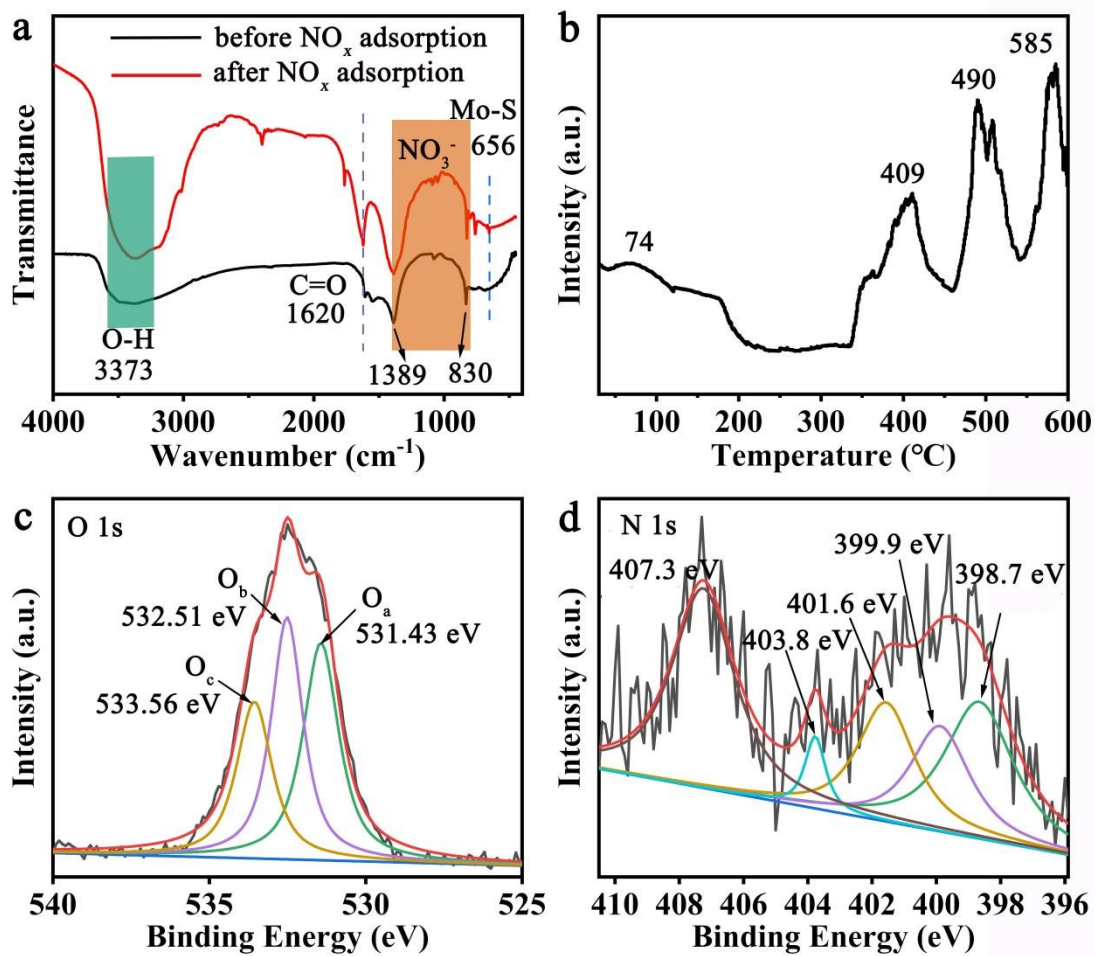


Fig. S11 MC-2 exposed to NO_x (a) FT-IR spectra; (b) TPD; (c) XPS analysis of O 1s; (d) XPS analysis of N 1s.[5]

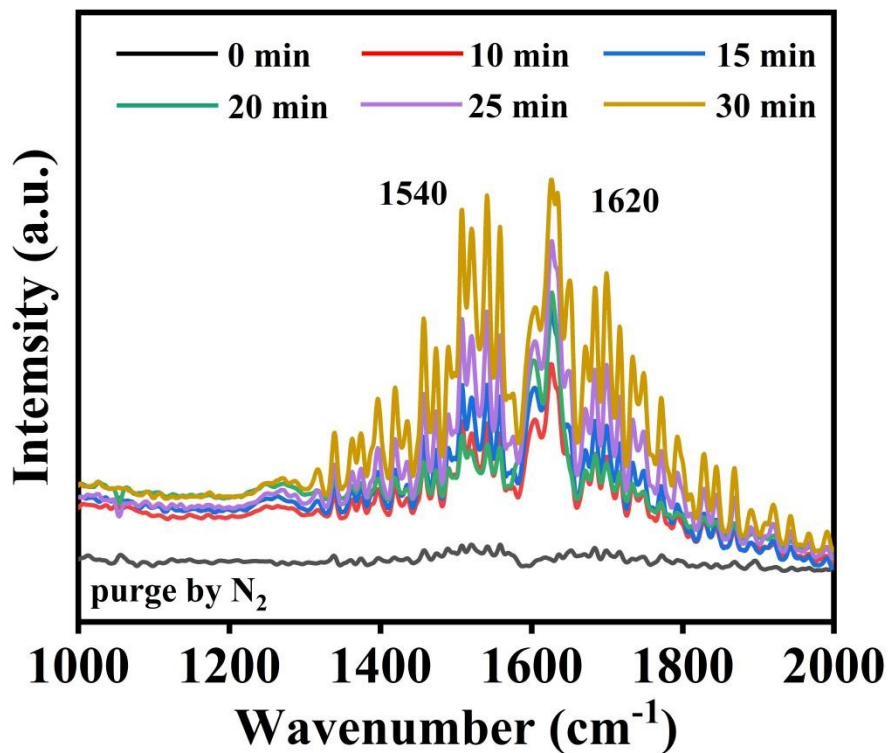


Fig.S12 DRIFT spectra of NO_x adsorption for the drift spectrum of MC-2.

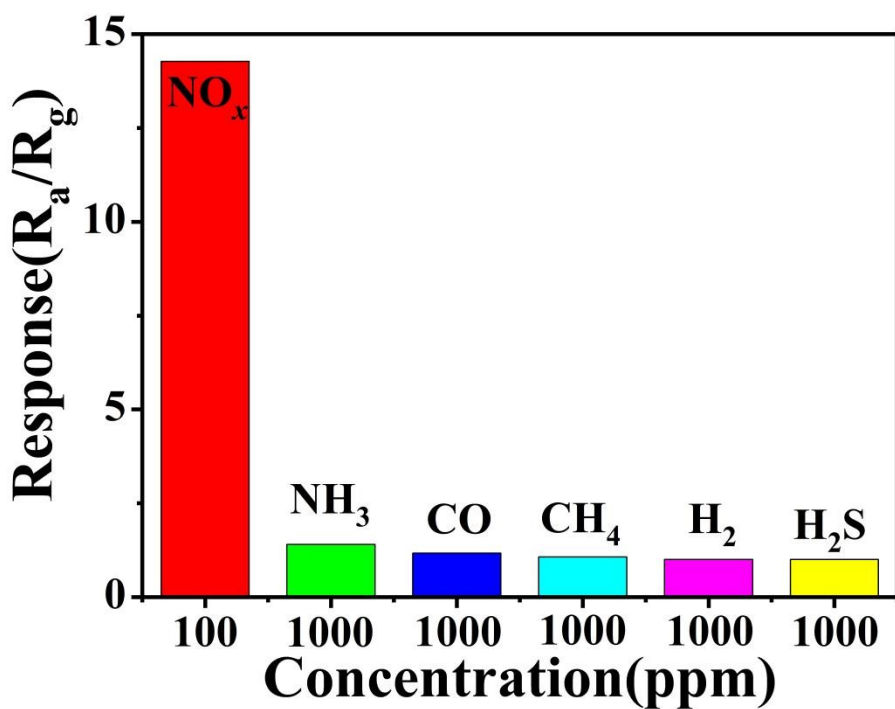


Fig. S13 Selectivity test of LDHs for different gases H₂, H₂S, CH₄, CO, and SO₂ at 1000 ppm, NO_x at 100 ppm.

Table S1 Comparison of gas sensing properties reported in previous literatures.

Sensor material	Gas	Operating temperature	Gas concentration (ppm)	Response (S)	Response time (s)	Recovery time (s)	Detection limit (ppm)	Ref.
Mixed MoS ₂ flakes	NO ₂	RT+UV	10	-21.78% ^①	6.09	146.49	-	[6]
MoS ₂ /ZnO	NO ₂	RT	0.5	20.9% ^①	-	-	-	[7]
CTAB-MoS ₂ /rGO	NO ₂	RT	17.5	45.5% ^①	-	-	0.05	[8]
MoS ₂ /RGO	NO ₂	160 °C	3	1.23 ^②	8	20	0.1	[9]
2D MoS ₂	NO ₂	100 °C	100	21.56% ^①	71	310	5	[10]
Thin-layered MoS ₂	NO ₂	RT	100	54% ^①	180	600	-	[11]
MoS ₂ /ZnO	NO ₂	RT	10	28% ^①	408	162	0.5	[12]
MoS ₂ hollow sphere	NO ₂	150 °C	100	40.3% ^①	79	225	0.5	[13]
MoS ₂ /Si ₃ N ₄ /Si	NO ₂	RT	5	59.4% ^①	24	41	1	[14]
MoS ₂ /LDHs	NO _x	RT	100	25.43 ^②	2.13	35.53	0.03	This work

①: $S = |R_a - R_g| / R_a \times 100\%$ or $S = |R_g - R_a| / R_a \times 100\%$

②: $S = R_a / R_g$

Table S2 Sample specific surface area and pore size.

Samples	BET Surface Area (m²·g⁻¹)	Pore Diameter (nm)
MC-1	55.87	2.38
MC-2	74.11	2.35
MC-3	68.71	2.37
LDHs	15.63	2.35
MoS ₂	13.17	2.35

Table S3 Percentage of atomic mass of MC-2.

Element	Atomic Percents					
	Mo	S	Co	Al	C	O
Weight (%)	3.17	1.66	22.27	5.33	12.39	55.18
Atomic (%)	0.6	1.07	7.35	3.84	20.06	67.08

Table S4 XPS analysis of the percentage content of O_a, O_b and O_c.

O 1s	MC-1	MC-2	MC-3	After NO₂ adsorption of MC-2
O _a (%)	53.14	42.55	46.48	36.92
O _b (%)	24.57	31.92	29.67	36.61
O _c (%)	22.29	25.53	23.85	26.47

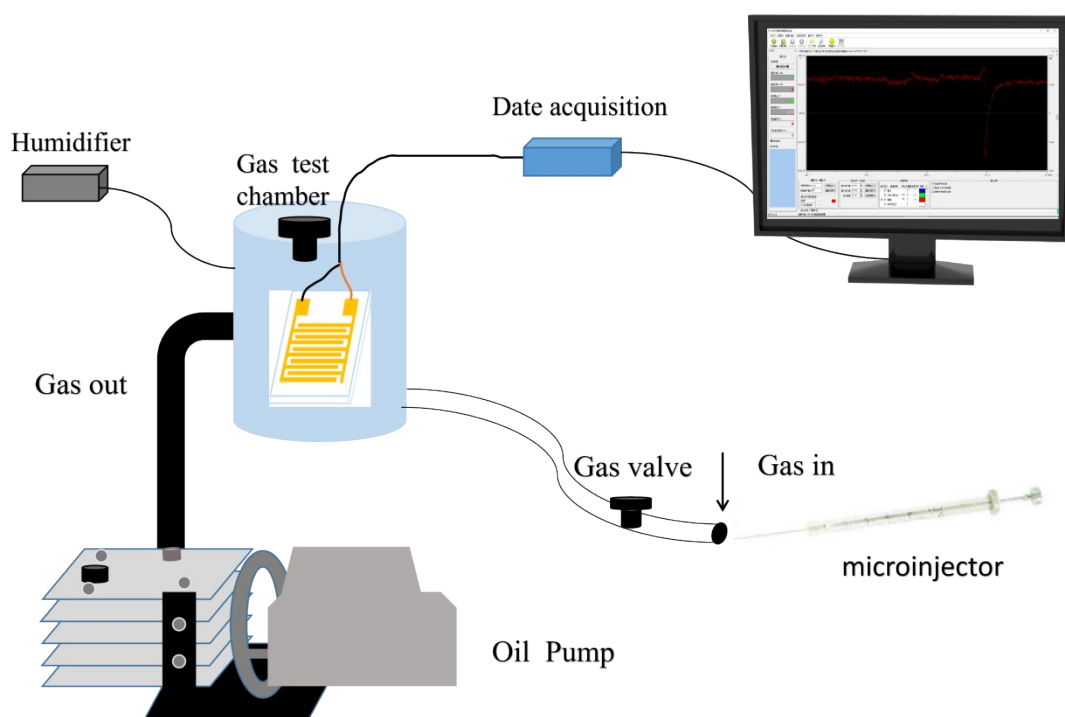
Table S5 Response, response time and recovery time of MC-2 and its comparative sensors at room temperature (RT = 25 °C, RH 25%)

Sample	MC-1			MC-2			MC-3			LDHs			MoS ₂			
	S	T _s /s	T _r /s	S	T _s /s	T _r /s	S	T _s /s	T _r /s	S	T _s /s	T _r /s	S	T _s /s	T _r /s	
NO_x																
(ppm)																
100	21.92	5.59	44.67	28.31	2.13	35.53	25.43	4.78	40.27	14.27	10.65	56.53	4.33	11.32	58.67	
50	15.65	7.13	40.53	21.41	2.67	33.40	19.59	6.42	38.40	8.29	13.33	50.13	3.41	14.94	53.87	
30	11.91	9.20	37.87	17.47	3.74	31.73	15.83	8.39	35.73	5.47	15.47	45.73	2.89	16.51	48.53	
10	7.47	11.13	35.73	11.18	4.85	27.66	9.39	10.22	32.53	4.12	17.94	37.34	2.61	19.80	42.93	
5	5.13	13.51	32.40	7.05	5.27	23.40	6.30	12.75	30.40	2.02	21.12	35.46	1.96	22.20	38.40	
3	3.47	17.20	29.33	4.70	8.53	20.80	4.11	15.06	28.80	1.73	25.23	31.45	1.71	26.55	35.20	
1	2.27	19.80	26.73	3.02	10.23	15.73	2.74	17.28	26.67	1.54	28.76	28.82	1.51	28.92	33.13	
0.5	1.75	22.87	22.40	2.05	14.57	13.53	1.92	21.59	22.40	1.26	32.34	24.53	1.19	33.60	29.33	
0.3	1.41	25.69	19.20	1.56	19.69	11.27	1.48	24.12	20.27	1.15	34.67	22.93	1.10	36.54	26.67	
0.1	1.24	31.56	16.27	1.34	25.48	10.40	1.27	30.66	16.53	1.13	35.75	17.07	1.06	38.56	25.07	
0.05	1.12	35.98	12.80	1.19	26.65	8.67	1.16	31.95	11.20	1.03	37.14	15.34				
0.03	1.05	36.16	10.13	1.14	28.22	6.27	1.09	35.67	9.60							

*S: Response T_s: Response time T_r: Recovery time

Table S6 Parameters obtained by fitting the experimental curve to the equivalent circuit.

Raw materials	$R1(\Omega)$	$C1(F)$	$R2(\Omega)$	$C2(F)$
MC-1	1.663×10^6	5.181×10^{-11}	1.670×10^7	1.383×10^{-11}
MC-2	1.585×10^5	1.316×10^{-11}	4.242×10^6	9.784×10^{-12}
MC-3	2.498×10^5	3.007×10^{-11}	2.198×10^7	2.584×10^{-11}
LDHs	2.333×10^6	6.087×10^{-11}	8.863×10^7	1.192×10^{-11}
MoS ₂	3.379×10^6	1.094×10^{-12}	1.056×10^8	1.051×10^{-11}



Scheme S1 Gas sensing device diagram.

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