Supporting Information

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Table S1. The gas sensing properties of ZAS-2 compared with the references based on LDHs.

Gas sensors	**Operation temperature (°C)**	**Gas (concentration)**	**Response (R_a/R_g)**	**Response/ recovery time**	**References**
HPTS/NiFe-LDHs	Room temperature	CO_2	--	--/--	[22]
Zn_2Al-LDHs	240°C	Ethanol (100 ppm)	12.5	--/--	[23]
PS@Co-LDHs	200°C	Dimethyl sulfide (125 ppm); Ethanol (4.3 ppm)	3; 2.48	--/-- --/--	[24]
Pt/ZnAl-LDHs	450°C	CH_4 (500 ppm);	5	--/--	[25]
NiAl-LDHs	Room temperature	O_3 (700 ppm)	1.84	8 s/74 s	[26]
ZnAl-LDHs	**Room temperature**	**NO_x (100 ppm)**	**28.6**	**2/29 s**	**Our work**

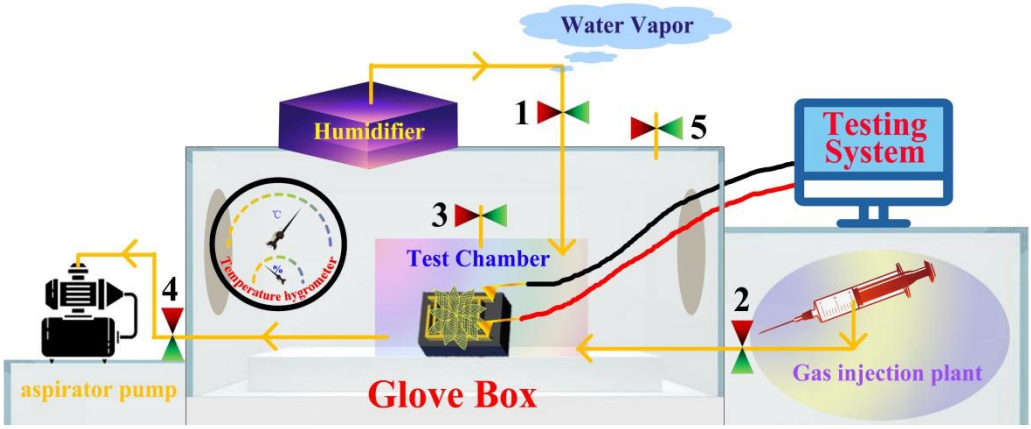

Fig. S1. A diagram of the delivery system for the gas sensing process.

Firstly, the prepared interdigital gold electrode sensor is installed in the test chamber, and the valves 3, 4 and 5 are kept open at the same time, so that the air can flow in the test chamber and the glove box for more than or equal to 3 minutes to remove the interference substances in the device.

After flushing, the valve 5 is still open and the valve 3, valve 4 are closed. At the same time, water vapor was injected into the box by valve 1. And the temperature and humidity change are controlled in the box. After reaching the target humidity, close valves 1, 3 and 5 at the same time.

Next, open valve 1 and inject a certain volume of nitrogen oxide gas to observe the resistance change. When the resistance is balanced, open valve 4 and valve 5. Use the air extraction pump to fill the chamber with air to restore the sensor resistance to its original state.

The above is a complete response recovery cycle. Repeat the previous work, inject NO_2 into the laboratory (control NO_2 gas concentration with micro syringe), complete

the second response recovery cycle, then measure the resistance change, and so on to get the required test results.

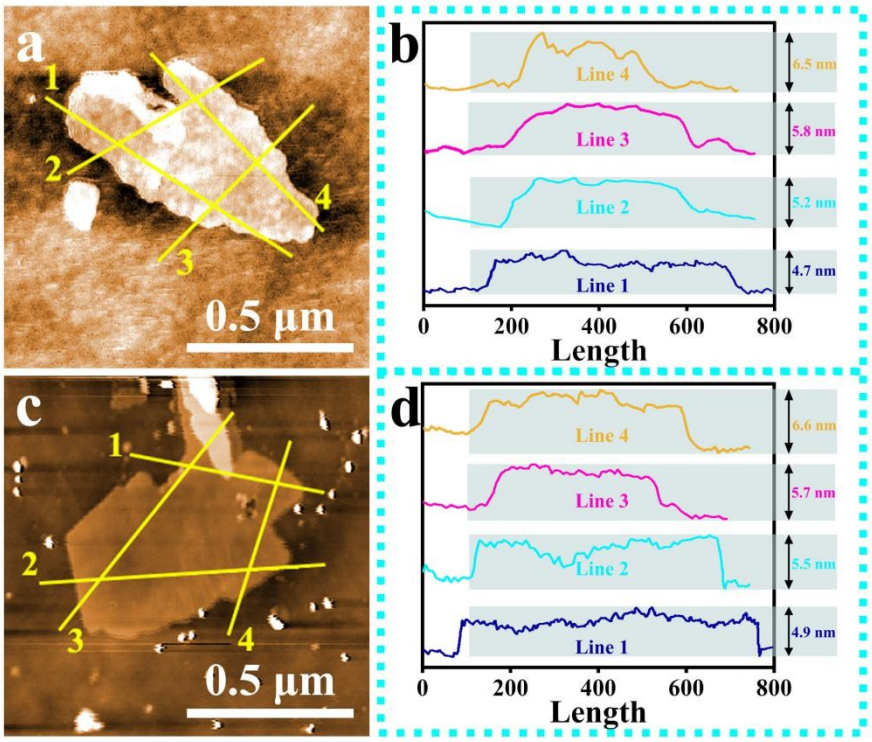

Fig. S2. AFM images and height profiles of ZAS-1 (a, b) and ZAS-3 (c, d).

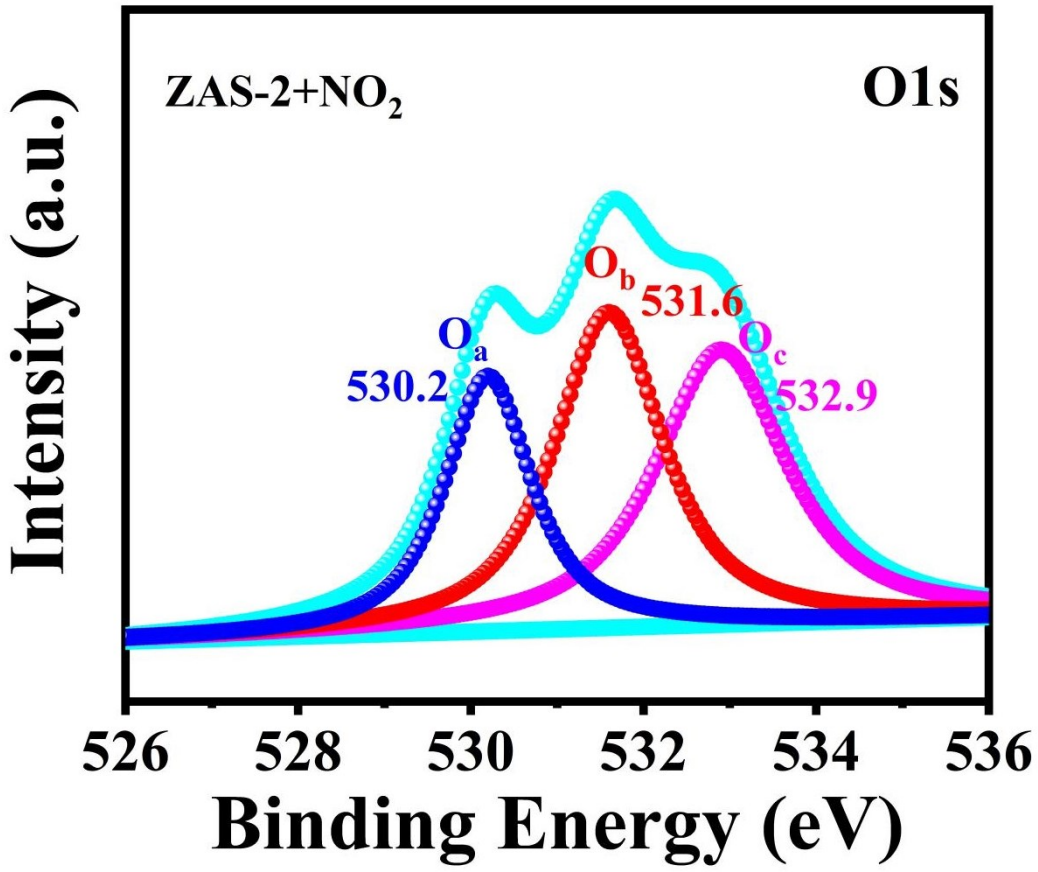

Fig. S3. O 1s high-resolution XPS spectra of ZAS-2+NO_2

Table S2. O1s peak position and peak area ratio (%) of the four samples.

Sample	ZAS-1			ZAS-2			ZAS-3			ZAS-2+NO_2		
Peak	O_a	O_b	O_c	O_a	O_b	O_c	O_a	O_b	O_c	O_a	O_b	O_c
Peak position (eV)	529.8	531.0	531.6	530.2	531.2	532.5	530.1	531.3	532.6	530.2	531.6	532.9
Peak area ratio (%)	24.7	**44.7**	30.6	18.0	**59.7**	22.3	21.2	**51.1**	27.7	24.1	**38.8**	37.1

⋆O_a: lattice oxygen; O_c: oxygen deficiency/vacancies structure; O_b: chemisorption oxygen.

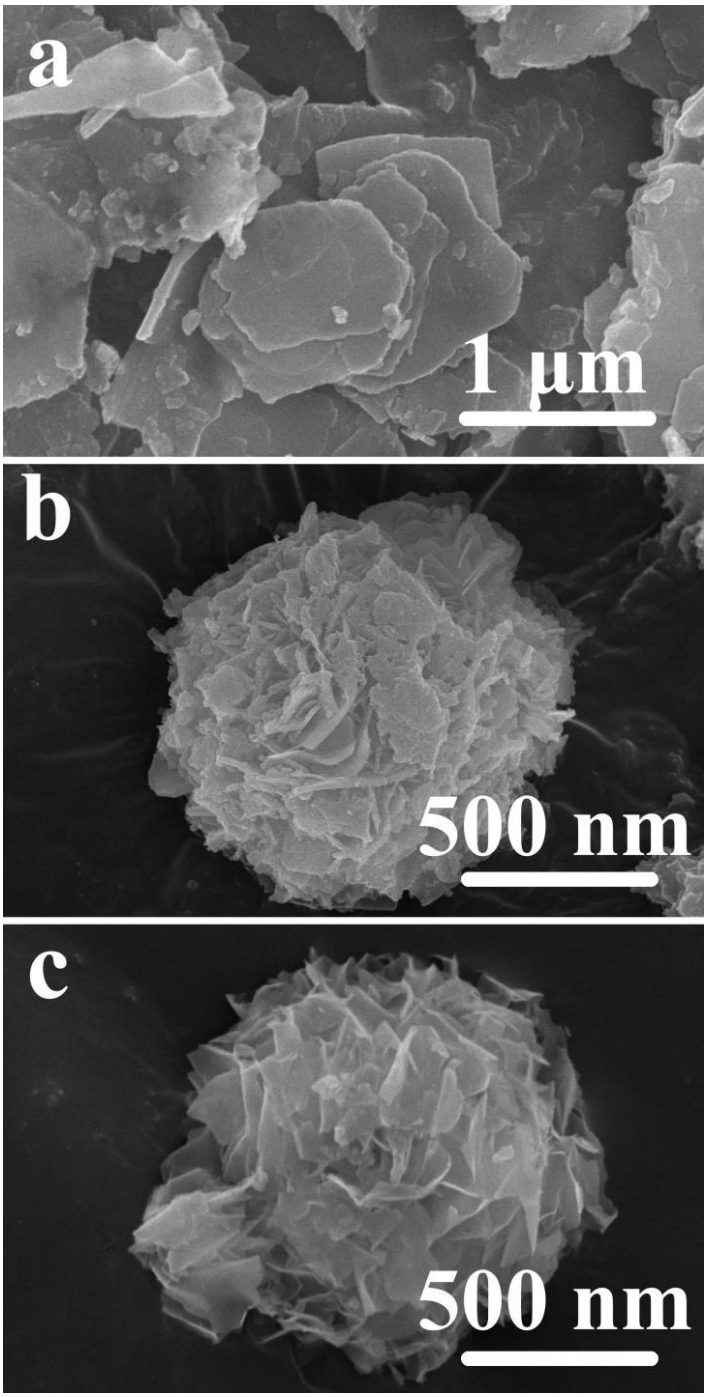

Fig. S4. SEM images of samples: (a) ZA-2; (b) ZAS-2*; (c) ZAS-2.

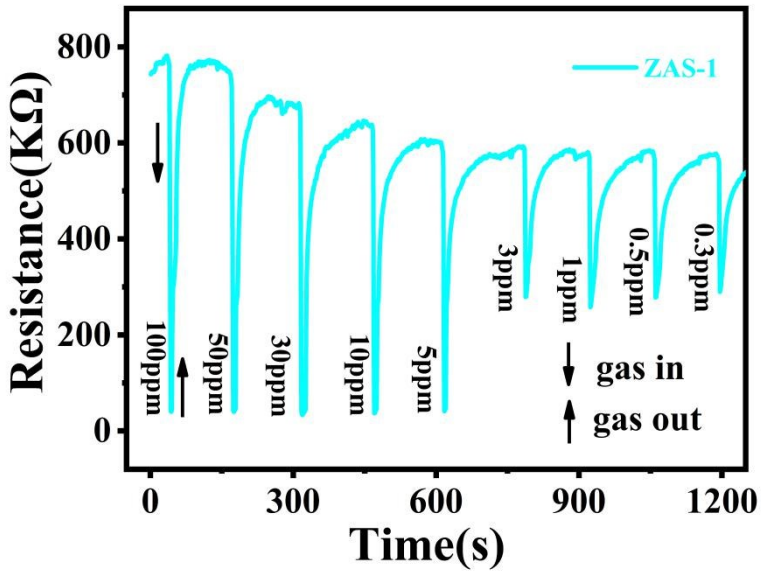

Fig. S5. Dynamical response transient of ZAS-1 to 100-0.3 ppm NO_2.

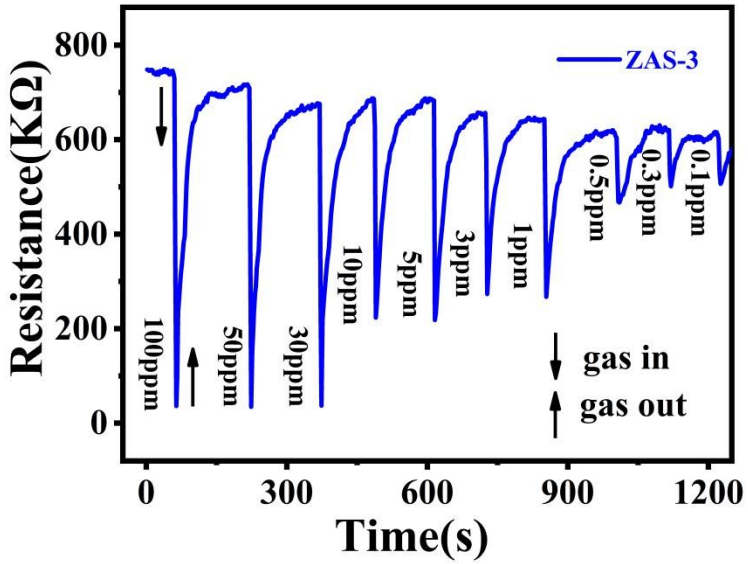

Fig. S6. Dynamical response transient of ZAS-3 to 100-0.1 ppm NO_2.

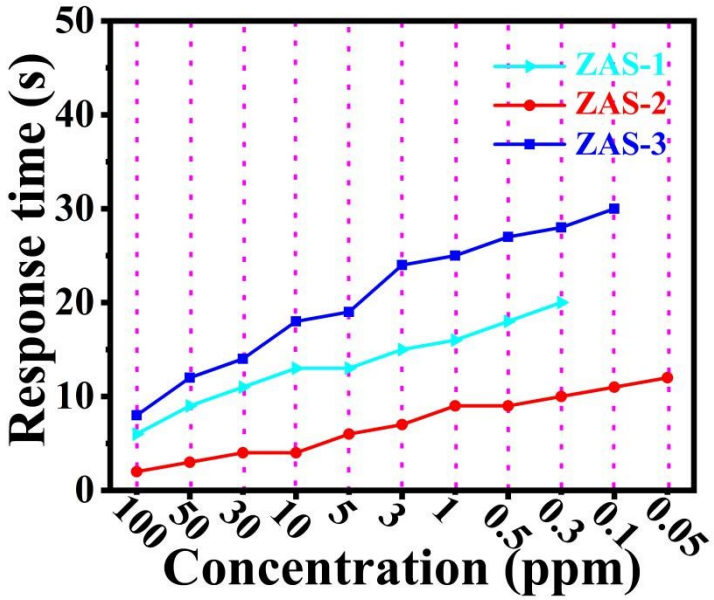

Fig. S7. Response time of the ZAS-1, ZAS-2 and ZAS-3 sensor.

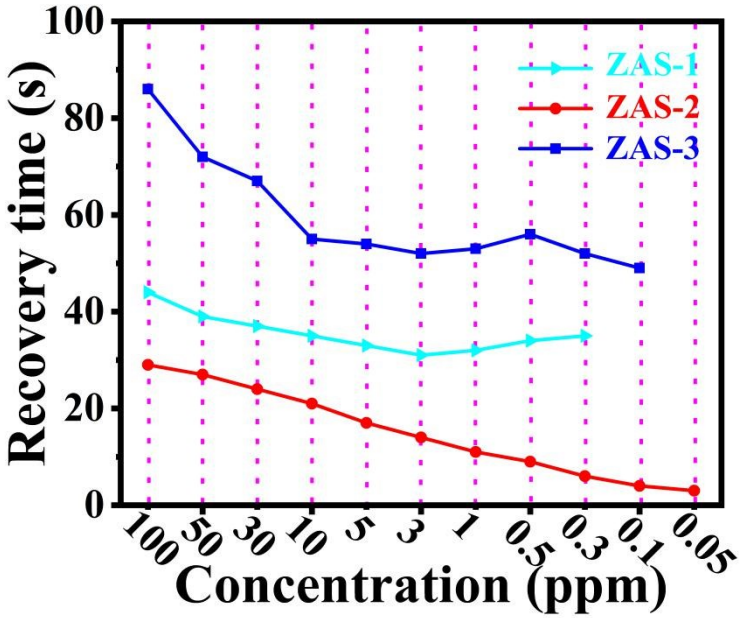

Fig. S8. Recovery time of the ZAS-1, ZAS-2 and ZAS-3 sensor.

Table S3. Response and response time of all samples to NO_2 at 25°C in air (RH: 26%)

Sample	ZAS-1			**ZAS-2**			ZAS-3		
NO_2 (ppm)	R	T_{R1}/s	T_{R2}/s	**R**	**T_{R1}/s**	**T_{R2}/s**	R	T_{R1}/s	T_{R2}/s
100	20	6	44	**28.6**	**2**	**29**	19	8	86
50	19	9	39	**23.9**	**3**	**27**	18	12	72
30	13	11	37	**17.5**	**4**	**24**	13.8	14	67
10	5	13	35	**10.1**	**4**	**21**	7	18	55
5	3	13	33	**6.9**	**6**	**17**	4	19	54
3	2.4	15	31	**5.2**	**7**	**14**	3	24	52
1	2.2	16	32	**4.2**	**9**	**11**	2.2	25	53
0.5	1.3	18	34	**3.5**	**9**	**9**	2.1	27	56
0.3	1.2	20	35	**3.1**	**10**	**6**	1.7	28	52
0.1	--	--	--	**2.62**	**11**	**4**	1.3	30	49
0.05	--	--	--	**2**	**12**	**3**	--	--	--

⋆R: Response; T_{R1}: Response time; T_{R2}: Recovery time.

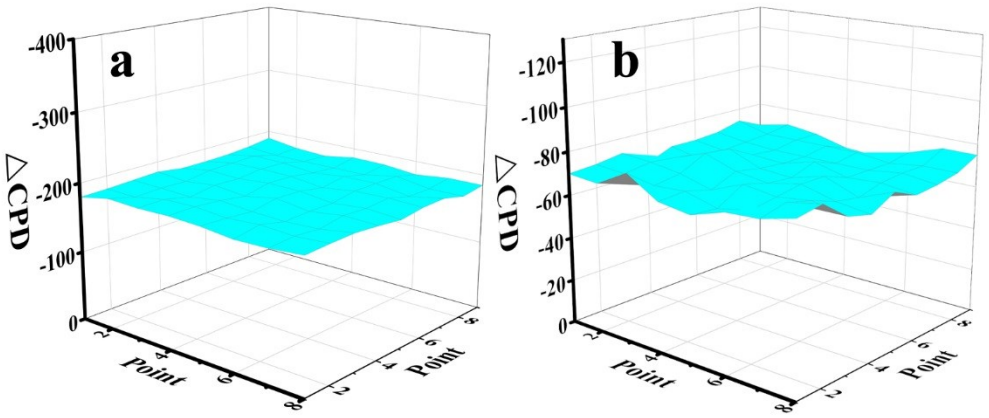

Fig. S9. Scheme of Kelvin probe of (a) $Al(OH)_3$, (b) $Zn(OH)_2$.