† Electronic Supporting Information (†ESI)

Boron Nanostructures Obtained via Ultrasonic Irradiation for High Performance Chemiresistive Methane Sensors

*Ravindra Kumar Jha,[a] Aman Nanda, [a] and Navakanta Bhat [*a]*

[a] Nano-Devices and Sensors Laboratory, Centre for Nanoscience and Engineering, Indian Institute of Science, Bangalore, Karnataka, India-560012

Email: navakant@iisc.ac.in

The custom-built gas sensing set-up is shown in Figure S1. An external bias voltage of 5V was applied during sensing measurement.

Figure S1. Custom built Gas sensing Setup

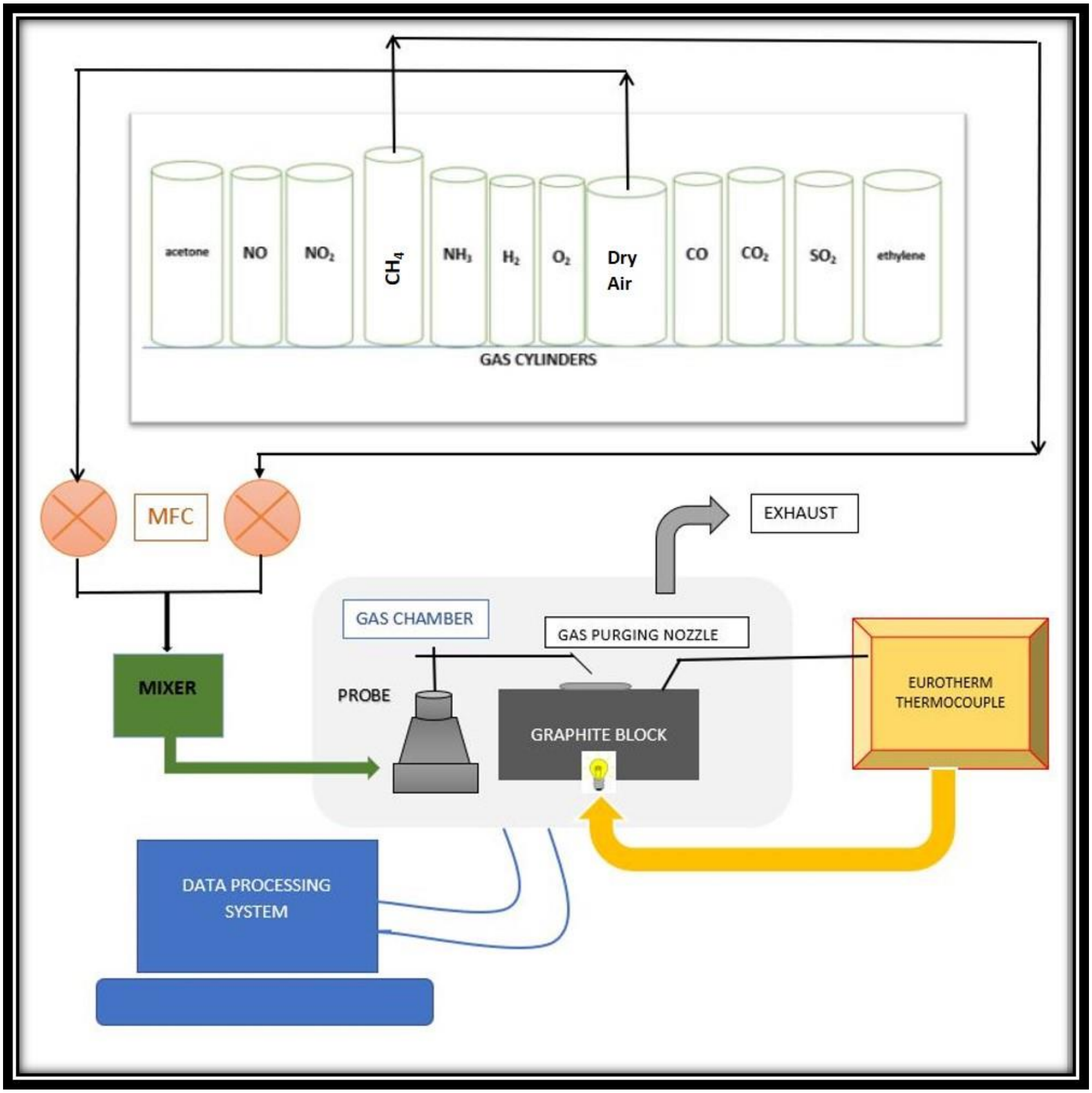


Figure S2. TGA Data for Bulk Boron and Few Layered Boron Sheets

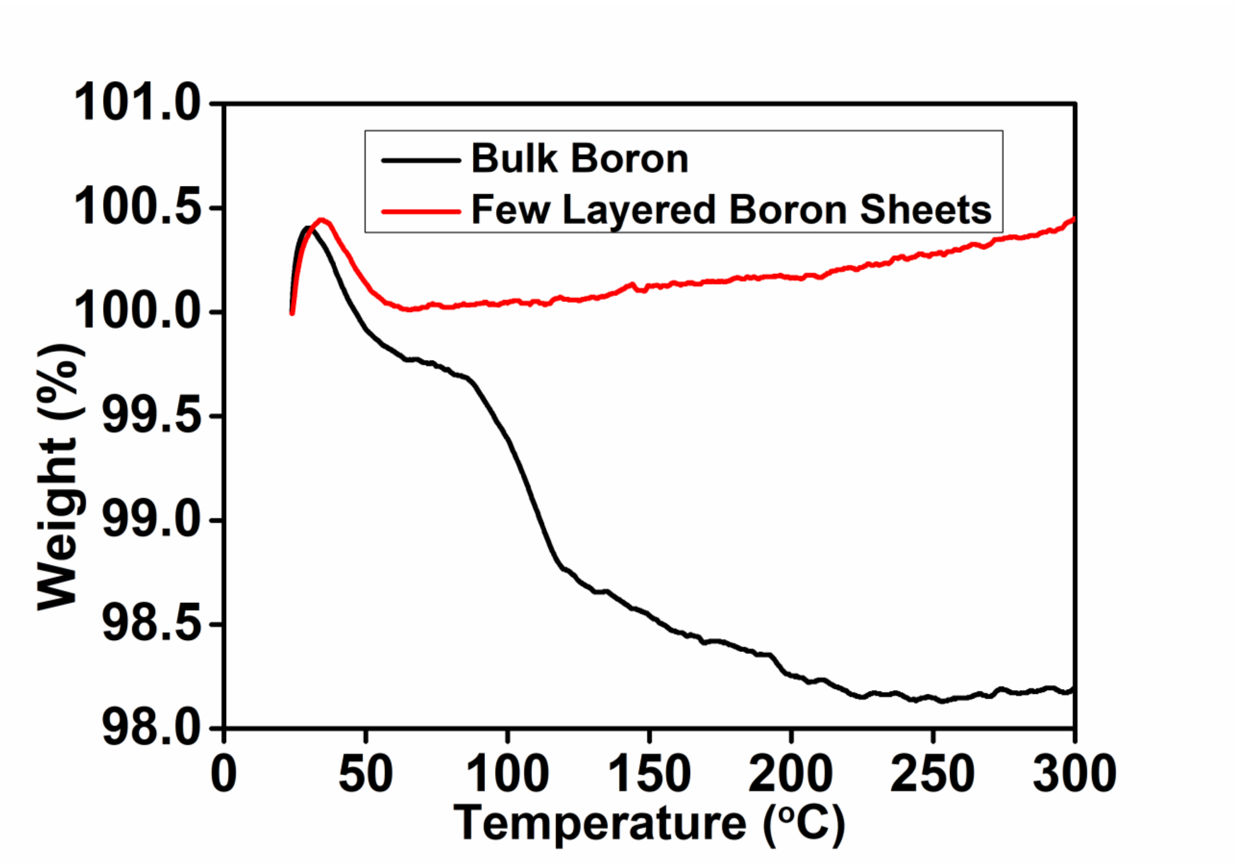


Figure S3. (a) pore size distribution in Bulk Boron

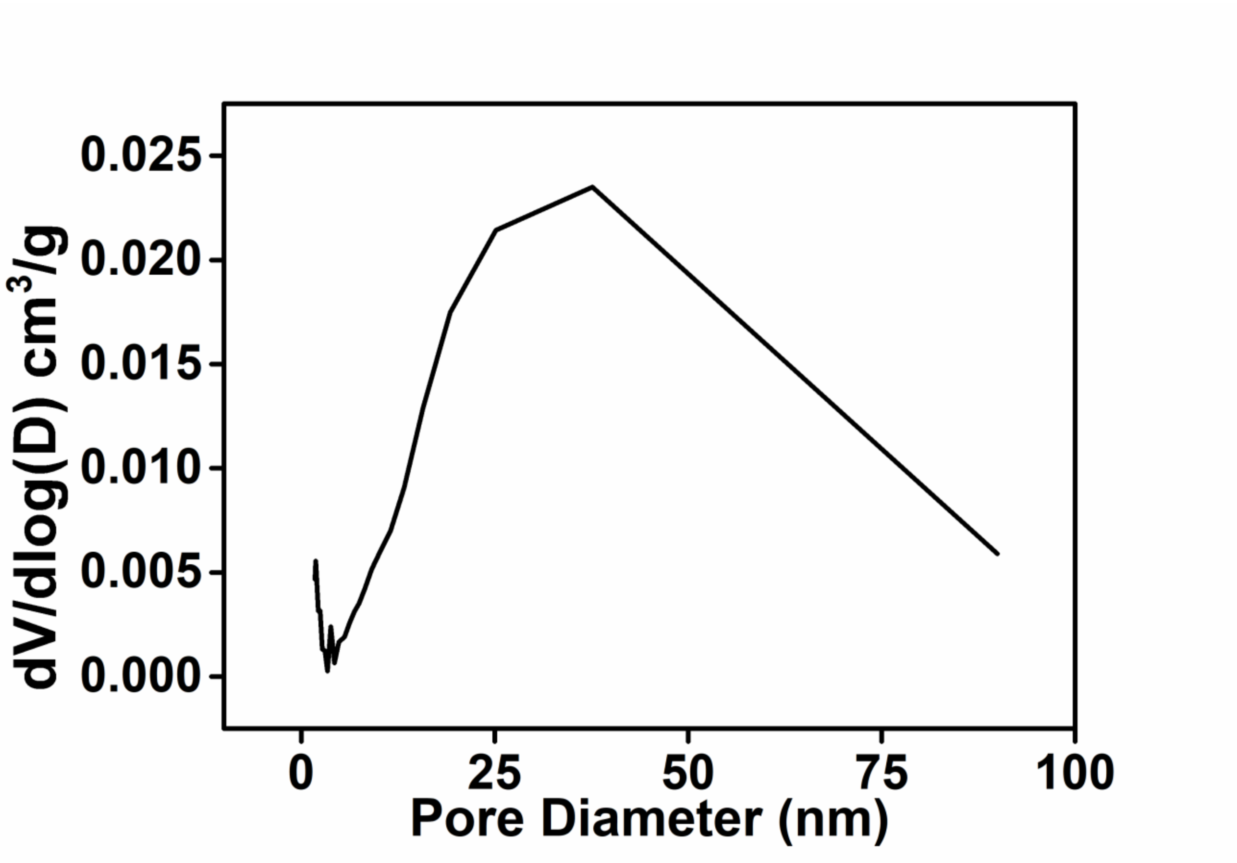


Figure S3. (b) pore size distribution in Exfoliated Boron Nanostructure

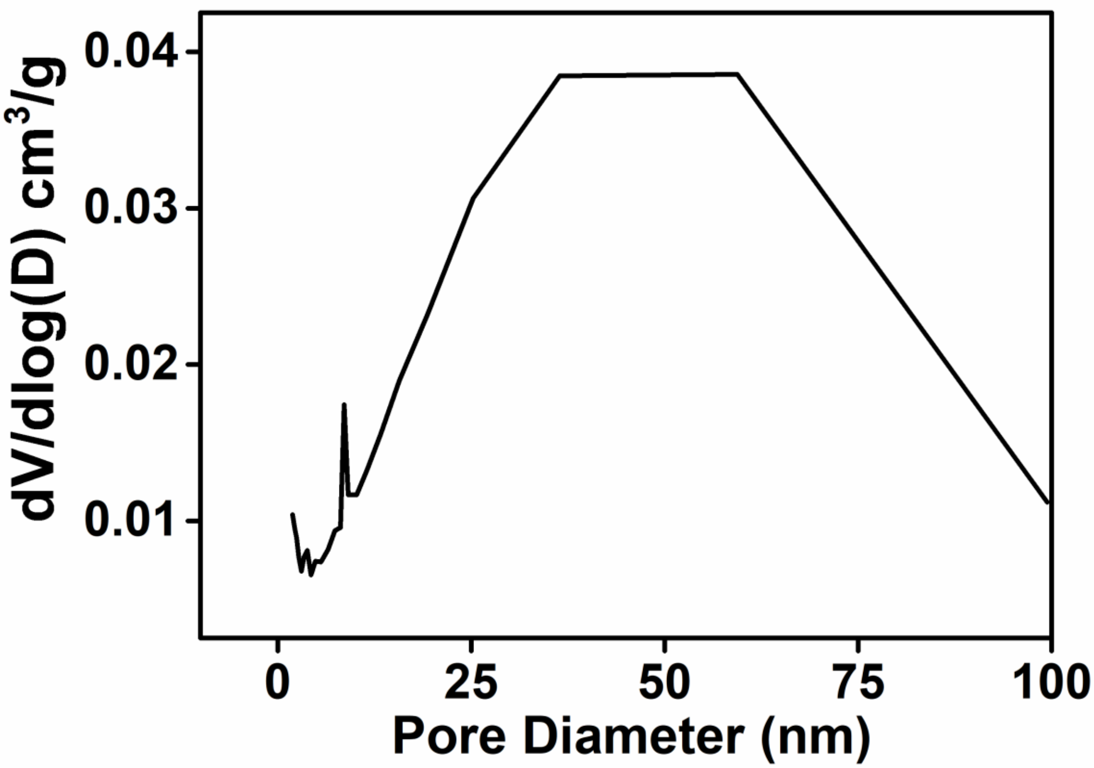


Figure S4. (a) AFM image of as obtained boron nanostructures

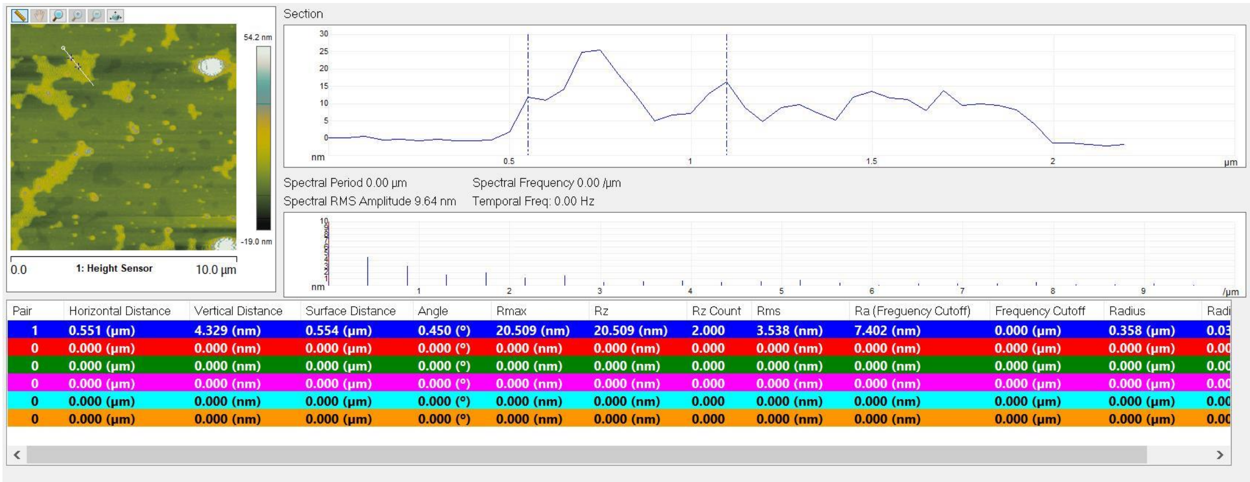


Figure S4. (b) and Raman spectrum corresponding to it

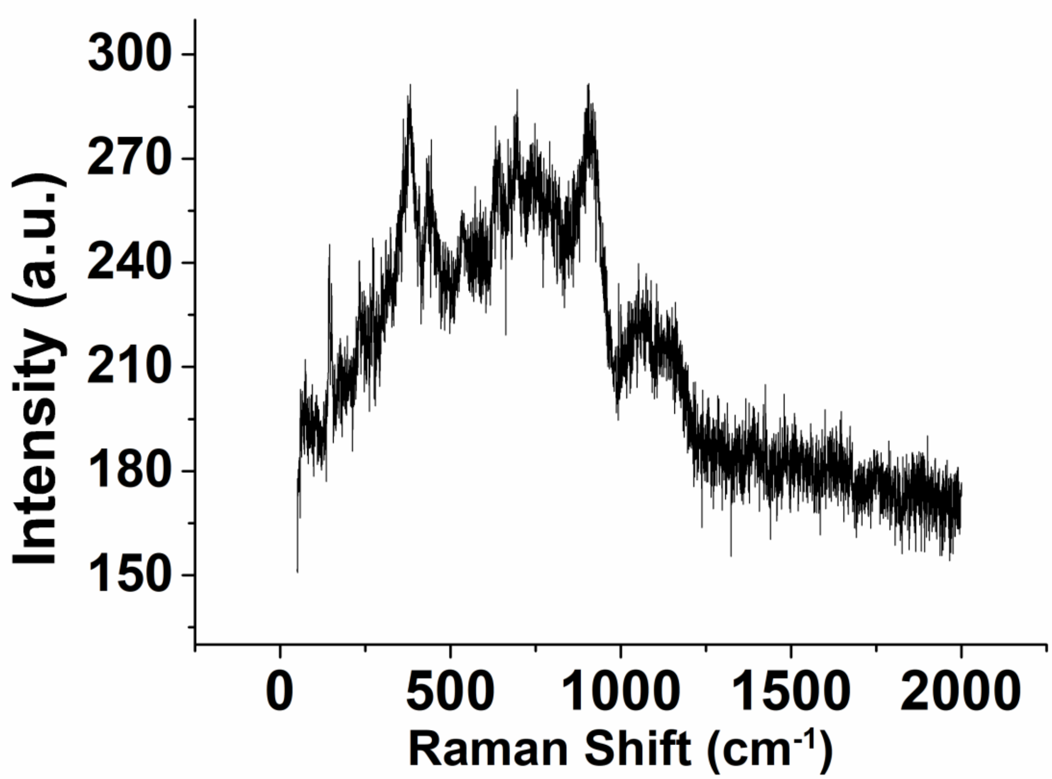
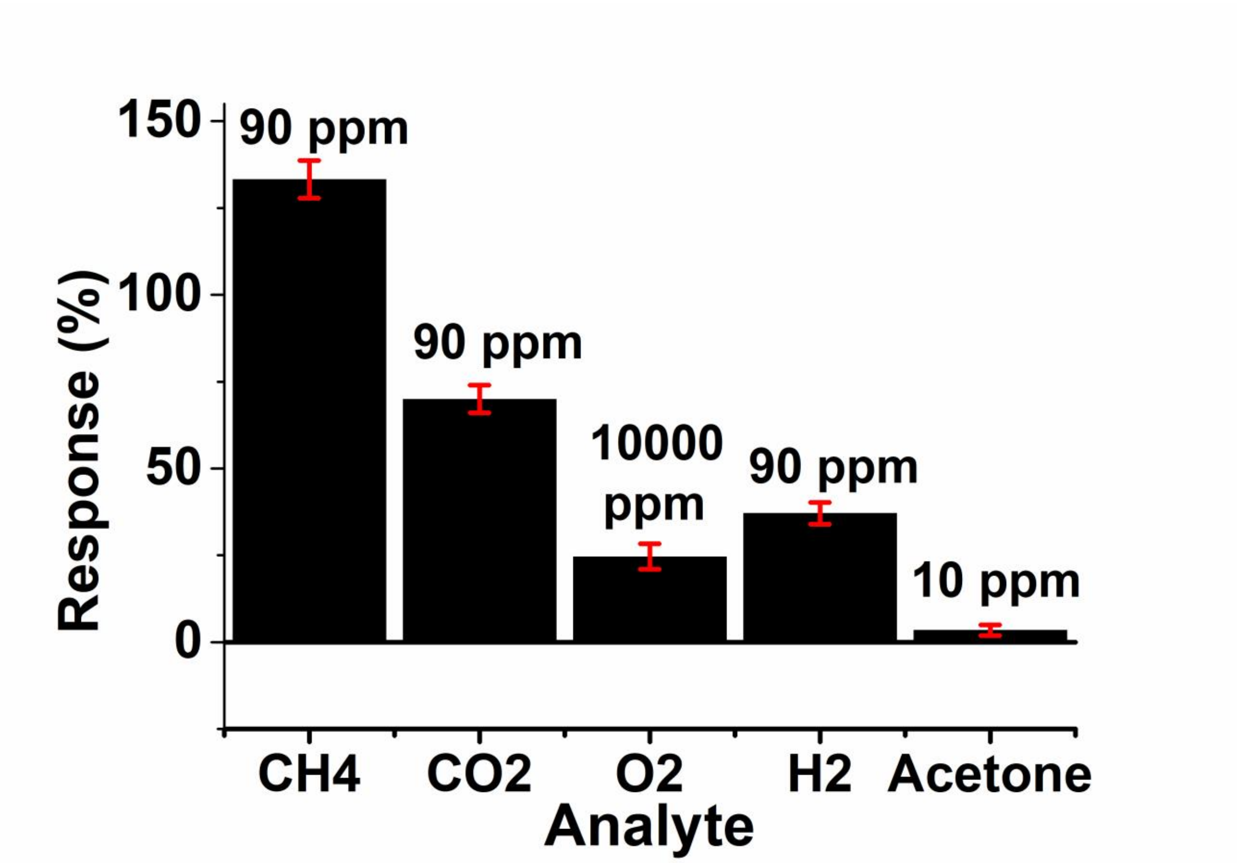


Figure S5. Selectivity of few layered boron nanostructures-based chemiresistive device



Supplementary Note 1: Fabrication of Device

Interdigitated electrodes (IDEs) with width and gap of 5 μm were fabricated on Si/SiO_2 by photolithography. In the first step, photoresist (AZ5214E) was spin coated (in steps of (i) 500 rpm for 5 seconds., (ii) 4000 rpm for 40seconds. and (iii) 500 rpm for 5 seconds.) on the Si/SiO_2 wafer. In the second step, it was exposed to UV light (for 6 seconds.; MJB4 mask aligner (Suss MicroTec)) after pre-baking (110°C for 600 seconds.). Development was done using MF 26A developer (for 18 seconds.) and was stopped by dipping in water. The Ti/Pt electrodes (10 nm/90 nm) were deposited using DC magnetron sputtering (Ti deposition: DC power 150 W, deposition time 25 seconds, Pt deposition: DC power 35 W, deposition time 150 seconds) followed by lift off. The optical micrograph of the finished device is shown in Fig. S6(a). 130 microlitre of exfoliated few layered boron sheets were dropcasted on the IDE fingers and calcinated at 120°C for half-an-hour hours in ambient to improve electrical contact. The optical micrograph of the finished device is shown in Fig. S6(b).

Fig. S6. Optical microscope image of the device (a) before dropcasting and (b) after dropcasting the sensing material

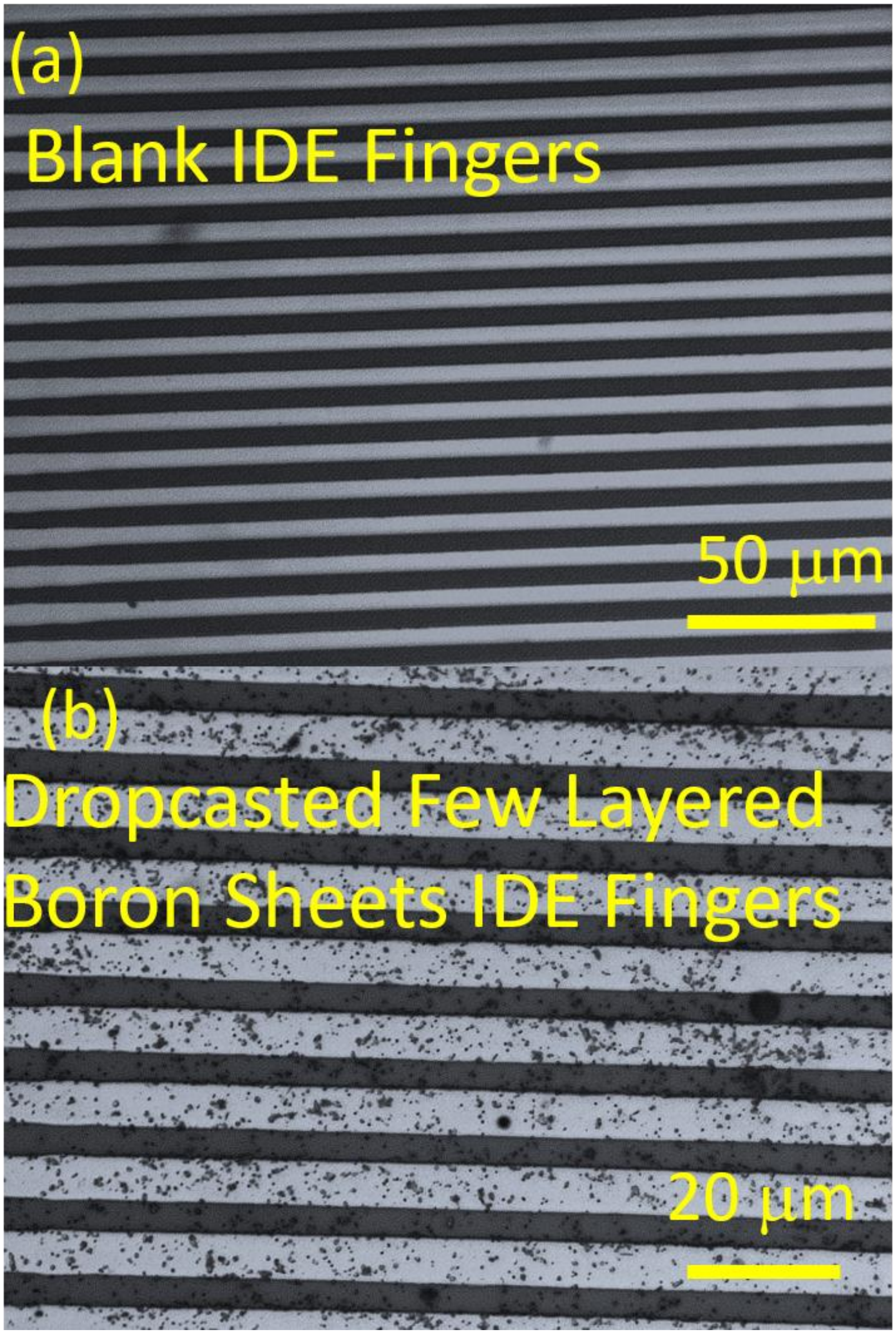


Fig. S7. Variation of response for four different devices

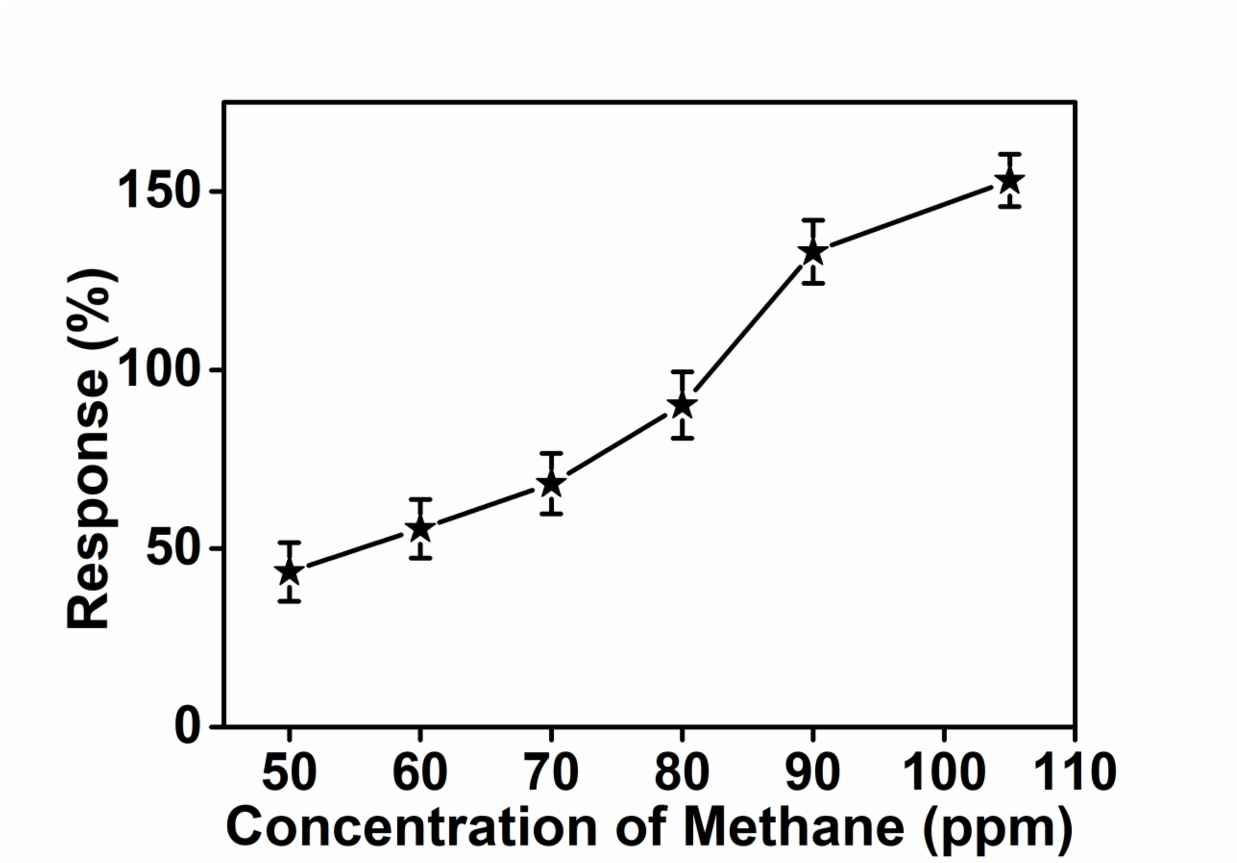


Fig. S8. (a) Large area Low Mag TEM image of as-prepared boron nanostructure

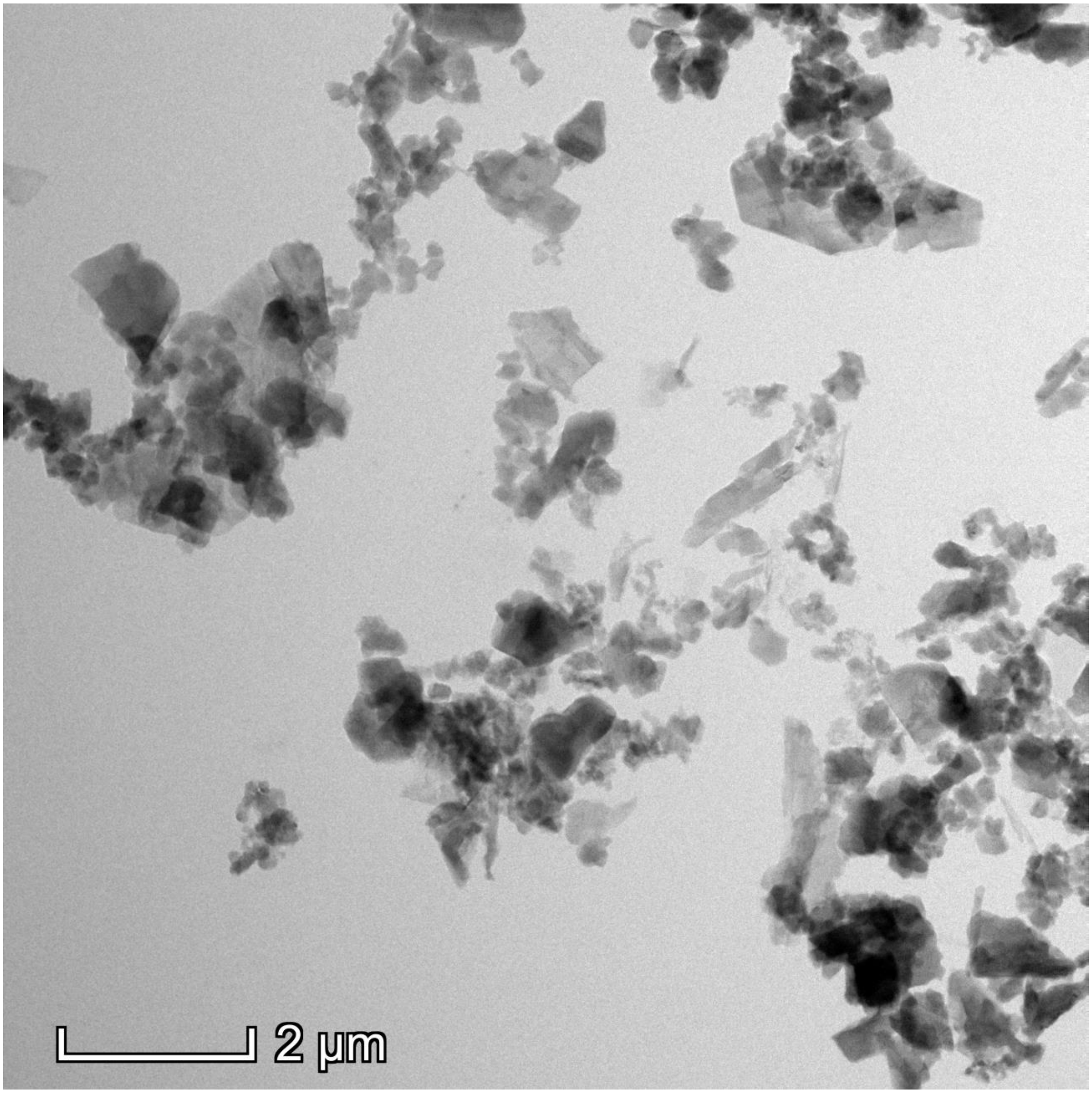


Fig. S8. (b) and its statistical distribution

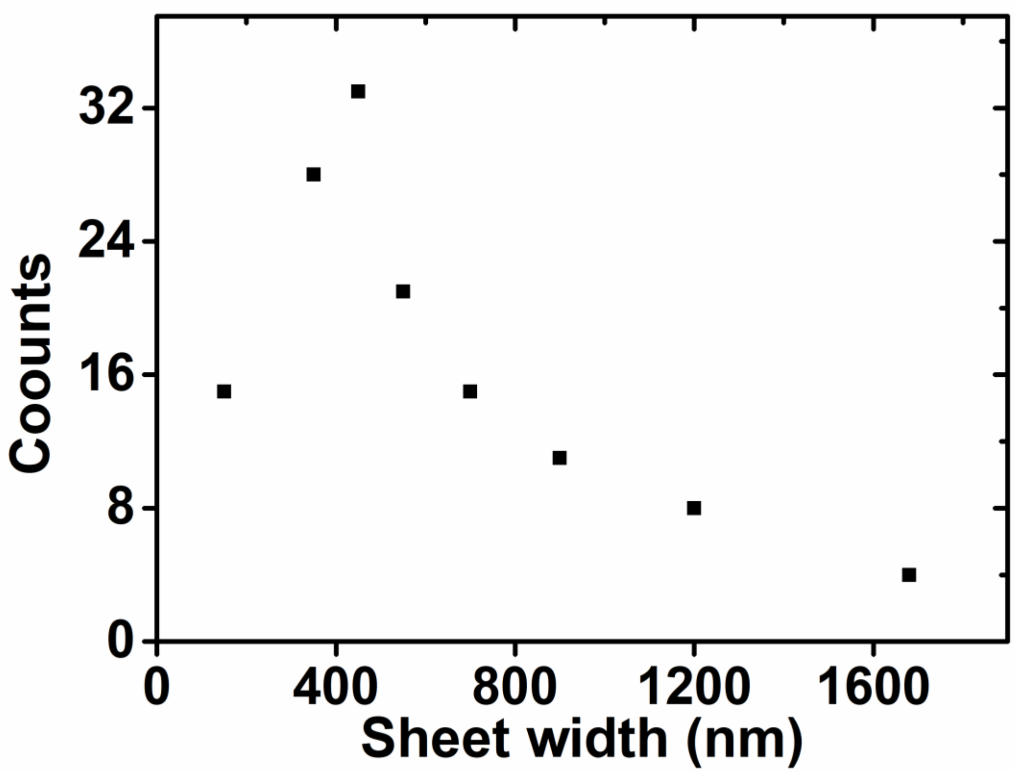


Fig. S9. XRD data of the exfolated boron nanostrucutre after 3 months

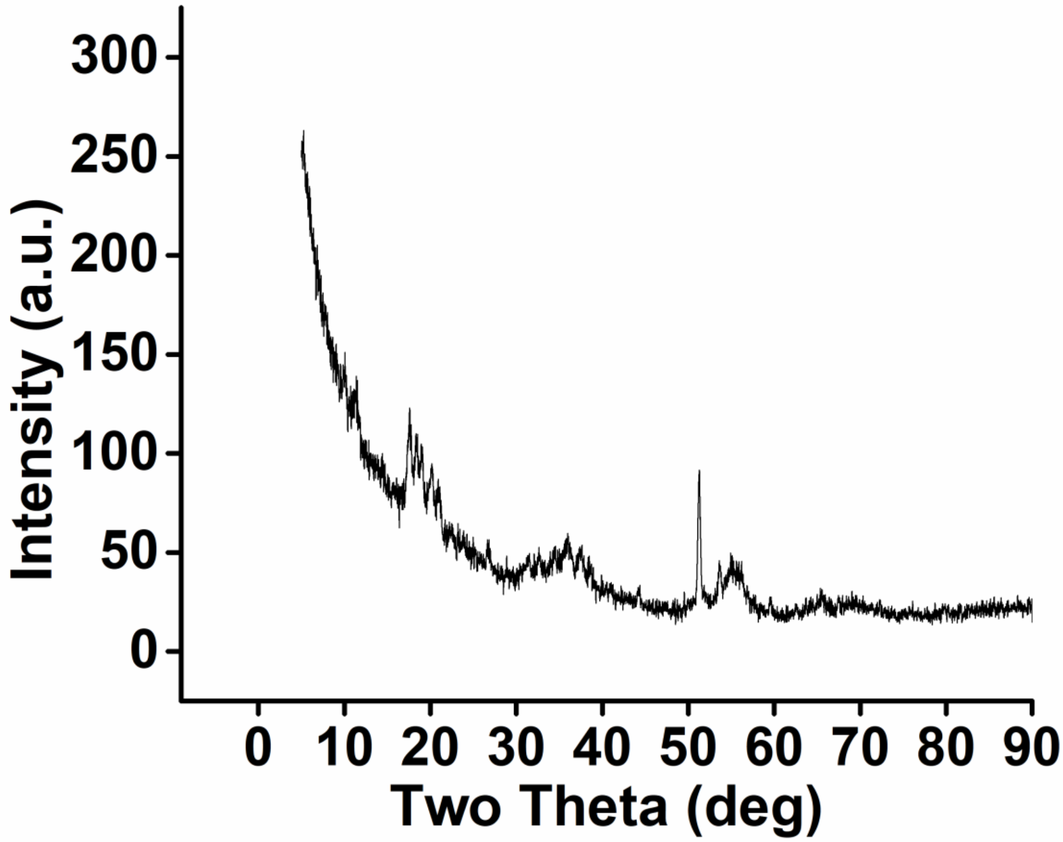


Fig. S10. XPS Survey Data for bulk and nanostructured Boron

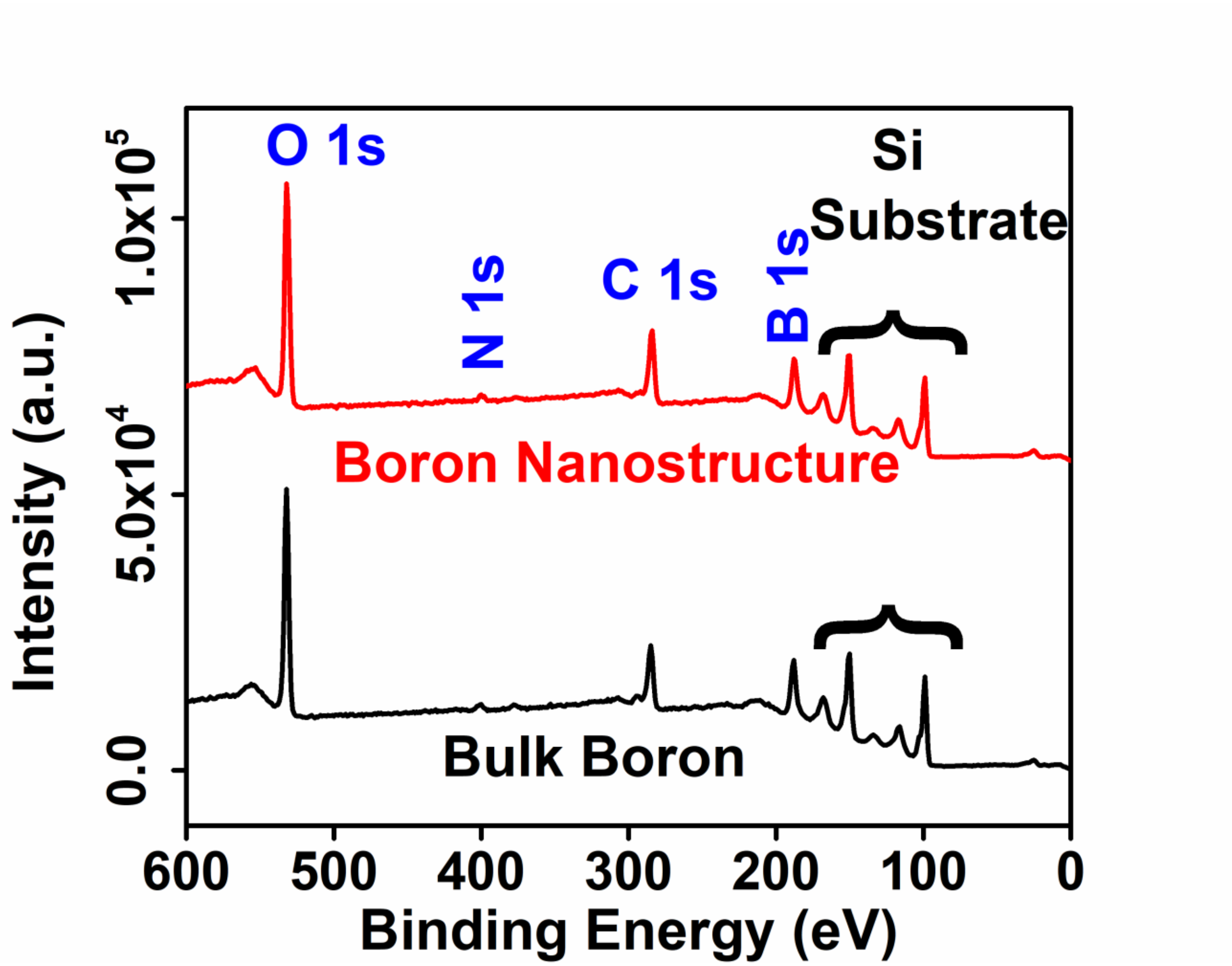


Table S1 Comparision of Few Layered Boron Sheets based methane sensor with other high performance methane sensor

S.No	Material	Response (%)	Response Time/ Recovery Time (Seconds)	Operating Temperature (°C)	LOD	Ref.
1.	ZnO	87.3 @ 10,000 ppm	8.3/17.8 @ 10,000 ppm	250	100 ppm	1
2.	SnO_2	862 @ 2500 ppm	~30/- @ 2500 ppm	300	125 ppm	2
3.	In_2O_3	225@ 10,000 ppm	200/- @10,000 ppm	300	2500 ppm	3
4.	Co_3O_4	128 @ 10,000 ppm	100/50 @ 10,000 ppm	200	2500 ppm	4
5.	Co doped ZnO	350 @ 100 ppm	19/27 @ 100 ppm	140	50 ppb	5
6.	Pt-Co_3O_4/MoS_2	7.43 @ 1000 ppm	-	170	500 ppm	6
7.	NiO/RGO	15.2% @ 1000 ppm	18/20 @ 500 ppm	260	100 ppm	7
8.	Few Layered Boron Nanostructure	90.2% @ 80 ppm	41.8/52 @ 39.6 @ 80 ppm	RT	50 ppm (50 ppm- 105 ppm)	Our work

Table S2 Comparision of Few Layered Boron Sheets based methane sensor with other Room Temperature Methane sensor.

S.No	Material	Response (%)	Concentration Range (ppm)	Response Time/ Recovery Time (Seconds)	LOD	Ref.
1	RGO/SnO_2	76 @ 10,000 ppm	10,000-80,000	200/- @ 10,000 ppm	10,000 ppm	8
2	Au/VO_x	18.2 @ 500 ppm	500-2000	~2000/1000 @ 500 ppm	500 ppm	9
3	PANI/ZnO	48@100 ppm	100-500	20/>250 @ 500 ppm	100 ppm	10
4	PbS	~12 @ 10,000 ppm	10,000-50,000	-/-	10,000 ppm	11
5	TiO_2	~610 @ 5 ppm	5-100	45/33 @ 60 ppm	5 ppm	12
6	Few Layered Boron Nanostructure	43.5 @ 50 ppm	50-105	41.8/52 @ 39.6 @ 80 ppm	50 ppm (50 ppm- 105 ppm)	Our work

Supplementary References

1. P. Bhattacharyya, P. Basu, B. Mondal and H. J. M. R. Saha, 2008, **48**, 1772-1779.
2. A. Biaggi-Labiosa, F. Solá, M. Lebrón-Colón, L. J. Evans, J. C. Xu, G. W. Hunter, G. M. Berger and J. M. González, *Nanotechnology*, 2012, **23**, 455501.
3. N. M. Shaalan, M. Rashad and M. A. Abdel-Rahim, *Materials Science in Semiconductor Processing*, 2016, **56**, 260-264.
4. N. M. Shaalan, M. Rashad, A. H. Moharram and M. A. Abdel-Rahim, *Materials Science in Semiconductor Processing*, 2016, **46**, 1-5.
5. J. Hu, F. Gao, Z. Zhao, S. Sang, P. Li, W. Zhang, X. Zhou and Y. Chen, *Applied Surface Science*, 2016, **363**, 181-188.
6. D. Zhang, H. Chang, Y. e. Sun, C. Jiang, Y. Yao and Y. Zhang, *Sensors and Actuators B: Chemical*, 2017, **252**, 624-632.
7. D. Zhang, H. Chang, P. Li and R. Liu, *Journal of Materials Science: Materials in Electronics*, 2016, **27**, 3723-3730.
8. K. C. Lam, B. Huang and S.-Q. Shi, *Journal of Materials Chemistry A*, 2017, **5**, 11131-11142.
9. J. Liang, J. Liu, N. Li and W. Li, *Journal of Alloys and Compounds*, 2016, **671**, 283-290.
10. T. Sen, S. Mishra, S. S. Sonawane and N. G. Shimpi, 2018, **58**, 1438-1445.
11. A. Mosahebfard, H. D. Jahromi and M. H. Sheikhi, *IEEE Sensors Journal*, 2016, **16**, 4174-4179.
12. Z. P. Tshabalala, K. Shingange, B. P. Dhonge, O. M. Ntwaeaborwa, G. H. Mhlongo and D. E. Motaung, *Sensors and Actuators B: Chemical*, 2017, **238**, 402-419.