## Supporting Information

## Pollutant capturing SERS substrate: Porous boron nitride microfibers with uniform silver nanoparticles decoration

Pengcheng Dai\*, Yanming Xue\*, Qunhong Weng, Chao Zhang, Xuebin Wang, Xiangfen Jiang\*, Xi Wang, Naoyuki Kawamoto, Yusuke Ide, Masanori Mitome, Dmitri Golberg\*, Yoshio Bando

## Content

1. Fig. S1 XPS spectra of BN microfibers.

2. Fig. S2 XPS spectra of BN microfibers with Ag+ absorbed on their surface.

3. Fig. S3 XPS spectra of BN microfibers with Ag nanoparticles on their surface.

4. Fig. S4 Low magnification SEM images of M2B precursor, BN microfibers and BN/Ag hybrids.

5. Fig. S5 TEM images of Ag nanoparticles with different sizes and shapes on BN microfibers.

6. Fig. S6 TEM images of two other parts of Ag nanoparticle-decorated BN microfibers and the size

distribution of Ag nanoparticles.

7. Fig. S7 Nitrogen adsorption-desorption isotherms of BN microfibers and Ag-decorated BN microfibers.

8. Fig. S8 UV-vis spectra of RhB solutions with different concentrations.

9. Fig. S9 UV-vis spectra of RhB solutions after BN/Ag adsorption.

10. **Fig. S10** Raman spectra of the solutions with the same amount of MB but at different concentrations and volumes.

11. **Fig. S11** Raman spectra of RhB (10<sup>-6</sup> M) on ten different parts of BN/Ag membrane before and after reusability test.

12. Fig. S12 Adsorption capacities and SERS activities of BN, BN/Ag and Ag NPs.



**Fig. S1** XPS spectra of BN microfibers. (a) Survey spectrum indicates the presence of B, N, C and O. (b) B 1s spectrum. The main sp<sup>2</sup>-hybridized BN structure shows a peak at 190.8 eV, while a shoulder peak located at 192.7 eV is attributed to the edge or interfacial BN structures linked with –OH. (c) N 1s spectrum. Besides the main sp<sup>2</sup>-hybridized BN structures, the shoulder peak located at 400.5 eV belongs to the surface –NH– or –NH<sub>2</sub> groups (d) O 1s spectrum. O 1s peak at 533.0 eV can be fitted to hydroxyl.



**Fig. S2** XPS spectra of BN microfibers with Ag<sup>+</sup> absorbed on their surface. (a) Survey spectrum indicates the presence of B, N, C, O and Ag. (b) B 1s spectrum. The main sp<sup>2</sup>-hybridized BN structure shows a peak at 190.8 eV, while a shoulder peak located at 192.7 eV is attributed to the edge or interfacial BN structures linked with –OH. (c) N 1s spectrum. Besides the main sp<sup>2</sup>-hybridized BN structures, the shoulder peak located at 401.0 eV belongs to the surface –NH– or –NH<sub>2</sub> groups linked with Ag<sup>+</sup>. (d) Ag  $3d_{5/2}$  spectrum. Peaks at 367.7 eV and 368.4 eV are attributed to Ag<sup>+</sup> and Ag<sup>0</sup>, respectively. Judging from the peak area, the main chemical state of silver is Ag<sup>+</sup>.



**Fig. S3** XPS spectra of BN microfibers with Ag nanoparticles on their surface. (a) Survey spectrum indicates the presence of B, N, C, O and Ag. (b) B 1s spectrum. The main sp<sup>2</sup>-hybridized BN structure shows a peak at 190.8 eV, while a shoulder peak located at 192.7 eV is attributed to the edge or interfacial BN structures linked with –OH. A third peak fitted at 188.4 eV usually presents in boride, which we attributed to the contact of B and Ag. (c) N 1s spectrum. Besides the main sp<sup>2</sup>-hybridized BN structures, the shoulder peak located at 401 eV belongs to the surface –NH– or –NH<sub>2</sub> groups. (d) Ag 3d<sub>5/2</sub> spectrum. Peaks at 367.7 eV and 368.4 eV are attributed to Ag<sup>+</sup> and Ag<sup>0</sup>, respectively. Most of the Ag<sup>+</sup> has transformed into metallic silver, as judged from the peak areas of Ag<sup>+</sup> and Ag<sup>0</sup>.

b) 80 μm 80 μm

Fig. S4 Low-magnification SEM images of a) M2B precursor b) BN microfibers and c) BN/Ag hybrids.

a) <u>20 m</u> <u>40 m</u> <u>10 m</u>

Fig. S5 TEM images of Ag nanoparticles with different sizes and shapes on a BN microfiber.



Fig. S6 TEM images of the other two parts of Ag nanoparticle-decorated BN microfibers and the size distribution of Ag nanoparticles.



**Fig. S7** Nitrogen adsorption-desorption isotherms of BN microfibers and Ag-decorated BN microfibers. Bare BN microfibers hold a high specific surface area of 930 m<sup>2</sup>/g. The loaded Ag nanoparticles occupied some holes within BN microfibers and, therefore, the resultant specific surface area decreased to 670 m<sup>2</sup>/g.



**Fig. S8** UV-vis spectra of RhB solutions whose concentrations are  $10^{-4}$  M,  $10^{-5}$  M,  $10^{-6}$  M,  $10^{-7}$  M,  $10^{-8}$  M and  $10^{-9}$  M.  $10^{-4}$  M RhB solution is so concentrated that it generates a flat peak tip. Therefore, we fit the curve to get the peak. RhB solutions of  $10^{-5}$  M,  $10^{-6}$  M,  $10^{-7}$  M and  $10^{-8}$  M show a characteristic peak at 552 nm.  $10^{-9}$  M RhB solution concentration is too low to be detected by UV-vis spectra. The dot-dash lines are baselines which are subtracted.



Fig. S9 UV-vis spectra of RhB solutions after BN/Ag adsorption.



Fig. S10 Raman spectra of the solutions with the same amount of MB but at different concentrations and volumes.



**Fig. S11** a) Raman spectra of RhB (10<sup>-6</sup> M) on ten different spots of BN/Ag membrane. b) Raman spectra of RhB (10<sup>-6</sup> M) on ten different parts of BN/Ag membrane after 10-cycles reusability test.



Raman shift (cm<sup>-1</sup>) **Fig. S12** a) Adsorption capacities of BN, BN/Ag and Ag NPs. b) Raman spectra of RhB ( $10^{-6}$  M) on BN/Ag, Ag NPs and BN.