

## Supplementary information

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# High-yield production of mono- or few-layer transition metal dichalcogenide nanosheets by an electrochemical lithium ion intercalation-based exfoliation method

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## *Supplementary information*

*for*

### **High-yield production of mono- or few- layer transition metal dichalcogenides nanosheets by electrochemical lithium ion intercalation-based exfoliation method**

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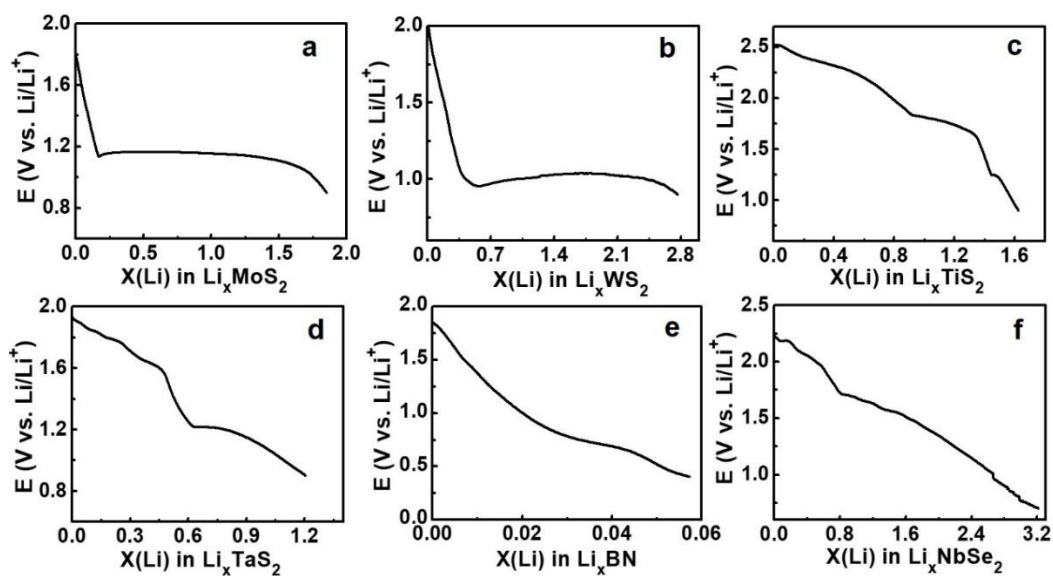
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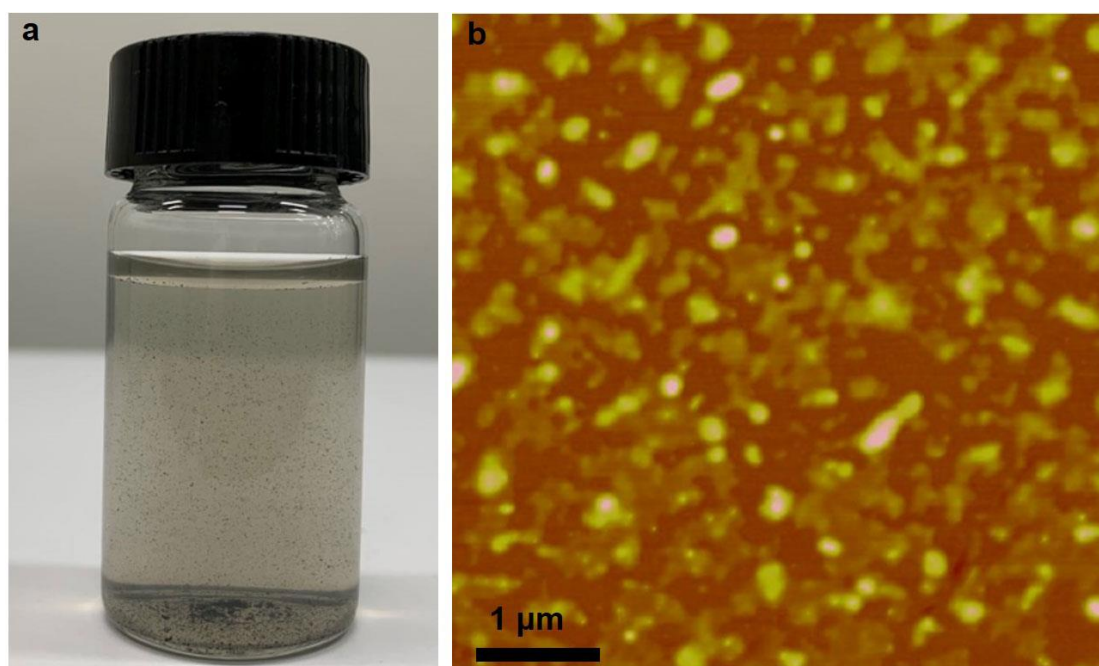
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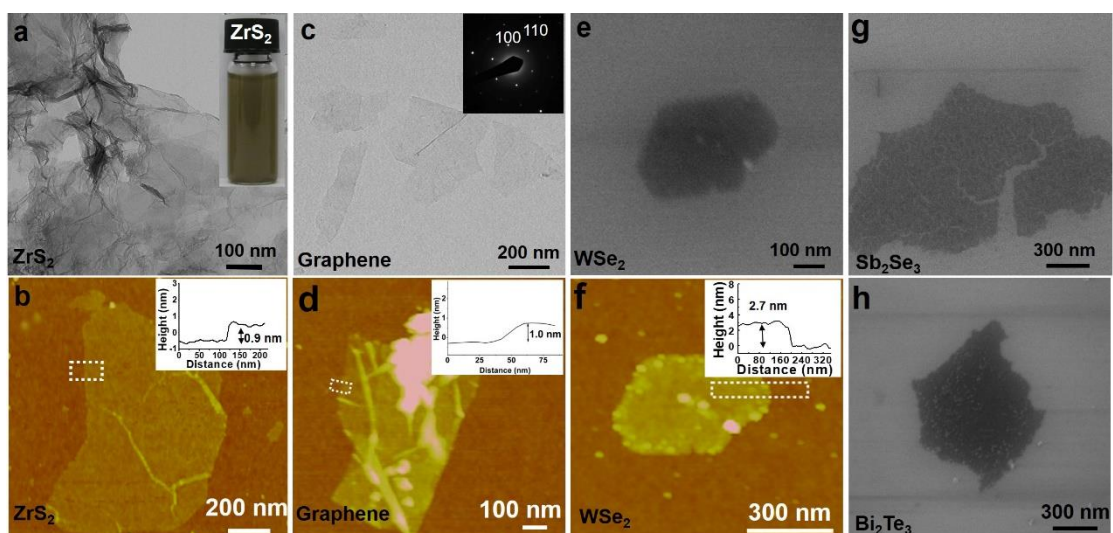
† Ruijie Yang, Liang Mei, and Qingyong Zhang contributed equally to this work.



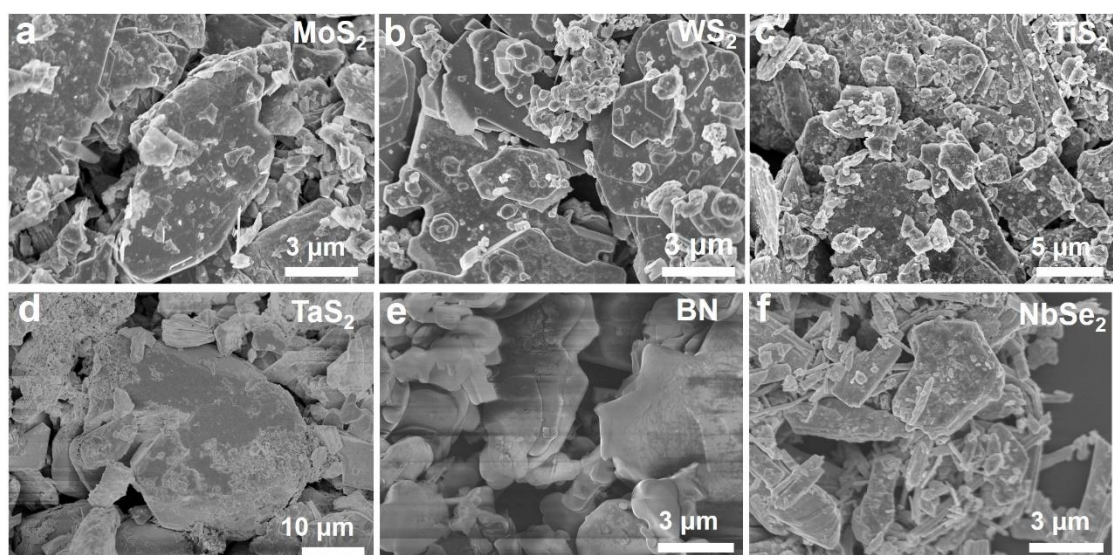
**Supplementary Figure 1** Galvanostatic discharge curve for (a)  $\text{MoS}_2$ , (b)  $\text{WS}_2$ , (c)  $\text{TiS}_2$ , (d)  $\text{TaS}_2$ , (e)  $\text{BN}$ , (f)  $\text{NbSe}_2$ . Adapted with permission from ref. <sup>1-3</sup>.



**Supplementary Figure 2** (a) Photographs of the exfoliated  $\text{MoS}_2$  nanosheets, which were not fully exfoliated, and some of the bulk particles can be clearly seen. (b) Unsatisfactory AFM image of the exfoliated  $\text{MoS}_2$  nanosheets, in which lots of particles are observed.

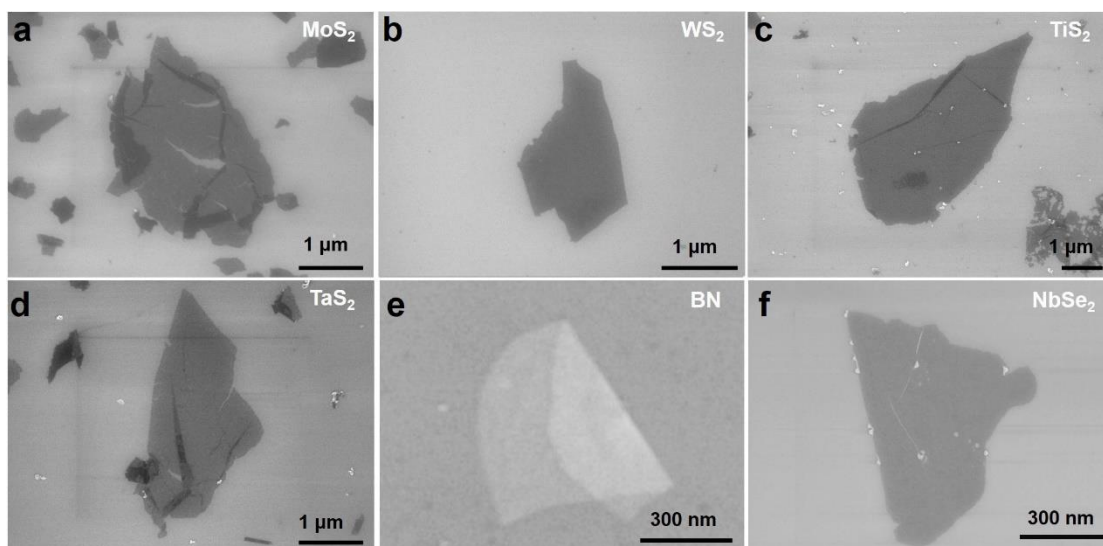


**Supplementary Figure 3** (a) TEM image of the exfoliated ZrS<sub>2</sub> nanosheets. (b) AFM image of the exfoliated ZrS<sub>2</sub> nanosheets, showing the average thickness of ~0.9 nm. (c) TEM image of the exfoliated graphene nanosheets. (d) AFM image of the exfoliated graphene nanosheets, showing the average thickness of ~1.0 nm. (e) SEM image of the exfoliated WSe<sub>2</sub> nanosheets. (f) AFM image of the exfoliated WSe<sub>2</sub> nanosheets, showing the average thickness of ~2.7 nm. (g) SEM image of the exfoliated Sb<sub>2</sub>Se<sub>3</sub> nanosheets. (h) SEM image of the exfoliated Bi<sub>2</sub>Te<sub>3</sub> nanosheets. Adapted with permission from ref. <sup>1,2</sup>.

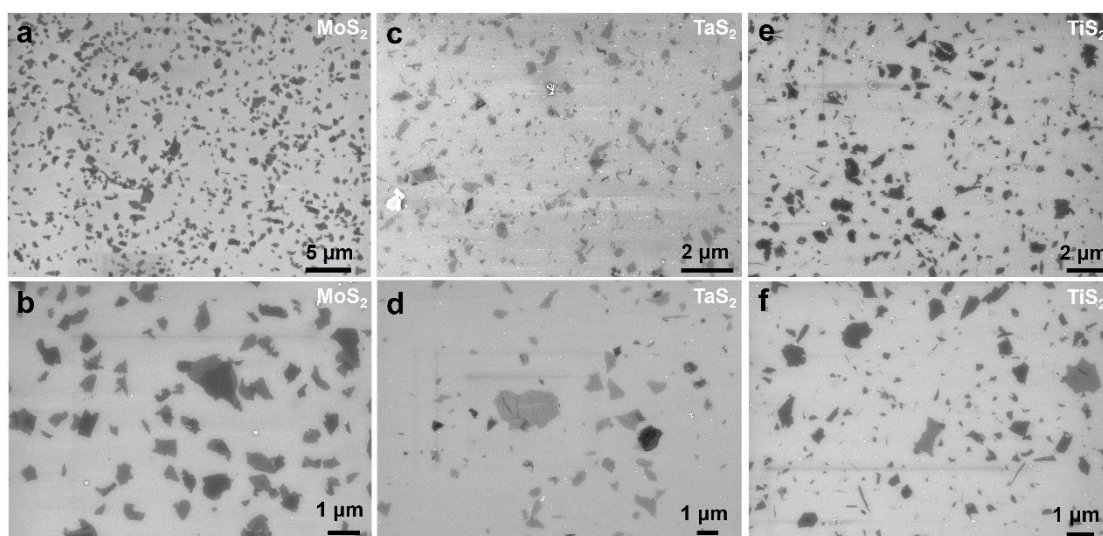


**Supplementary Figure 4** SEM images of (a) bulk MoS<sub>2</sub>, (b) bulk WS<sub>2</sub>, (c) bulk TiS<sub>2</sub>, (d) bulk TaS<sub>2</sub>, (e) bulk BN, (f) bulk NbSe<sub>2</sub>.

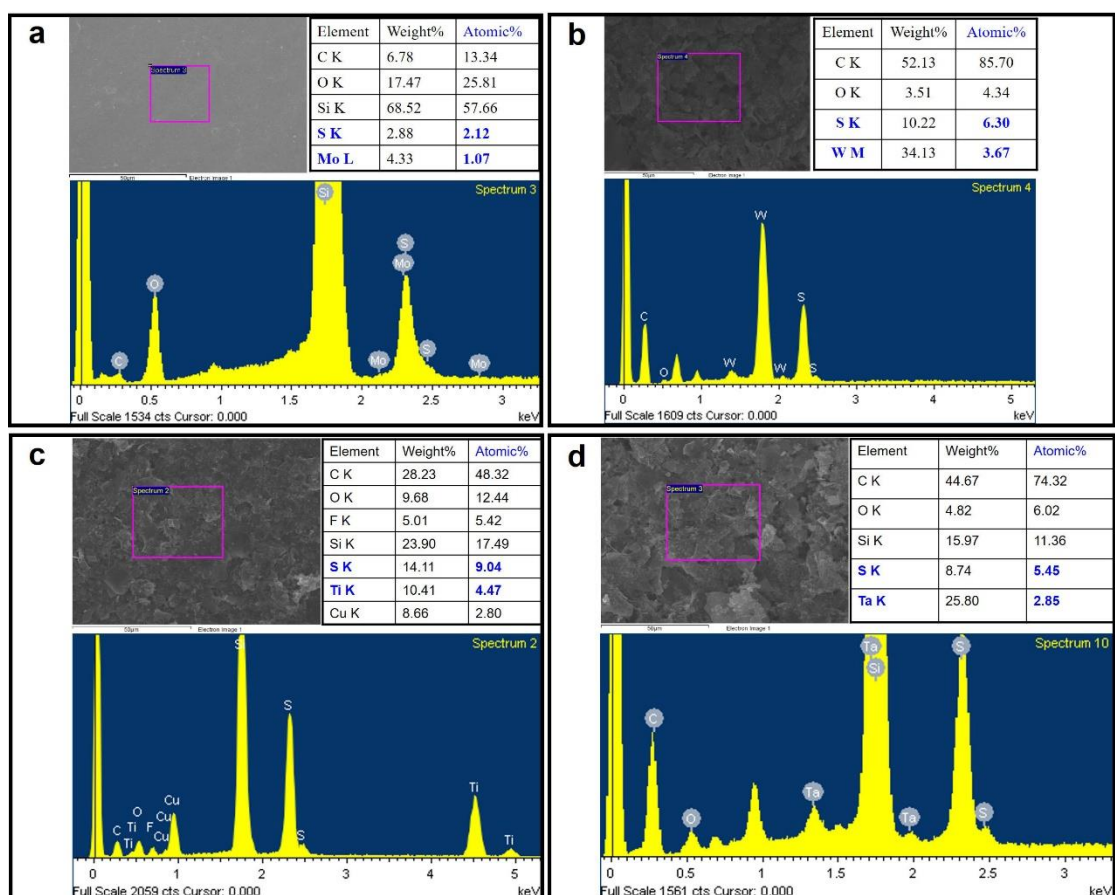




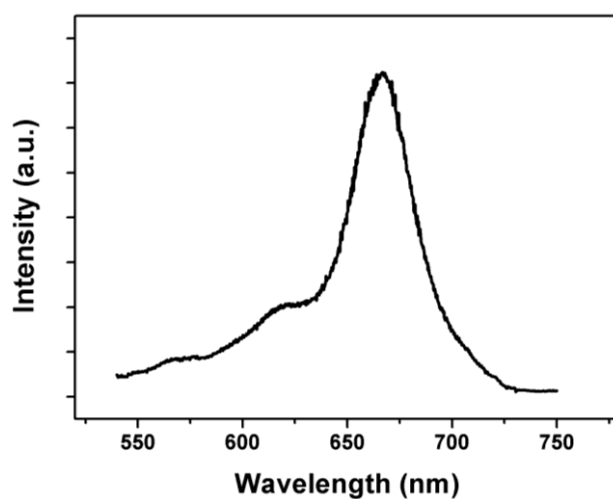
**Supplementary Figure 5** SEM images of the exfoliated (a) MoS<sub>2</sub>, (b) WS<sub>2</sub>, (c) TiS<sub>2</sub>, (d) TaS<sub>2</sub>, (e) BN, (f) NbSe<sub>2</sub> nanosheets. Note that TMDs nanosheets shows the darker contrast in comparison with SiO<sub>2</sub> substrate since the conductivity of TMDs nanosheet are better than SiO<sub>2</sub> substrate, while BN shows white or lighter contrast.



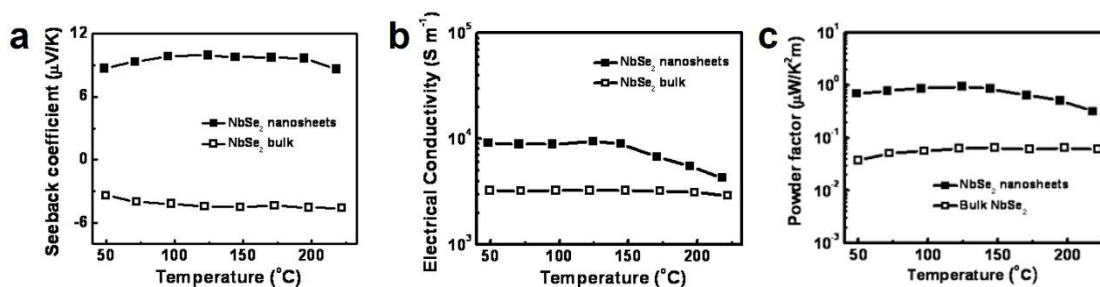
**Supplementary Figure 6** SEM images of the large area TMDs nanosheets deposited on Si/SiO<sub>2</sub> substrate: (a, b) MoS<sub>2</sub>, (c, d) TiS<sub>2</sub>, (e, f) TaS<sub>2</sub> nanosheets. Note that TMDs nanosheets shows the darker contrast in comparison with SiO<sub>2</sub> substrate since the conductivity of TMD nanosheets are better than SiO<sub>2</sub> substrate.



**Supplementary Figure 7** EDS of the exfoliated MoS<sub>2</sub> (a), WS<sub>2</sub> (b), TiS<sub>2</sub> (c), TaS<sub>2</sub> (d) nanosheets deposited on Si/SiO<sub>2</sub> substrate.



**Supplementary Figure 8** Photoluminescence spectrum of a single-layer MoS<sub>2</sub> nanosheet deposited on Si/SiO<sub>2</sub> substrate. After the nanosheet was annealed at 450 oC in Ar gas for 2 h, it was excited with 488 nm laser in air at room temperature to give the PL spectrum. Adapted with permission from ref. <sup>1</sup>.



**Supplementary Figure 9** Temperature-dependent Seebeck coefficient (a), electrical conductivity (b) and powder factor (c) for NbSe<sub>2</sub> nanosheet and bulk material. Adapted with permission from ref. <sup>2</sup>.

**Supplementary Table 1** The molecular weights of different layered compounds, and the corresponding discharge capacities as lithium-ion batteries with one lithium ion intercalated, i.e., LiMN.

Layered compound	Molecular weight (g·mol <sup>-1</sup> )	Capacity(1 Li) = LiMN (mAh·g <sup>-1</sup> )
MoS <sub>2</sub>	160	167.2
WS <sub>2</sub>	248	107.9
TiS <sub>2</sub>	112	238.9
TaS <sub>2</sub>	245	109.2
ZrS <sub>2</sub>	155	172.5
Graphite	12	2229.6
BN	25	1070.2
NbSe <sub>2</sub>	251	106.6

**Supplementary Table 2** Summary of the characteristics of the exfoliated 2D materials in this protocol.

Materials	MoS <sub>2</sub>	WS <sub>2</sub>	TiS <sub>2</sub>	TaS <sub>2</sub>	BN	NbSe <sub>2</sub>
Thickness (nm)	1.0 ± 0.2	1.0	1.6 ± 0.2	0.9 ± 0.1	4.0	2.4
Lateral dimensions (μm)	0.3-3.0	0.4-1.0	0.3-3.0	0.3-3.0	1.0	1.0
Yield	92%	-	93%	93%	-	-

## Supplementary References

- 1 Zeng, Z. *et al.* Single-Layer Semiconducting Nanosheets: High-Yield Preparation and Device Fabrication. *Angewandte Chemie International Edition* **50**, 11093-11097, doi:10.1002/anie.201106004 (2011).
- 2 Zeng, Z. *et al.* An Effective Method for the Fabrication of Few-Layer-Thick Inorganic Nanosheets. *Angewandte Chemie International Edition* **51**, 9052-9056, doi:<https://doi.org/10.1002/anie.201204208> (2012).
- 3 Zeng, Z., Tan, C., Huang, X., Bao, S. & Zhang, H. Growth of noble metal nanoparticles on single-layer TiS<sub>2</sub> and TaS<sub>2</sub> nanosheets for hydrogen evolution reaction. *Energy & Environmental Science* **7**, 797-803, doi:10.1039/C3EE42620C (2014).