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Electronic Supplementary Information

Synthesis of 3D free standing Crystalline $NiSe_x$ matrix for Electrochemical Energy Storage Application

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Calculation of the mass of working electrode material:

To calculate the mass of the working electrode, we have subtracted the initial weight of Ni foam from its final weight after growth. The increase in weight is due to the growth of nickel selenide and the Se precursor is only responsible for the extra weight after deposition. So from this extra weight, we have calculated the equivalent mole fraction of nickel selenide. And then the corresponding weight has been calculated which is the actual weight of nickel selenide to act in electrochemical performance. By following this method, the amount of deposited working nickel selenide material is 1.5 mg.

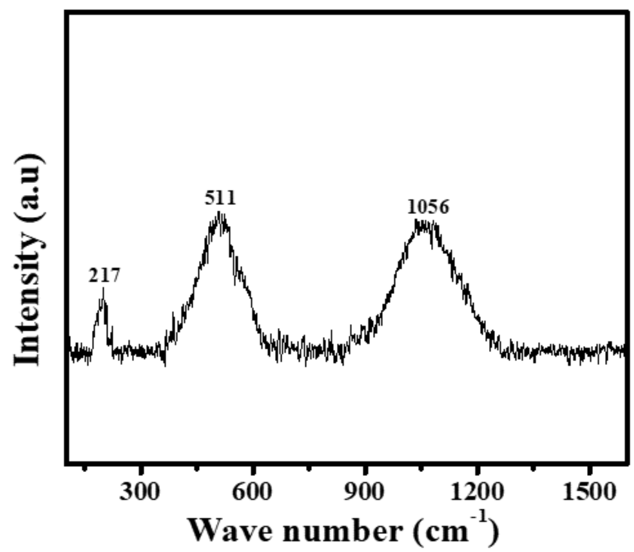

Fig. S1: The Raman spectrum of CVD grown polycrystalline $NiSe_x$ showing three characteristic peaks around 217, 511 and 1056 cm^{-1} corresponding to T_g mode of $NiSe_2$, longitudinal optical (LO) one-phonon mode and transverse optical (TO) two-phonon mod of NiSe respectively.

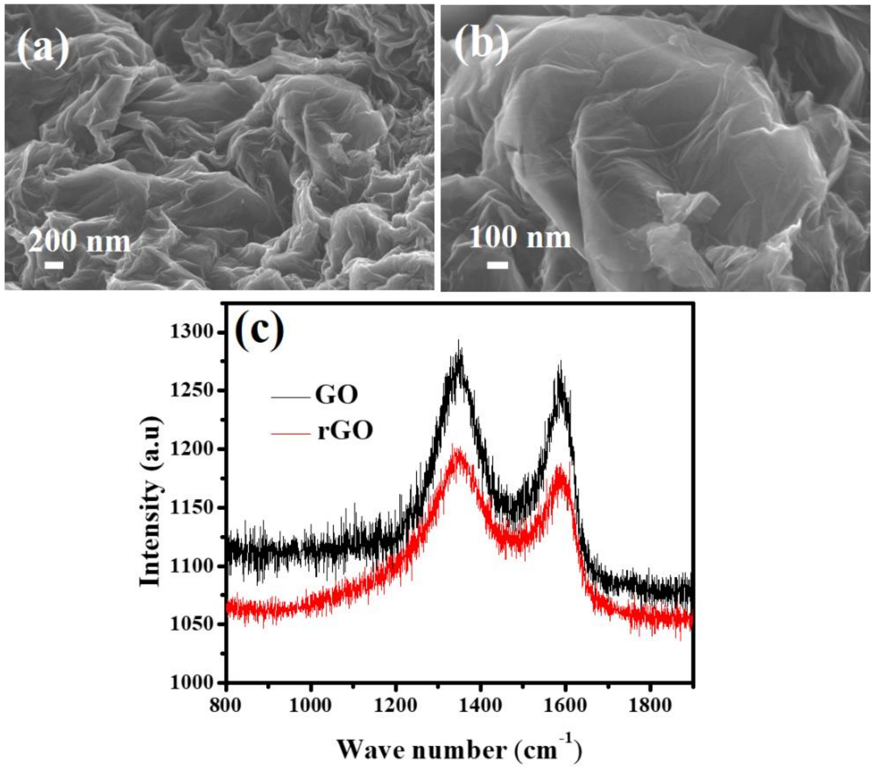

Fig. S2: (a-b) FESEM images of rGO at different magnifications and (c) The Raman spectra of GO and rGO.

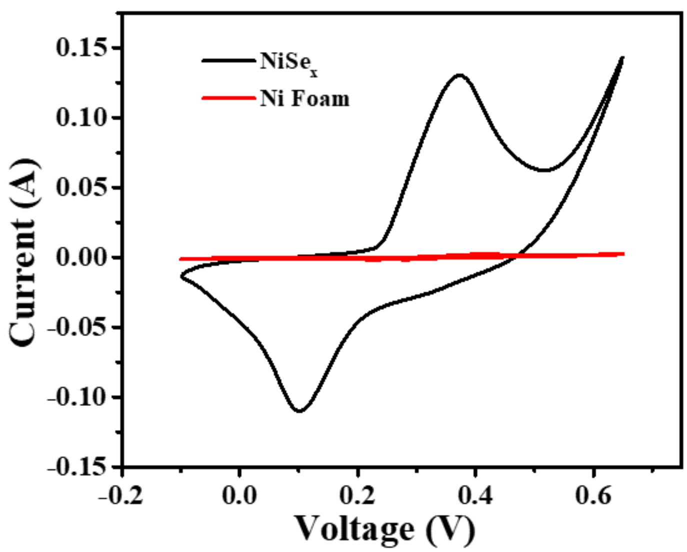

Fig. S3 Cyclic voltammogram of self-supported $NiSe_x$ nanocrystal and bare Ni foam electrode in three-electrode configuration at 100 mV/s sweep rate in 6.0 M KOH electrolyte (All potentials are recorded against Ag/AgCl reference electrode).

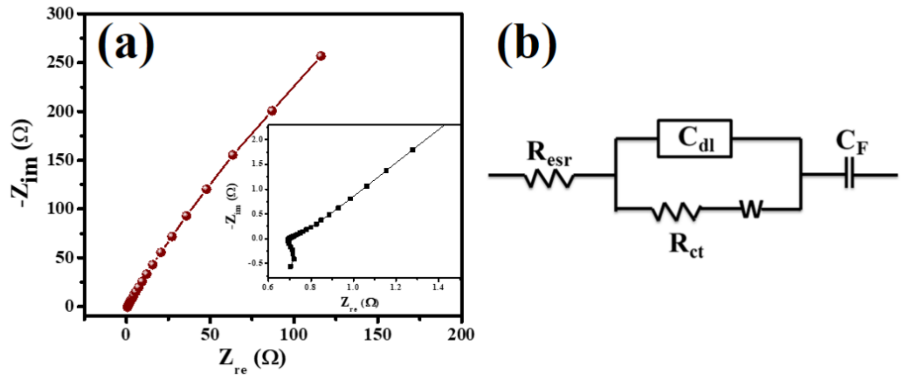

Fig. S4: (a) EIS spectra of self-supported $NiSe_x$ nanocrystal electrode: Nyquist impedance plot with the inset showing the magnified half circle regime and (b) the corresponding equivalent circuit fitted to the impedance characteristic.

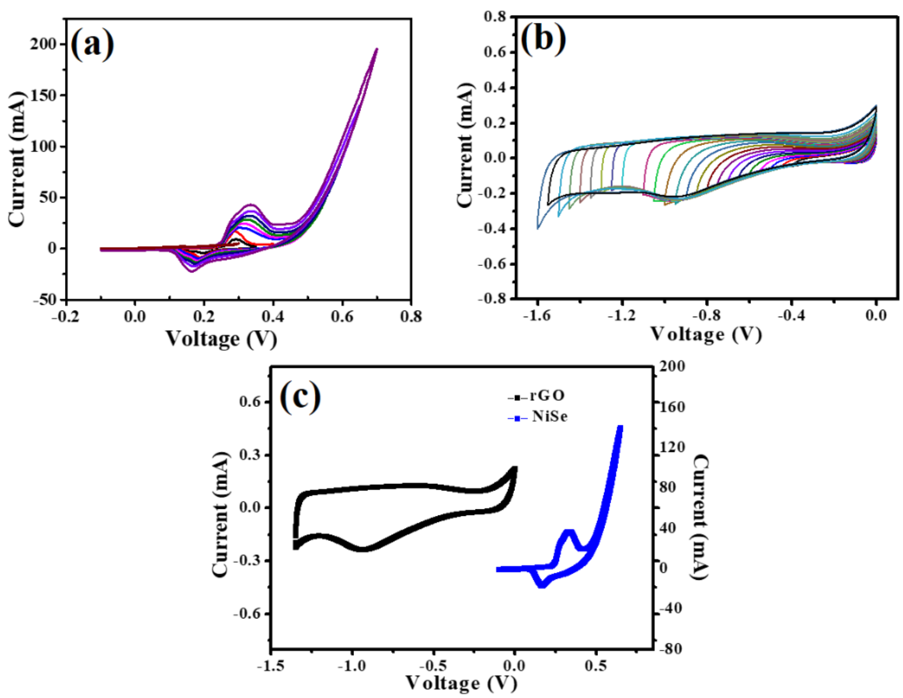

Fig. S5: Cyclic voltammogram at 10 mV/s sweep rate recorded with variable potential window and presented for (a) $NiSe_x$, (b) rGO and (c) both $NiSe_x$ and rGO in single frame (All potentials are recorded against Ag/AgCl reference electrode).

Table:S1 Comparison of electrochemical performance of self-supported 3D $NiSe_x$ nano crystalline electrode and other reported Ni_xSe_y based electrode materials

Electrode	Specific capacitance	Energy density	Power density	Capacitance retention	Measure-ment	References
$Ni_{0.85}Se$	1354 F g^{-1}	40.7 W h kg^{-1}	16 kW kg^{-1}	92.4% after 20000 cycles	Three electrodes	1
$NiSe_2$	92 F g^{-1}	32.7 Wh kg^{-1}	800 W kg^{-1}	86% after 2000 cycles	Two electrodes	2
NiSe nano-rod arrays	466 mF cm^{-2}	38.8 Wh kg^{-1}	13.5 kW kg^{-1}	90.09% after 3000 cycles	Two electrodes	3
Truncated cube-like $NiSe_2$ single crystals	104 F g^{-1}	44.8 Wh kg^{-1}	17.2 kW kg^{-1}	87.4.% after 20000 cycles	Two electrodes	4
Honey comb like $Ni_{0.85}Se$	196.65 F g^{-1}	65 Wh kg^{-1}	3200 W kg^{-1}	93.7 % after 10000 cycles	Two electrodes	5
Hexagonal NiSe/NiO nanostructure	83.5 F g^{-1}	721 mWh kg^{-1}	250 W kg^{-1}	94 % after 5000 cycles	Three electrodes	6
$Ni_{0.85}Se$/graphene microstructures	103.4 F g^{-1}	32.3 Wh kg^{-1}	1.5 KW kg^{-1}	90.6% after 10000 cycles	Two electrodes	7
Hexapod like $NiSe_2$ nanostructure	75 F g^{-1}	-	-	94% after 5000 cycles	Three electrodes	8
NiSe nano particles	-	22.3 Wh kg^{-1}	6.2 KW kg^{-1}	76% after 5000 cycles	Two electrodes	9
Layered $MoSe_2$ nanosheets	49.7 F g^{-1}	-	-	75% after 10000 cycles	Two electrodes	10
Bamboo-like $CoSe_2$ arrays	50.3 F g^{-1}	20.2 Wh kg^{-1}	1.4 KW kg^{-1}	93.3% after 2000 cycles	Two electrodes	11
Hollow NiSe-CoSe nanoparticles	-	41.8 Wh kg^{-1}	30 KW kg^{-1}	-	Three electrodes	12
WSe_2/rGO nanosheets hybrid	389 F g^{-1}	34.5 Wh kg^{-1}	4 KW kg^{-1}	98.7% after 3000 cycles	Three electrodes	13
3D cuboidal vanadium diselenide	680 F g^{-1}	212 Wh kg^{-1}	3.3 KW kg^{-1}	81% after 10000 cycles	Two electrodes	14

$NiSe_2$-NiSe nano-crystal	1333 F g^{-1}	105 Wh kg^{-1}	54 kW kg^{-1}	-	Three electrodes	This work
	40 F g^{-1}	22 Wh kg^{-1}	5.8 kW kg^{-1}	88 % after 10000 cycles	Two electrodes	

References:

1 S. Wang and S. Ma, *Dalt. Trans.*, 2019, **48**, 3906–3913.

2 Q. Bao, J. Wu, L. Fan, J. Ge, J. Dong, J. Jia, J. Zeng and J. Lin, *J. Energy Chem.*, 2017, **26**, 1252–1259.

3 Y. Tian, Y. Ruan, J. Zhang, Z. Yang, J. Jiang and C. Wang, *Electrochim. Acta*, 2017, **250**, 327–334.

4 S. Wang, W. Li, L. Xin, M. Wu, Y. Long, H. Huang and X. Lou, *Chem. Eng. J.*, 2017, **330**, 1334–1341.

5 L. Du, W. Du, H. Ren, N. Wang, Z. Yao, X. Shi, B. Zhang, J. Zai and X. Qian, *J. Mater. Chem.* A, 2017, **5**, 22527–22535

6 K. S. Bhat and H. S. Nagaraja, *Mater. Res. Express*, 2018, **5**, 105504.

7 S. Ma, S. Zhou, S. Wang and M. Liu, *J. Alloys Compd.*, 2017, **72**8, 592–599.

8 N. S. Arul and J. I. Han, *Mater. Lett.*, 2016, **181**, 345–349.

9 B. Yuan, W. Luan and S. T. Tu, *Cryst. Eng. Comm.*, 2012, ***14*(6)**, 2145-2151.

10 S. K. Balasingam, J. S. Leeb and Y. Jun, *Dalton Trans.*, 2015, **44**, 15491–15498.

11 T. Chen, S. Li, P. Gui, J. Wen, X. Fu and G. Fang, *Nanotechnology*, 2018, **29**, 205401.

12 H. Chen, M. Fan, C. Li, G. Tian, C. Lv, D. Chen, K. Shu and J. Jiang, *J. Power Sources*, 2016, **329**, 314-322.

13 C.V.M. Gopi, A.E. Reddy, J.S. Bak, I.H. Cho and H.J. Kim, *Mater. Lett.*, 2018, **223**, 57–60.

14 S. R. Marri, S. Ratha, C. S. Rout and J. N. Behera, *Chem. Commun.*, 2017, **53**, 228-231.