Supporting Information

Over-oxidized Mo_3Se_4 enriched with selenium: an anode for high performance Li-ion batteries

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Materials and methods

Materials

Sodium borohydride ($NaBH_4$) was purchased from Thomas Baker, Ammonium Molybdate (Para) Tetrahydrate [$(NH_4)_6Mo_7O_{24}.4H_2O$], Selenium Oxide (SeO_2) were purchased from Alfa Aesar Pvt. Ltd. All reagents were used without further purification.

Synthesis of Mo_3Se_4

In a typical synthesis, 0.05 mol of $(NH_4)_6Mo_7O_{24}.4H_2O$, 0.7 mol of SeO_2 and 0.05 mol of $NaBH_4$ powders were added step by step to 30 ml stirring double distilled water (DDW). The solution was stirred for 1 hr. Further 30 ml ethanol was added to the solution and stirred for another 30 min. The mixture was transferred to a 100 ml Teflon container. Hydrothermal autoclave reactor was heated at 200 °C for 24 h in a vertical muffle furnace. After the completion of hydrothermal treatment, obtained sediment was washed with DDW using 50 ml centrifuge tubes (3500/5min) for 3 times followed by single wash with ethanol. It was further vacuum dried at 80 °C for 24 h. Finally, black coloured powdered Mo_3Se_4 was obtained.

Materials Characterizations

The crystal structure, phase analysis and chemical composition of the obtained sample were investigated using XRD (Bruker Ltd. Germany, AXS D8 Advances), micro-RAMAN (Renishaw UK, INVIA0120-02) and X-ray photoelectron spectroscopy (XPS, Kratos AXIS Supraspectrometer). The morphological studies of the material were performed using a field emission scanning electron microscope (FESEM, TESCAN MIRA) and high-resolution transmission electron microscopy (HRTEM) (JEOL 2100).

Electrochemical measurements

All the electrodes were tested out using CR2032 coin cell assembly. The working electrodes were prepared by casting the slurry made from active material (60 wt.%), Super P (30 wt.%) and PVDF (Polyvinylidene fluoride) binder (10 wt.%) dispersed with drops of NMP (N-Methyl pyrrolidone) homogeneously. All chemicals purchased were of battery grade. The resultant slurry was coated on copper foil followed by vacuum drying at 90° C for 12 h. The average mass loading of active material alone on each electrode was ~ 0.25-0.26 mg/cm². CR2032 cells were assembled using coined copper foil with active material as anode, lithium foil as a counter electrode, electrolyte and glass microfiber separator in an argon-filled glove box with concentrations of moisture and oxygen below 0.5 ppm. The non-aqueous commercial electrolyte (Sigma) contained 1 M $LiPF_6$ (lithium hexafluorophosphate) dissolved in ethylene carbonate (EC), dimethyl carbonate (DMC) and ethyl

methyl carbonate (EMC) in 1:1:1 volumetric ratio. Galvanostatic charge–discharge tests were carried out using a Neware battery tester from 0.01 V to 3.0 V versus Li^+/Li. Cyclic voltammograms (CVs) were measured using a Biologic VMP-3e electrochemical workstation.
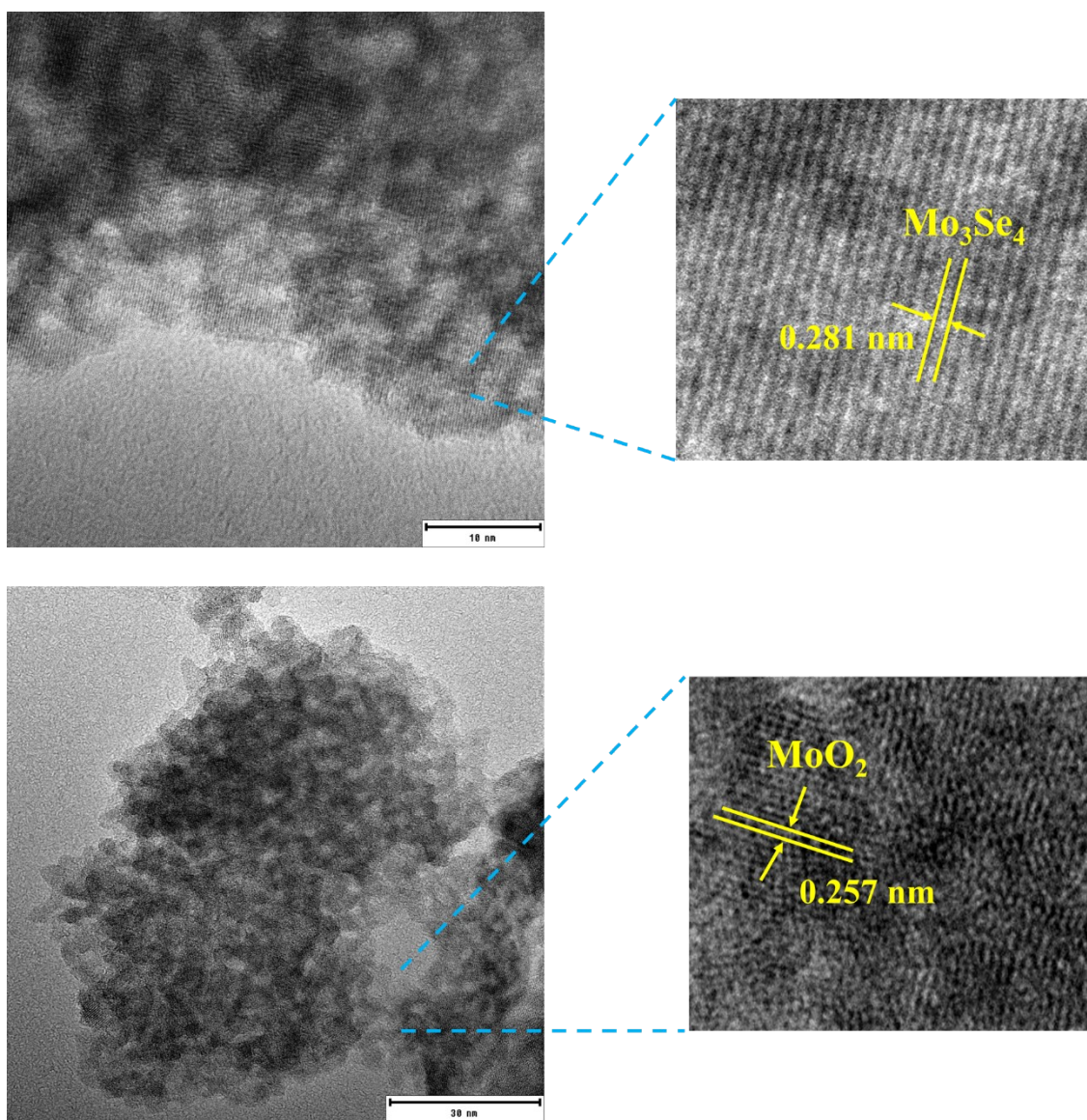

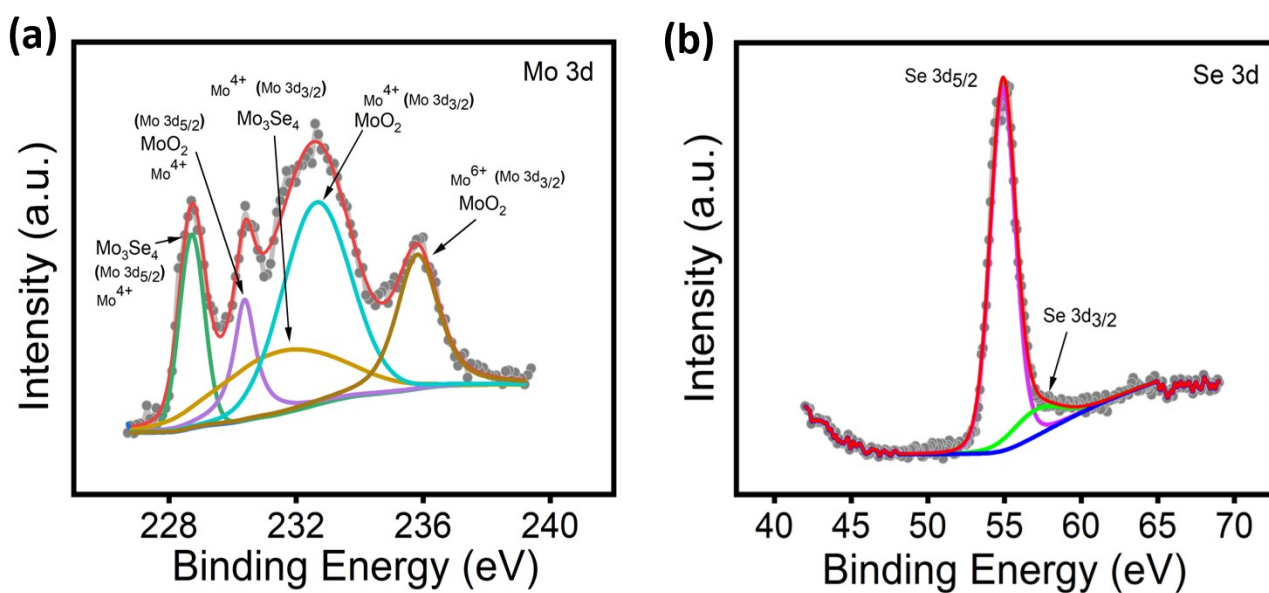**Fig. S1.** Enlarged magnification of TEM images of Mo_3Se_4.

Fig. S2. XPS spectra of Mo_3Se_4 (a) Mo 3d, (b) Se 3d core level spectra
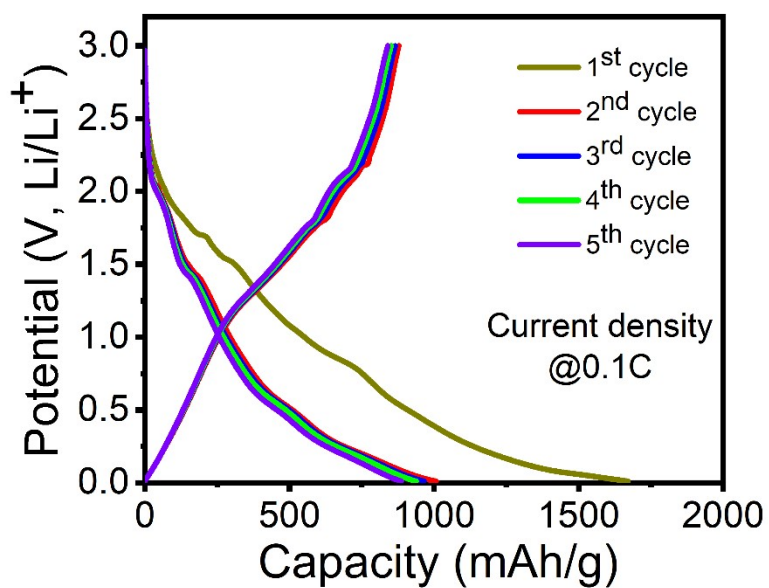

Fig. S3. Charge discharge profile of Mo_3Se_4 at 0.1C (where 1C equals to 670 mA/g).
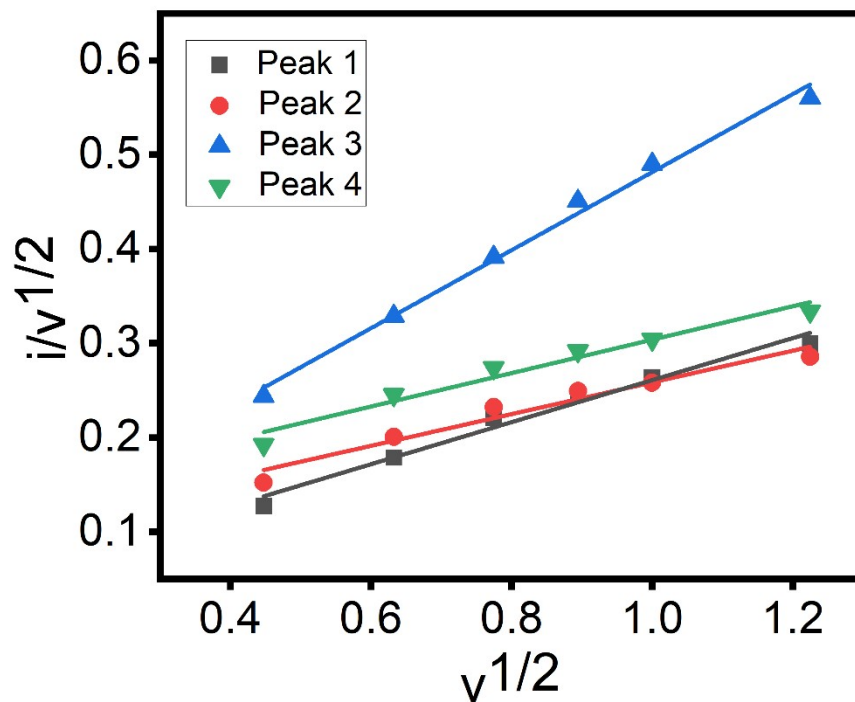
Fig. S4. Plot of $i/v^{1/2}$ vs. $v^{1/2}$

Table S1. Comparison of electrochemical performance of various electrodes as anode materials for LIBs.

Electrode	Technique	Current density (mA/g)	Reversible Capacity (mAh/g)	Cycle Number	Ref.
MoSe$_2$/MoO$_2$	Hydrothermal method	100	1042	100	[1]
MoSe$_2$	Hydrothermal method	100	581	100	[1]
CNTs@C@MoSe$_2$ @Se	Solvothermal method	5000 100	508 1010	500 100	[2]
CNTs@C@MoSe$_2$	Solvothermal	5000	415	500	[2]

	method				
1T-MoSe$_2$/ SWCNTs	Solvothermal method	300	971	100	[3]
MoSe$_2$@CC	Hydrothermal method	5000	638	1200	[4]
MoSe$_2$@N-C	Hydrothermal method	1000	1219	150	[5]
MoSe$_2$/C	Nano-casting technique	200	618	300	[6]
Mo-MoSe$_2$	Jet cavitation process	0.1C/ 0.2C	550	50	[7]
MoSe$_2$	Commercial powder	0.1C/ 0.2C	305	50	[7]
MoSe$_2$/rGO	Hydrothermal method	500 1000	917 750	100 100	[8]
MoO$_2$@MoSe$_2$	Hydrothermal method	2000	520.4	400	[9]
Mo$_3$Se$_4$	**Hydrothermal method**	**268**	**897.62**	**332**	**This work**

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