SUPPLEMENTARY INFORMATION

**Defect-free MoS2-flakes/amorphous-carbon Hybrid**

**as Advanced Anode for Lithium-ion Batteries**

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**S1. Raman analysis of bulk and exfoliated MoS2**

Raman spectroscopy measurements, reported in Figure 1c of the main text, are carried out to investigate the vibrational modes of exfoliated MoS2 with respect to that of bulk MoS2. Figure S1 shows the statistical Raman analysis of the peaks position difference of the A1g(Γ) and E2g1(Γ) modes, *i.e.* Pos(A1g) - Pos(E2g1), full width at half maximum values of the A1g and E2g1 (FWHM(E2g1) and FWHM(A1g)) for exfoliated MoS2 (Fig. 1a, b and c, respectively), and bulk MoS2 (Fig. 1d, e and f, respectively).

According to the statistical Raman analysis shown in Figure 3a and d, the peaks position difference values between the A1g and E2g1 modes, *i.e.* Pos(A1g) - Pos(E2g1) are ~ 26 cm-1 for bulk MoS2 and ~ 25 cm-1 for exfoliated MoS2, respectively. The decrease in peaks position difference value of exfoliated MoS2 with respect to bulk MoS2 indicates the decrease in the number of MoS2 layers after the exfoliation process. 1-3 Also, both FWHM(A1g) and FWHM(E2g1) of exfoliated MoS2 flakes increase ~ 1 cm-1 with respect to the corresponding modes of bulk MoS2, see Figure 3b, c, e and f. The increase of FWHM(E2g1) and FWHM(A1g) for exfoliated MoS2 is attributed to the variation of interlayer force constants between the inner and outer layers. 3



**Figure S1.** Statistical Raman analysis of (a), (d) Pos(A1g) - Pos(E12g); (b), (e) FWHM(E2g1) and (c), (f) FWHM(A1g) for bulk and exfoliated MoS2

**S2. Optical absorption spectroscopy of exfoliated MoS2**

The exfoliated MoS2 flakes are studied by optical absorption spectroscopy (OAS). The optical absorption spectra are acquired with a Cary Varian 5000UV-Vis spectrophotometer with 1 nm resolution. Measurements are carried out in the 250-800 nm range, limited by the strong absorption features of 2-propanol (IPA) (cut-off wavelength 240 nm). The absorption spectra were acquired using a 1 mL quartz glass cuvette. Briefly, the UV–vis spectrum of the exfoliated MoS2 exhibits the typical excitonic peaks at: (A) 660 nm and (B) 604 nm, ascribed to excitons associated with the direct gap transition at the K point of the Brillouin zone in 2D MoS2 4-6; (C) 441 nm and (D) 383 nm due to excitonic transitions from the valence band to the conduction band. 5-7 The concentration of MoS2 flakes in IPA can be estimated to be 0.03 g L-1 from the optical absorption coefficient at 672 nm, using A = αlc where l (m) is the light path length, c (g L−1) is the concentration of the dispersed flakes, and α (L g−1 m−1) is the absorption coefficient, with α ≈ 3400 L g−1 m−1 at 672 nm.8



**Figure S2.** Absorption spectra of exfoliated MoS2

**S3. Cyclic voltammogram MoS2 and MoS2/C electrodes**

The cyclic voltammetry measurement is carried out with the scan rate of 0.5 mVs-1 over the potential range from 3.00 to 0.005 V vs Li/Li+. Figure S3 shows the 1st, 2nd and 3rd CV curves of MoS2 electrode. In the 1st cycle, the first reduction peaks at ~1.03 V links to the formation of LixMoS2 by the intercalation of Li+ into MoS2 layers. The small reduction peak at ~0.70 V is attributed to the decomposition of electrolyte, resulting in the formation of solid electrolyte interface (SEI) on surface MoS2 electrode. 9 The reduction peak at ~ 0.46 V associated with the formation of Li2S and metallic Mo nanoparticles via a conversion reaction of LixMoS2. 9-11 The pronounced oxidation peak at ~ 2.32 V, is attributed to oxidation of Li2S into Li+ and sulphur. 9-11 Meanwhile, the broad and weak oxidation peak at ~ 1.63 V, indicates the partial oxidation of metallic Mo to form MoS2. 12, 13 In the 2nd cycle, a new reduction peak at ~ 1.90 V is observed, which corresponds to the reduction of sulphur to form Li2S. Besides, the oxidation peak is observed at ~ 2.32 V, attributed to oxidation of Li2S. The two reduction and oxidation peaks in 2nd cycle constitute a reversible redox couple. 9-11 From the 2nd cycle, the electrochemical mechanism of MoS2 is mainly dominated by the reversible conversionreaction of sulphur to Li2S.14 In general, the two redox processes can be summarized according to the following reactions: 9

MoS2 + *x*Li+ → Li*x*MoS2 (1)

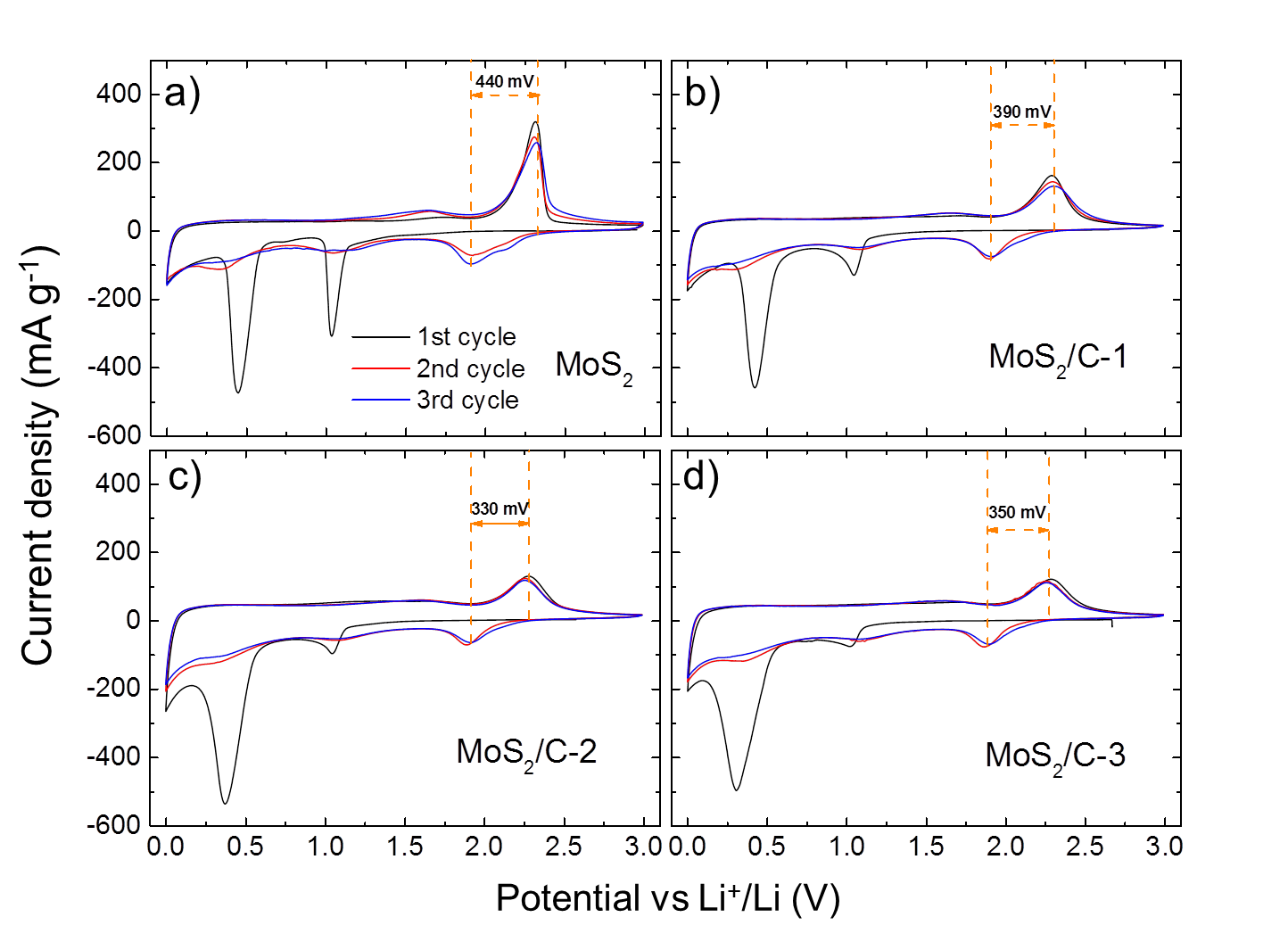
Li*x*MoS2 + (4-*x*)Li+ + (4-*x*)e- → Mo + 2Li2S (2)

Li2S → S + 2Li + 2e- (3)

S + 2Li + 2e- → Li2S (4)

However, the intensities of two reduction peaks (at ~ 1.03 and ~ 0.45 V) drastically decrease in the 2nd and 3rd cycles because of the consumption of residual MoS2, which is not completely reduced during the 1st cycle. From 3rd cycle, the intensity of reduction peak at ~ 1.90 V is slightly increased (24.9 mA g-1) with respect to the 2nd cycle, which is attributed to an activation process of the electrode materials. 15 Importantly, in the case of MoS2 and MoS2/C-1 electrodes, it is observed that the intensities of oxidation peaks at ~ 2.32 V significantly decrease with the increasing of cycle number. In specific, the intensity differences of these peaks between 1st and 2nd cycles are 45 mA g-1 and 18 mA g-1 for MoS2 and MoS2/C-1 electrodes, respectively. These CV behaviours suggest that the in the case of MoS2 electrode, irreversible electrochemical processes take place due to irreversible redox reactions during lithiation/de-lithiation (equations 3 and 4).12 These irreversible redox reactions progressively produce insulated sulfur (S) (see Figure S6) which can hinder the electron transport of MoS2 electrode during cycling, resulting in its irreversible electrochemical processes 16. These irreversible electrochemical processes are alleviated in the case of MoS2/C-1 electrode due to the support of carbon network. However, the irreversible electrochemical processes are still taking place because the insufficient carbon content cannot completely cover all MoS2 flakes in the case of MoS2/C-1 electrode (see HR-TEM images, Figure 2d), which still can not completely improves the electron transport within the electrode.

For MoS2/C-2 and MoS2/C-3 electrodes (Figure S3c and d), the intensities of oxidation peaks at ~ 2.32 V in the initial three cycles are overlapped. The MoS2/C-2 and MoS2/C-3 electrodes exhibit a reversible electrochemical process, in contrast to MoS2 and MoS2/C-1 electrodes which do not present this process, indicating that the sufficient carbon content improves the electrical conductivity of MoS2. It is noteworthy to mention that the potential differences between redox peaks at ~ 1.92 and 2.32 V, after 3 cycles, is 440, 390, 330 and 350 mV, for MoS2, MoS2/C-1, MoS2/C-2 and MoS2/C-3 electrodes, respectively. This fact confirms that MoS2/C electrodes have lower overall resistance and better electrochemical reversibility than in the MoS2 electrode. 17, 18 As the carbon content increase, a widening of the cathodic peak set at ~0.46 V Li+/Li, together with a slight reduction of the peak current, is observed. This modification finds its explanation in kinetics limitation, *i.e.* lithium diffusion through carbon thickness, of Li*x*MoS2 – Li2S conversion buffered by carbon network.

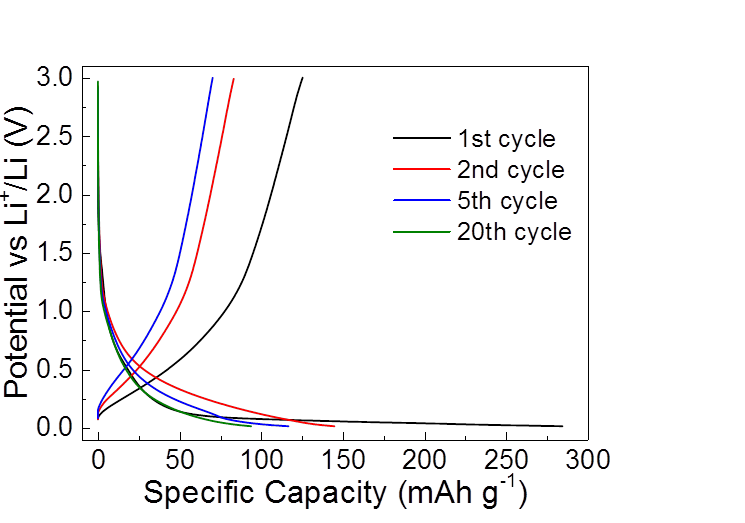


**Figure S3.** Cyclic voltammograms of (a) MoS2, (b) MoS2/C-1, (c) MoS2/C-2 and (d) MoS2/C-3 electrodes at a scan rate of 50 µVs-1.

**S4. Electrochemical properties of amorphous carbon**

The amorphous carbon is produced by the thermal decomposition of only PAA; the annealing profile of amorphous carbon is same as that of MoS2/C composites see Experimental in the main text. The electrode slurries are prepared by dispersing amorphous carbon powder (active material), carbon black, and PAA with a weight ratio of 8:1:1 in IPA. The slurry is pasted onto copper disks with diameter of 1.5 cm. The disk is then dried in an oven (BÜCHI, B-585) at 60° C and 10-3 bar pressure for 12 hours. The amorphous carbon electrode is tested in half-cell configurations against Li foils (Sigma-Aldrich) as the counter and reference electrodes (two electrodes system). The assembling of half-cell follows the same procedure as MoS2 and MoS2/C based half-cells, see Experimental in the main text.

Galvanostatic charge/discharge cycling measurements are conducted at a current density of 100 mA g-1 over the potential range from 3.00 to 0.005 V vs Li/Li+ in order to fully investigate electrochemical response for the Li+ storage of the amorphous carbon electrodes. As shown in Figure S4, the voltage profile of amorphous carbon electrode shows the specific capacity of ~ 285 mAh g-1 in the initial charge process (lithiation). From subsequent charge processes, the electrode exhibits the continuously capacity fading of ~ 145, 117 and 94 mAh g-1 corresponding to the 2nd, 5th and 20th cycles, respectively. The capacity fading during cycling suggests the irreversible Li+ storage process of amorphous carbon electrode. This can be attributed to the presence of active sites, *e.g.*, defects in amorphous carbon. 19-21 The irreversible Li+ storage in amorphous carbon also contributes to the irreversible capacities of MoS2/Celectrode (see Figure 4 in the main text)



**Figure S4.** Voltage profiles upon galvanostic charge/discharge of amorphous carbon

**S5. Electrochemical properties of bulk MoS2 and carbon-coated bulk MoS2 (bulk MoS2/C) electrodes**

The preparation of bulk MoS2/C powder with the weight ratio of bulk MoS2:PAA=1:1, is same as the preparation of MoS2/C composites, which has been reported in experimental session (see the main text). The electrode slurries are prepared by homogeneously dispersing bulk MoS2 bulk MoS2/C powders (active material), carbon black, and PAA with a weight ratio of 8:1:1 in IPA. The slurries are spread onto copper disks with diameter of 1.5 cm and then dried in an oven at 60° C and 10-3 bar pressure for 12 hours in oven (BÜCHI, B-585) to remove residual IPA. The mass loading of active materials (~1.0 mg) for each anode is calculated by subtracting the weight of bare Cu disks (using an analytical balance of Mettler Toledo XSE104) from the total weight of the electrode. The cycling performance of bulk MoS2, bulk MoS2/C electrodes at a current density of 100 mA g− 1 are shown in Figure S3, together with their Coulombic eﬃciencies. The bulk MoS2 electrode delivers fast capacity fading after 100 cycles. This is mainly due to the volume change of bulk MoS2 (black plots) during lithiation/de-lithiation, leading the pulverization of electrode. 22-24 The capacity fading still appeared in the case of bulk MoS2/C electrode (orange plots), however, it is less serious than that of bulk MoS2 electrode. It suggests the role of carbon as buffer layer to limit the volume change of bulk MoS2. However, the micro-size of bulk MoS2 coupling with the insufficient of carbon content is a reason of capacity fading in bulk MoS2/C electrode.



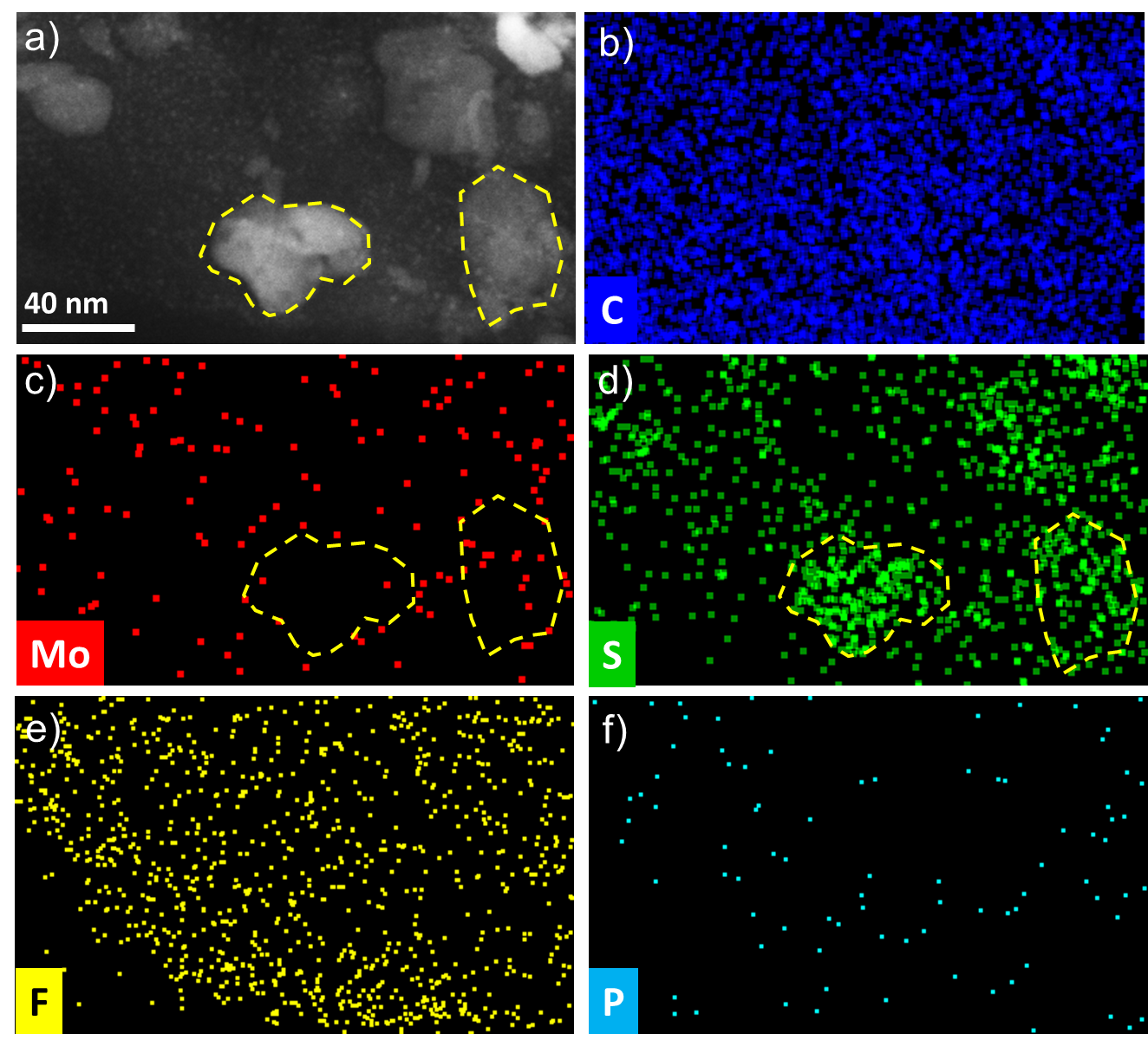
**Figure S5.** Specific capacity and Coulombic efficiency over charge/discharge galvanostatic cycles at 100 mA g-1 of bulk MoS2 and bulk MoS2/C electrodes

**S6. Elemental maps using scanning TEM (STEM)-energetic dispersive spectroscopy (EDS) of pristine MoS2 electrode after cycling**

Elemental mapping of materials from pristine MoS2 electrode after 100 charge/discharge cycles is carried out by STEM-EDS measurements. Figure S6a shows a high angular annular dark field (HAADF)-STEM image of MoS2 electrode materials, for which the corresponding elemental maps are demonstrated in Figure S6b-f. Carbon originates from the binder (PAA) and conductive agent (carbon black super-P). S is distributed in the particles labelled by the yellow dashed lines, while no Mo is detected in these particles. The presence of S alone confirms these particles are not MoS2, but insulating S or Li2S which can hinders the electron transport, leading to irreversible electrochemical processes of MoS2 electrode (see Figure S3a). 9, 15, 16

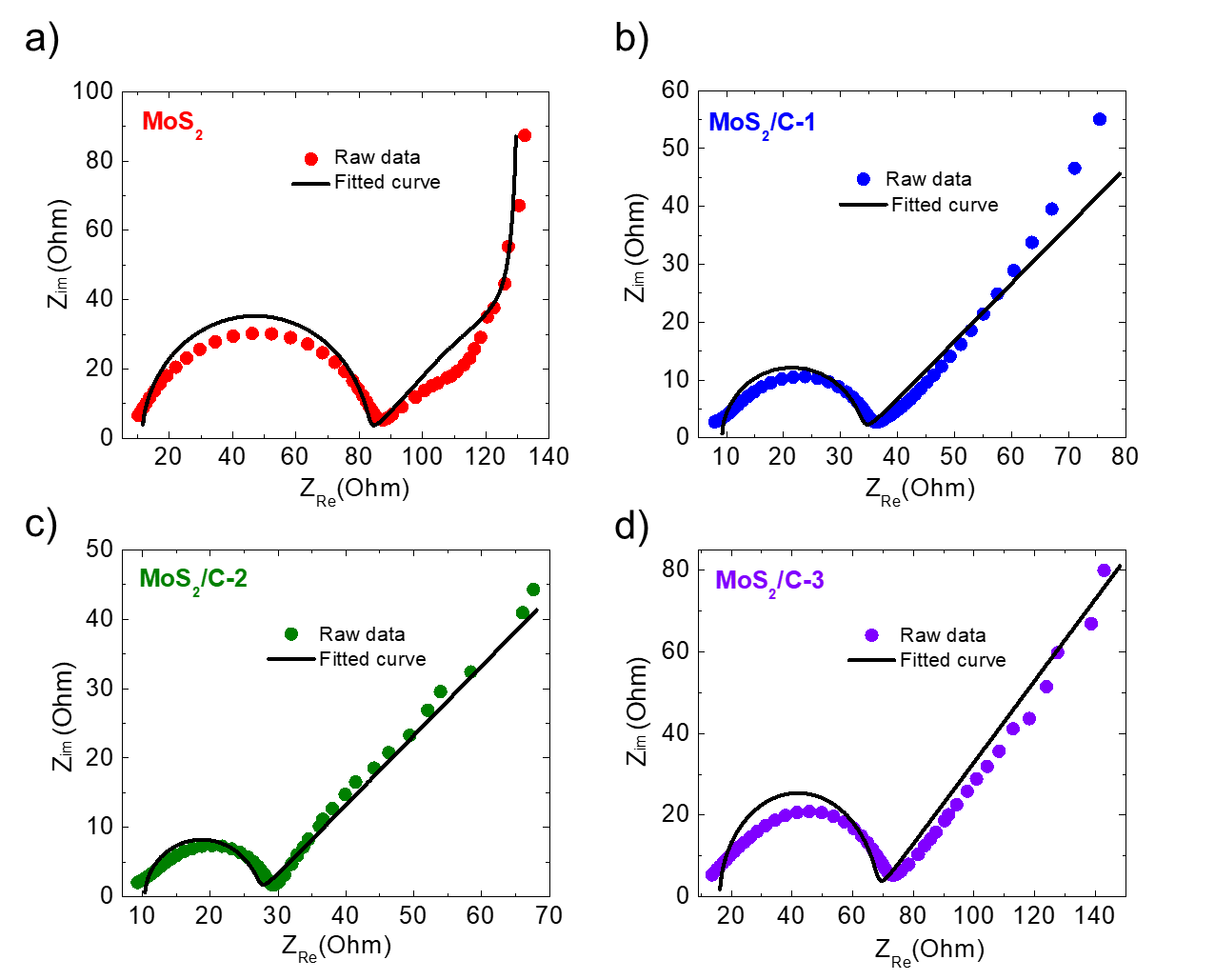
The distribution of fluorine (F) and phosphor (P) on the electrode can be used to identify the formation of SEI. The components of SEI are always contributed from the reduction and decomposition of the electrolyte. In this work, the LiPF6 is used as the Li salt (see the Experimental in main text), which decomposes into LiF (s) and a small portion of PF5 (s) through the reactions (5) (see the equation below). 25, 26 The presence of F confirms the formation of LiF, while P is barely visible probably due to small amount of PF5 in the observed area.

LPF6 (solv) → LiF (s) + PF5 (s) (5)



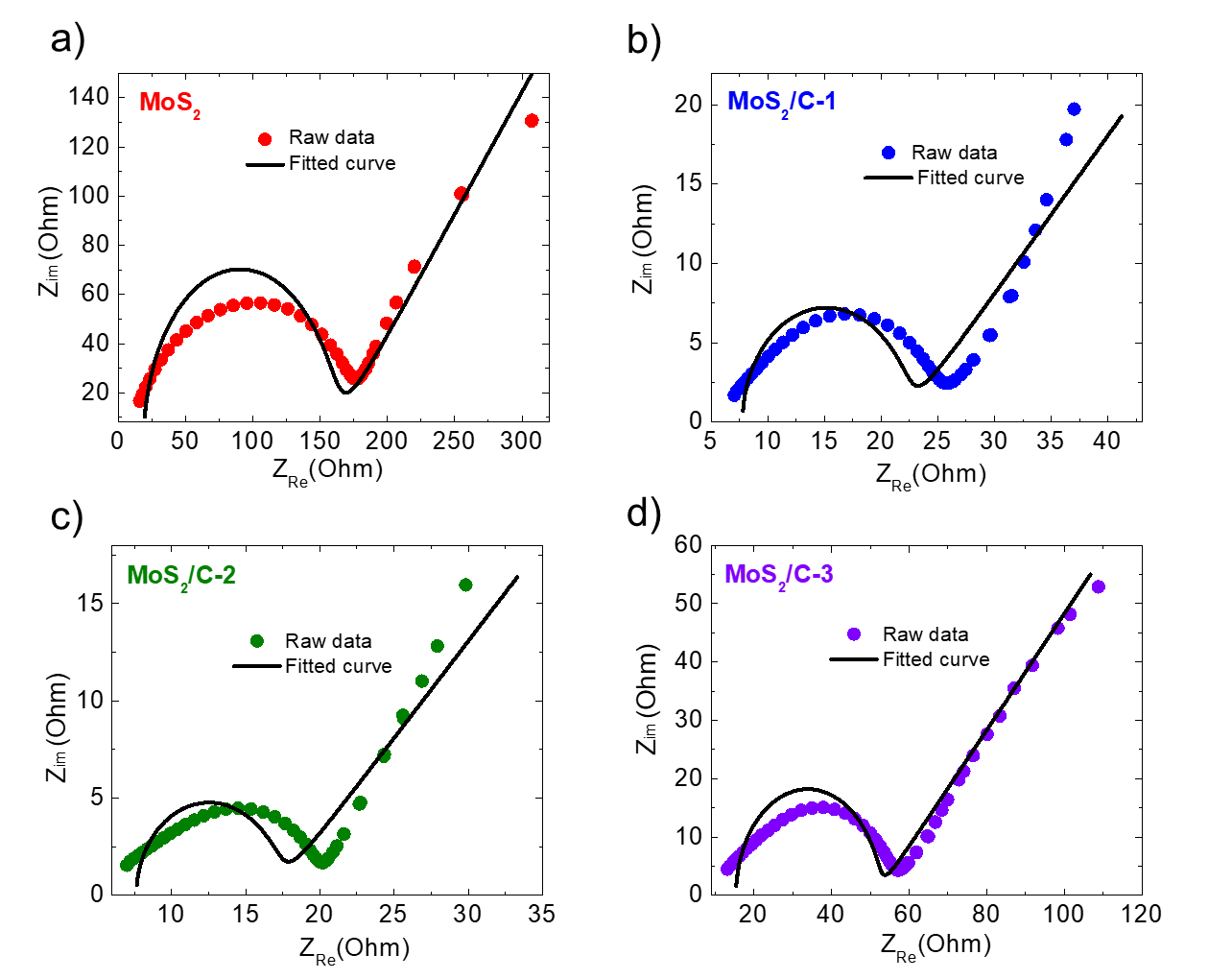
**Figure S6.** (a) HAADF-STEM image of MoS2 electrode after 100 charge/discharge galvanostatic cycles and elemental maps of (b) C, (c) Mo, (d) S, (e) F and (f) P acquired by STEM-EDS.

**S7.** **Fitting of electrochemical impedance spectra of MoS2 and MoS2/C electrodes after first cycle, at charged state.**

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**Figure S7.** Fitting of Nyquist plots of MoS2 (a), MoS2/C-1 (b), MoS2/C-2 (c), MoS2/C-3 (d) electrodes after first cycle, at charged state.

**S8. Fitting of electrochemical impedance spectra of MoS2 and MoS2/C samples after 100th cycle, at charged state**

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**Figure S8.** Fitting of Nyquist plots of MoS2 (a), MoS2/C-1 (b), MoS2/C-2 (c), MoS2/C-3 (d) electrodes after 100th cycle, at charged state.

The nyquist plots of MoS2 and MoS2/C electrodes after 1st cycle and after 100th cycle, at charged state are fitted via EC-Lab software, powered by Bio-Logic Inc. According to the fitting results, the nyquist plots of all electrodes consist of a depressed semi-circle in the high frequency region and a sloping straight line in the low-frequency region. The semi-circles are described by means of a generalized RC-circuit (the inset in Figure 4a) with electrolyte resistance RE (resistance caused by the mass transport of Li+ through electrolyte), charge transfer resistance (RCT) (including the electron transfer to the reactive centre (MoS2), and a constant phase element (CPE) for the electrode/electrolyte interface. The high frequency region is associated with the RCT, whereas the low-frequency line is descriptive of the Li+ diﬀusion impedance within electrode which is described by the Warburg circuit element (ZW ) in RC-circuit.

**S9. Thermogravimetric analysis of MoS2 and MoS2/C composites**

As shown in Figure 2a in the main text, the derivative weight curve of MoS2 sample (red dash-curve) shows the first weight loss mainly occurs from ~ 350oC to 470oC, which is attributed to oxidation of MoS2 to MoO3, 11 and the weight loss of sulfur is calculated ~ 11.2 wt%. As presented in table 1, the weight loss of sulfur and carbon is estimated approximately ~ 21.3, 27.3 and 41.1 wt% for MoS2/C-1, MoS2/C-2, MoS2/C-3 sample, respectively. The weight of carbon in each MoS2/C samples are calculated by the subtracting the weight losses of sulfur and carbon (wt%) to weight loss of sulfur (11.2 wt%). As a result, the carbon contents in MoS2/C-1, MoS2/C-2, and MoS2/C-3 samples are estimated to be ~ 10.1, 16.1 and 29.9 wt%.

**Table S1.** Calculation of wt% of carbon based on TGA measurement.

|  |  |  |  |
| --- | --- | --- | --- |
| Samples | Wt% of  MoS2 : PAA | Weight loss of Sulfur  and Carbon (%) | Weight of carbon  (%) |
| MoS2/C-1 | 1 : 1 | 21.22 | **10.00** |
| MoS2/C-2 | 1 : 2 | 27.33 | **16.11** |
| MoS2/C-3 | 1 : 4 | 41.08 | **29.86** |

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