Supplementary Material (ESI) for Chemical Communications


# Supporting information

## $MoS_2$ Hierarchical Hollow Cubic Cages Assembled by Bilayers: One-step Synthesis and Their Electrochemical Hydrogen Storage Properties

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### S1. FESEM study on the intermediate $K_2NaMoO_3F_3$

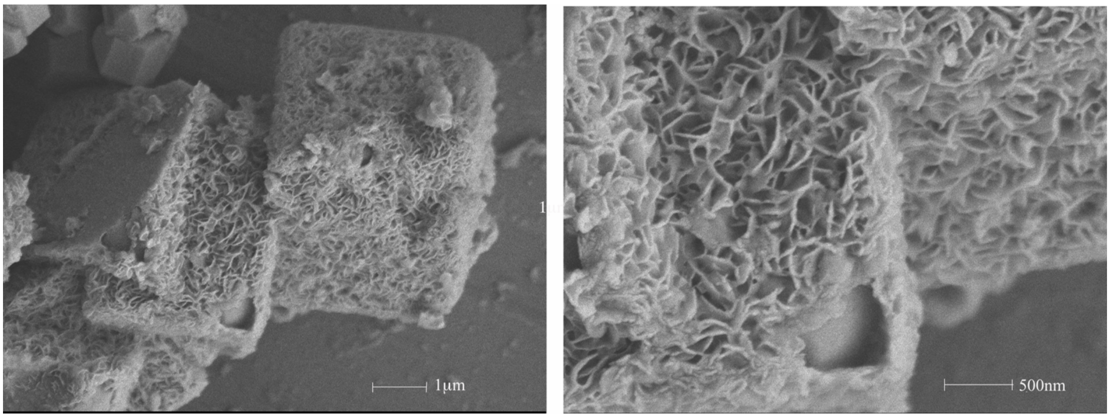


**Figure S1:** low magnification (left) and high magnification (right) FESEM images of a broken sample collected at 220°C for 2.5 h with other experiment conditions unchanged. We can see that a thin loose $MoS_2$ shell made up of many upstanding nanoplates is clearly covered on the solid $K_2NaMoO_3F_3$ core. Combined with the XRD study (Figure 1B in the text), it can be affirmed that the intermediate $K_2NaMoO_3F_3$ really formed and subsequently sacrificed as the template.

## S2. The formation of the bilayer structure

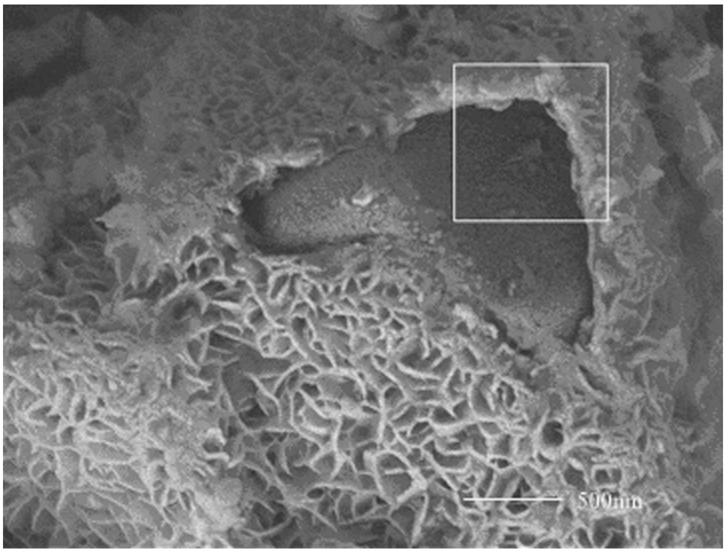


**Figure S2:** FESEM image of a cracked sample collected at 220°C for 2.5 h with other experiment conditions unchanged, from which we can see that the inner layer began to grow on the internal surface of the preformed $MoS_2$ shell, as shown in the box above.

## S3. $MoS_2$ products obtained without using NaF or with high-concentration NaF starting solution

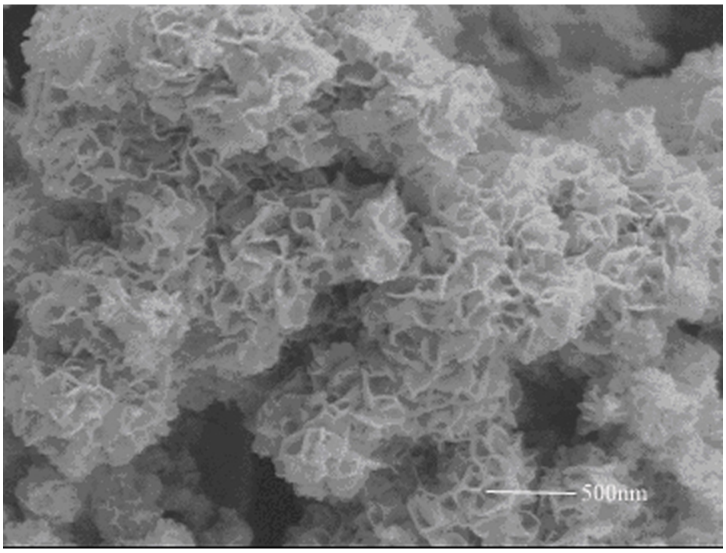


**Figure S3-1:** FESEM image of flower-like $MoS_2$ assembled by nanoplates obtained at 220°C for 24 h without using initial NaF, showing that introduction of NaF to the starting solution is a prerequisite to form cubic $K_2NaMoO_3F_3$.

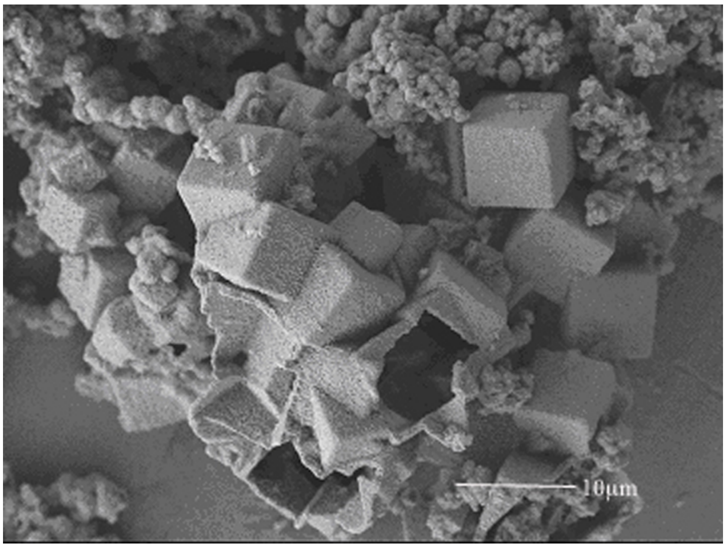


**Figure S3-2:** FESEM image of the larger $MoS_2$ cubic cages obtained at 220°C for 24 h with high-concentration of initial NaF (0.030mol). Some broken cages confirmed their hollow nature and indicated that the larger $MoS_2$ cubic cages are easier to collapse.

## S4. Charge-discharge hydrogen storage capacity of the $MoS_2$ electrode at both charge-discharge current of 50 mA/g and 200mA/g

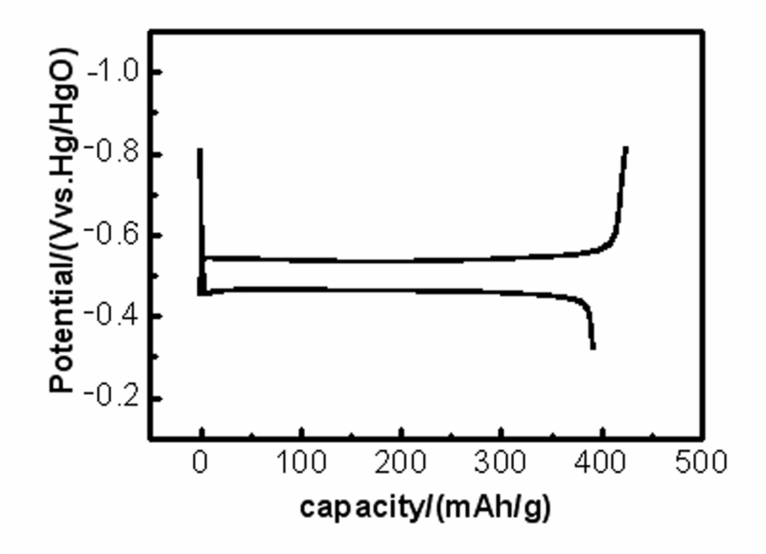


**Figure S4-1** The charge-discharge hydrogen storage curves at a given constant current density of 50mA/g showed an electrochemical charging capacity of 375 mAh/g of the as-prepared $MoS_2$ cubic cages electrode.

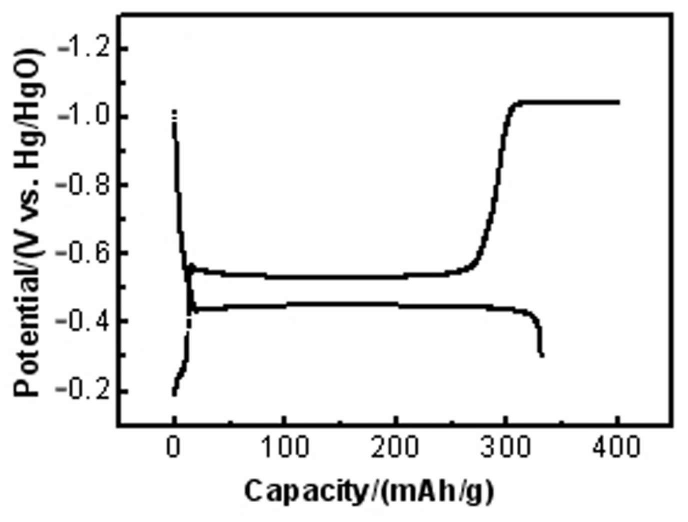


**Figure S4-2:** The charge-discharge hydrogen storage curve at a constant current density of 200 mA/g showed an electrochemical charging capacity of 324 mAh/g of the as-prepared $MoS_2$ cubic cages, indicating that higher current density resulted in lower electrochemical charging capacity.