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Critical Review

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MOF-Derived Materials Enabled Lithiophilic 3D Hosts for Lithium Metal Anode — A Review

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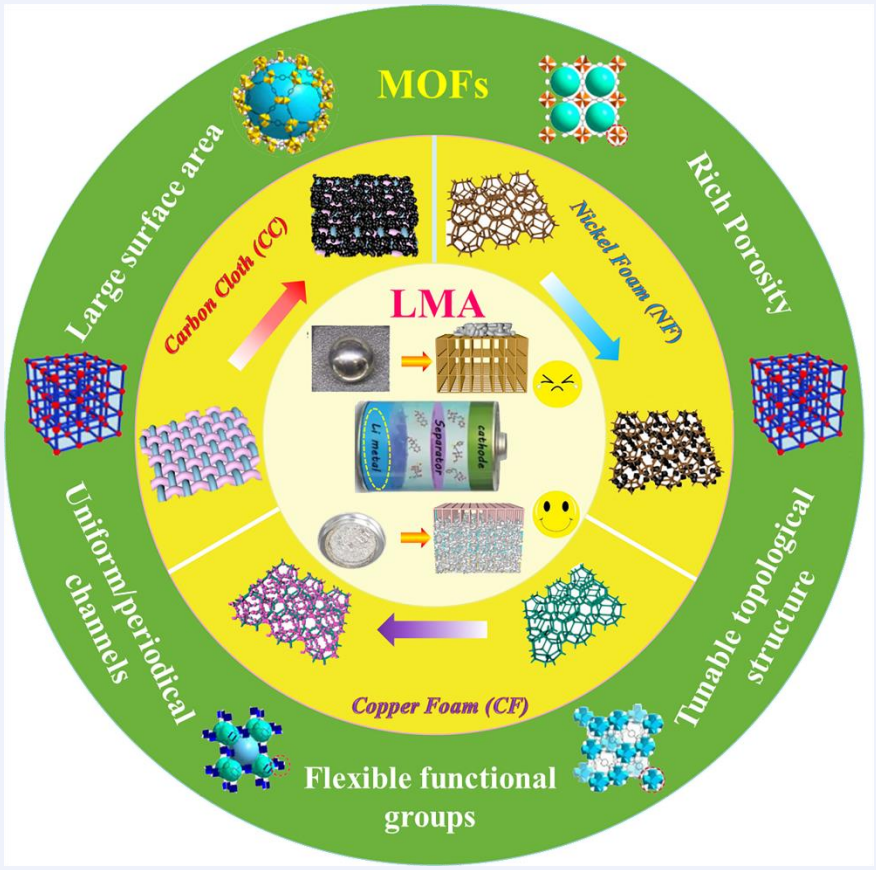
Keywords

Metal-organic frameworks (MOFs) | Lithium metal anode (LMA) | Lithiophilic materials | Three-dimensional (3D) host | MOF-derived materials | Interfacial modification | Lithium-metal batteries | Electrochemistry

Comprehensive Summary



This work systematically reviews recent progresses in the applications of MOF-derived materials modified 3D porous conductive framework as hosts for uniform lithium deposition in LMBs. A series of commonly used lithiophilic materials and several kinds of representative MOF-derivation-modified 3D hosts as lithium metal anode (LMA) are presented. Finally, the challenges and future development of employing MOF-derived materials to modify the 3D porous conductive framework for LMA are included.

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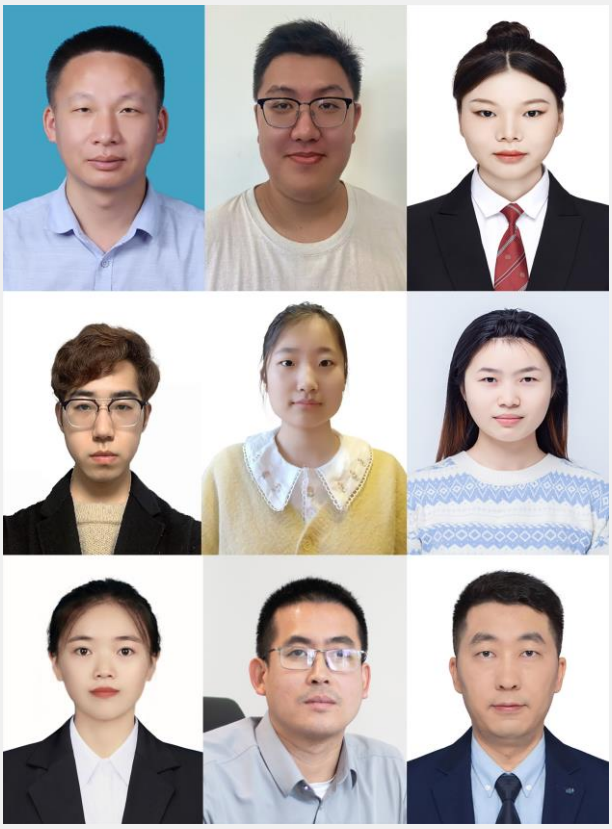

Tao Wei (top left) received his B.S. degree in 2009 and Ph.D. degree in 2015 from China University of Mining and Technology and then he carried out postdoctoral studies with Prof. Ya-Qian Lan in Inorganic Chemistry at Nanjing Normal University. Now he is employed as an associate professor by Jiangsu University of Science and Technology. His current research is mainly focused on lithium-based energy storage materials, especially solid electrolyte and Li metal anodes.

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Contents

1. Introduction

As the fossil fuels are consumed at an increasing rapid pace throughout the globe, environmental pollution and the energy crisis are becoming worse. In the 21st century, these problems have continuously promoted people to a search for clean and effective energy conversion technologies.[1-6] Lithium-ion batteries are recognized as a significant advancement in the field of energy storage technology and have a significant influence on contemporary civilization.[7-14] However, the growing demand for higher energy densities cannot be met by the current battery configurations with graphite as the anode. In recent years, due to its ultrahigh specific capacity (3860 mAh/g) and the lowest electrochemical potential (–3.04 V vs. H^+/H_2), metallic lithium (Li) has been dubbed as the "Holy Grail" anode for lithium metal batteries (LMBs).[15-19] However, lithium metal anode (LMA) has many notorious problems in the process of charging/discharging due to the intrinsic properties of itself.[20-22] Among them, dendrite formation upon repetitive Li plating/stripping and infinite relative volume change during repeating cycling are currently the two main issues facing the development of Li metal anode.[23-26] In more detail, the inhomogeneous deposition of Li^+ can induce the formation of Li dendrites that can even penetrate the separators, posing a major threat to safety. The endless relative volume change causes the solid electrolyte interphase (SEI) to repeatedly fracture and expand, which results in the ongoing consumption of electrolytes and Li^+, leading to the low Coulombic efficiency (CE) and dead Li, thus shortening the cycle lives of LMBs.[27-28]

To address these problems, a plenty of strategies have been proposed, such as three-dimensional (3D) conductive hosts restricting volume expansion,[29-30] artificial SEI layer stabilizing the electrode/electrolyte interface,[31-32] lithophilic matrixes inducing uniform Li plating/stripping,[33-34] solid-state electrolytes improving energy density and safety,[35-38] surface coating on Li anodes modifying unstable SEI,[39-41] and so on. Among these solutions, the 3D conductive hosts have attracted more and more attentions due to the following reasons. Firstly, the 3D hosts afford space for

Li metal plating/stripping and there might be lithiophilic sites in some carbon-based hosts which can guide Li metal nucleation. Secondly, the 3D conductive hosts can decrease local current density noticeably, resulting in the delaying of initial nucleation of Li, thus restraining the Li dendrites growth.[42] Furthermore, the porous structure of the 3D framework can alleviate the volume expansion of metallic lithium during continuous cycling.[43-44] However, most 3D hosts are lithiophobic so that lithiophilic materials need to be deposited on the surface of the 3D hosts. As a result, in order to improve the Li affinity, many strategies have been taken for modifying the 3D hosts, such as electrodeposition,[45] atomic layer deposition,[46-49] thermal spraying,[50-51] and so on. The advantages and disadvantages of modifying 3D hosts in different ways mentioned above have been reviewed well in ref.,[27,52-53] thus these reported methods in previous studies are not the topic of this work.

A growing number of researchers are becoming interested in metal-organic frameworks (MOFs) derived materials because of their enormous surface area, controlled structure, high porosity, and changeable porosity.[54-65] Highly controllable nano materials derived from MOFs, such as metal oxides, metal sulfides and porous carbon structures have been employed in different energy conversion and storage technologies.[66-70] The advantages and compositions of MOF-derived materials have also been summarized well in ref.[71-73] Since Zhao *et al.*[74] firstly employ the MOF-derived materials to modify the 3D host for LMA, some excellent works about this topic have been reported.[75-77] However, to the extent that we know, there have no reviews about the MOF-derived materials which have been applied in modifying the 3D hosts for LMBs, thus this topic was chosen in this work.

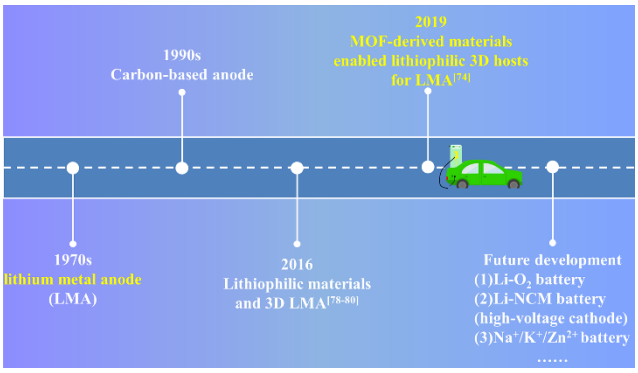
In this review, we systematically review recent progresses in the applications of MOF-derived materials modified 3D porous conductive framework as hosts for uniform lithium deposition in rechargeable LMBs. The role of several kinds of representative 3D porous frameworks, *i.e.*, nickel foam (NF), copper foam (CF) and carbon cloth (CC) in improving the performance of LMA is presented. Finally, the challenges and development prospect of employing MOF-derived materials to modify the 3D porous conductive framework for LMA are included. This work aims at illustrating the development in this field and inspiring innovation of future design of LMA.

2. Lithiophilic Materials and LMA

For several decades, various lithiophilic materials have been used in the field of LMBs through different ways. In about 2016, Cui *et al.*[78-80] have carried out the pioneer works and firstly proposed a novel concept of "lithiophilicity" and successfully prepared a lithiophilic framework for LMA by absorbing the melted lithium metal into the pores of carbon-based materials. Since then, the LMA becomes one of the hotspots for researchers in this area. In order to obtain a satisfying LMA, it is significant to improve the wettability of materials with Li metal. For instance, common nanostructured carbon materials have inferior compatibility with Li metal, which leads to a large nucleation overpotential during the Li nucleation process.[81] Over the years, a lot of researchers have devoted on looking for the approaches of addressing the above-mentioned problems. Here, we will briefly summarize several representative materials that can improve the wettability with lithium metal and their applications in LMA. The research history of LMA in LMBs is shown in Scheme 1.

In 2019, Duan *et al.*[82] introduced a graphite additive to synthesize a ceramic compatible lithium anode (Figure 1a and Figure 1b). The authors found that the Li-C composite can be simply casted onto the surface of lithiophobic garnet LLZTO, which might be attributed to the lower fluidity and higher viscosity compared to pure Li. Subsequently, Lu *et al.*[83] demonstrated a facile strategy for coating uniform aluminium-zinc oxides (AZO) layers in Cu

Scheme 1 Schematic diagram of the development process of LMA in lithium metal batteries



foams through magnetron sputtering deposition in 2020 (Figure 1c). The AZO coating efficiently modifies the surface characteristics of Cu foam for hosting Li metal, which could be obviously seen in Figure 1d. More importantly, this approach not only maintains the excellent mechanical properties of metal foams but also keeps the benefits of the readily reaction of Li and AZO coating. In the same year, in order to provide functional ions with sustained flow into the electrolyte, Yuan *et al.*[84] created "spansules" constructed of NaMg(Mn)F_3@C (NMMF@C) core@shell microstructures as the matrix of the LMA. The authors claimed that a novel LiF-involved bilayer structure and an *in-situ* produced metal layer on the Li/electrolyte interface will be helpful for reducing the formation and growth of lithium dendrites efficiently, which could be demonstrated in the SEM images after cycling (Figures 1e—g). Recently, Fang *et al.*[85] created a 3D macroporous framework out of carbon fibers doped with nitrogen and implanted with Ag nanoparticles (Ag@CMFs) as a host for LMA. (Figure 1h and Figure 1i). On one hand, the 3D macroporous framework can prevent the formation of dendritic Li by capturing metallic Li in the matrix and reducing local current density. On the other hand, the lithiophilic nitrogen-doped carbons serve as homogeneous nucleation sites owing to the small nucleation barrier, and the Li nucleation and growth behaviour has been improved superiorly by the reversible solid solution-based alloying reaction brought by the addition of Ag nanoparticles, which can be also proven in Figure 1j.

In addition, there are still plenty of lithiophilic materials unmentioned in this work, which also can be the choice for improving the performance of LMA, such as trasitional metal oxides (TMOs). It has been discovered that the TMOs can be completely reduced through their conversion reactions with lithium metal ($MO_x + 2xLi \leftrightarrow M + xLi_2O$, followed by the alloying process: $M + xLi \leftrightarrow Li_xM$).

We have listed some commonly used TMOs and calculated their Gibbs free energy change (ΔG) when they reacted with molten Li in Table 1. It can be concluded that MnO_2, Co_3O_4, Mn_2O_3 and SnO_2 can react with Li more easily because the ΔG is much lower than those of other materials. This criterion (ΔG) is easy and meaningful for the development of lithiophilic materials for LMA. For instance, Co_3O_4 and Mn_2O_3 can be applied in LMA, in contrast, FeO and NiO are inferior as lithiophilic materials.

Table 1 The computed Gibbs free energy of the reactions between molten Li and TMOs

	Materials								
	MnO_2	Co_3O_4	Mn_2O_3	SnO_2	CuO	FeO	ZnO	NiO	MnO
ΔG/kcal (180 °C)	−154	−342	−189	−142	−102	−73	−58	−82	−46
Violent reaction	√	√	√	√	×	×	×	×	×

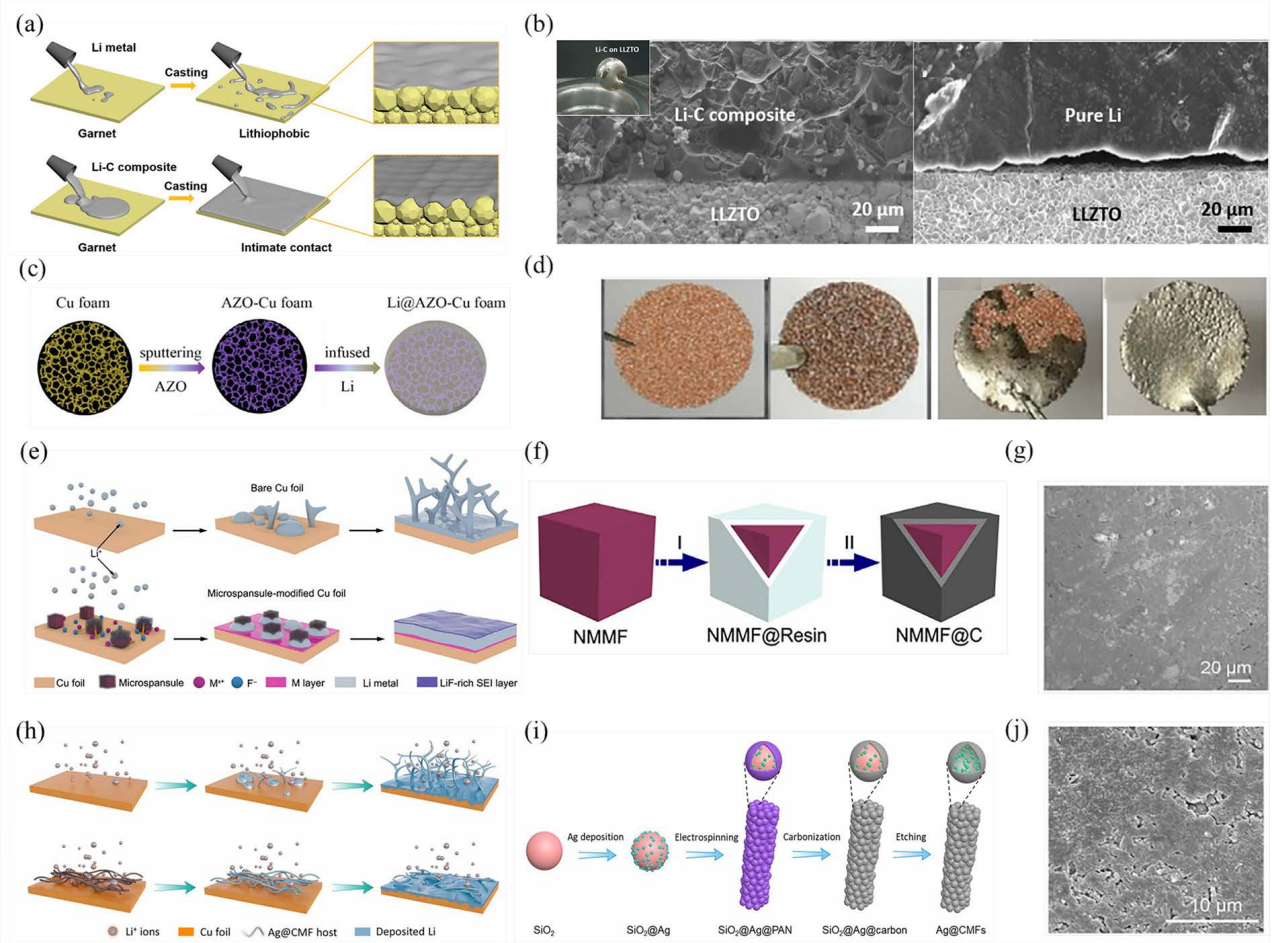


Figure 1 (a) Schematic diagrams of casting pure lithium (Li) and lithium-graphite (Li-C) composite on garnet SSEs. (b) FESEM images of Li-C/garnet and pure Li/garnet interfaces.[82] Copyright 2019, Wiley-VCH. (c) Schematic illustration of the fabrication of Li@AZO-Cu foam hybrid anodes. (d) Digital photographs of the preparation process of Li@AZO-Cu foam anodes (pristine Cu foam, AZO coated Cu foam, Li@pristine Cu foam, Li@AZO coated Cu foam).[83] Copyright 2020, Elsevier. (e) Schematic illustration of the expected growth behavior of Li when plating on bare Cu foil and microspansule-modified Cu foil. (f) Synthetic scheme of the NMMF@C cubes. (g) FESEM images of the NMMF@C-Li electrode in a symmetric cell after 400 cycles.[84] Copyright 2020, American Association for the Advancement of Science. (h) Schematic illustration of Li deposition behaviors on a planar copper foil and Ag@CMFs hosts. (i) Schematic illustration of the synthesis of Ag@CMFs. (j) SEM image of CMFs electrode after being plated with Li metal onto the CMFs.[85] Copyright 2021, American Association for the Advancement of Science.

Nevertheless, there are still some issues that require to be addressed immediately, such as how to prepare lithiophilic materials in a cheap cost or convenient way. Traditional ways like electrodeposition, atomic layer deposition, chemical/physical vapor deposition (CVD/PVD) were not suitable for most laboratories, fortunately, *in-situ* growth of the MOFs combined with facile calcination-thermolysis strategy can solve the problems effectively.

3. MOF-Derivation-Modified 3D Hosts

Apart from the Li affinity, the capillarity of the 3D hosts should be also a critical point to absorb molten Li to fill the pores of the structures.[86] Under the synergetic effects of the two factors, molten Li can perfectly fill the porous structure and wet the surface of the host.

3.1. MOF-derivation-modified metal foams

Metal foams have been demonstrated to be a viable choice in the energy storage field, which stand for their high porosity, good thermo-physical properties and mechanical strength.[87] Among various commercial metal foams, the Nickel Foams (NFs) and Copper Foams (CFs) have been frequently used as the supporting substrate and current collector for electrochemical applications due to their superior structural integrity and electrical conductivity.[88] Here we will discuss the applications of the representative two metal foams (NFs, CFs) for LMA.

3.1.1. MOF-derivation-modified nickel foam (NF). NFs are increasingly used as electrodes in advanced electrochemical systems such as supercapacitors and lithium-ion batteries, which are well-known for their low density and excellent mechanical properties.[89] Unfortunately, the bare NFs without modification might lead to high nucleation overpotentials or unstable Li plating/stripping process due to the "lithiophobicity" of bare NFs, which was demonstrated in previous studies.[90] Thus, a series of methods on modifying NFs have been proposed for the preparation of lithiophilic NFs in LMA in recent years. Among various ways, MOF-derivation-modified NFs are a more convenient way to guide the uniform lithium deposition and this has gained much attentions of researchers.

In 2019, Zhao *et al.*[74] firstly suggested vertically aligned ZnO nanosheets derived from zinc-based-MOF grown on NF (LZNF), which were used as the lithiophilic porous scaffold. Figure 2a depicted how the LZNF 3D matrix was prepared. Firstly, a simple liquid-phase deposition approach was used to develop a Zn-containing zeolitic imidazolate framework with a leaf-like morphology (Zn-ZIF-L) vertically on the NF at room temperature. As presented in Figure 2b, the symmetric cells of LZNF@Li exhibited the ultralow overpotentials of 45 mV for about 250 cycles, and the GZNF@Li (GZNF: NF modified with grain-like ZnO particles) anode and pure Li anode showed worse cycling performances.

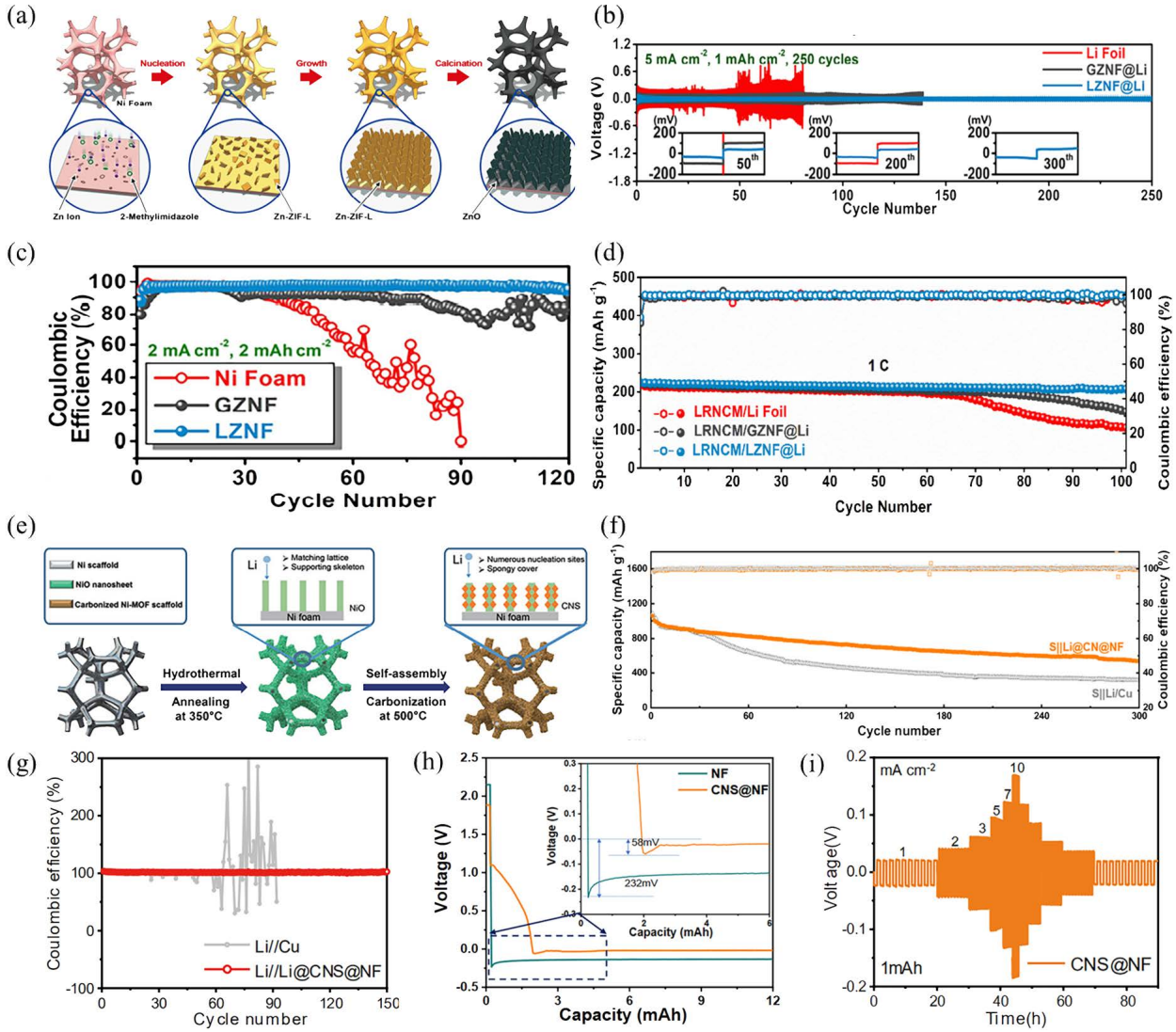


Figure 2 (a) Schematic diagram of the synthetic procedure of LZNF. (b) Galvanostatic cycling voltage profiles of planar Li foil, GZNF@Li and LZNF@Li electrodes in symmetric Li|Li cells at 5 mA/cm^2 with the areal capacity of 1 mAh/cm^2. (c) Comparison of Coulombic efficiencies of NF, GZNF and LZNF electrodes in half-cells using bare Li foil as the counter electrodes at 2 mA/cm^2 with areal capacity of 2 mAh/cm^2. (d) Long-term cycling performance of the LRNCM|Li, LRNCM|GZNF@Li and LRNCM|LZNF@Li full-cells at 1 C.[74] Copyright 2019, Elsevier. (e) Schematic representation of synthesis process and the proposed mechanism with regard to the electrochemical performance improvement. (f) Cycling stability of pristine Li|S and Li@CNS@NF|S full cells at 0.5 C (1 C = 1678 mAh/g). (g) CE of the Li stripping/plating process of Li|Li@CNS@NF cells, with Li|Cu cells as comparison (1 mA/cm^2). (h) Voltage-time curve during the electrodeposition process (1 mA/cm^2). (i) Rate performance of cells comprising CNS@NF at current density from 1 to 10 mA/cm^2.[91] Copyright 2021, Royal Society of Chemistry.

Figure 2c displayed the CE of various electrodes in half cells with Li foil serving as the counter electrode. Obviously, the CE of the LZNF electrode was relatively high and constant compared with the other two electrodes. This probably benefited from the homogeneous lithium deposition and stable SEI formation brought by the unique structure of LZNF. Finally, to further evaluate the compatibility with $(Li_{1.14}Ni_{0.136}Co_{0.136}Mn_{0.544})O_2$ cathodes and practical viability of LZNF@Li anodes, they were assembled into full cells and tested at 1 C (Figure 2d). All of these findings suggest that LZNF with arrays of lithiophilic ZnO nanosheets can function as a reliable host for 3D LMA for practical applications. Recently, Zhao *et al*.[91] have proposed a novel design to carbonize Ni-MOF on NF scaffold (CNS@NF), which can serve as a stable structural composite anode for uniform Li^+ deposition. The preparation process of CNS@NF is described in Figure 2e. By smoothening the sharp edges of NiO, the loose structure of the carbonized Ni-MOF can lower the local current density. To assess the applicability of Li@CNS@NF in lithium-sulfur batteries, the Li@CNS@NF|S cells were assembled and tested at 0.5 C (Figure 2f). It showed a stable and consistent cycling performance. Moreover, the performance of the lithium stripping and plating was also investigated via evaluating the CE during the processes (Figure 2g). In comparison to Li|Cu cells, Li|Li@CNS@NF demonstrated a significantly more stable CE after 150 h at a current density of 1 mA/cm^2. The electrodeposition curve presented in Figure 2h proved that CNS@NF showed a smaller final overpotential (58 mV) in contrast with that of Li@NF (232 mV) owing to the lithiophilicity of CNS@NF. In addition, the rate performance of symmetric cells revealed Li@CNS@NF has a more significant boost on cycling performance improvement (Figure 2i).

In our previous work,[92] a ZIF-67-derived Co_3O_4 nanoparticles modified NF has been proposed as a 3D host for even distribution of molten Li (Figure 3a). It can be observed that the molten Li was rapidly and uniformly infused into the whole Co_3O_4-NF host (within 10 s), which implies good Li affinity of Co_3O_4-NF host. Figure 3b presented the rate properties of symmetric cells with Li-Co_3O_4-NF electrode, which indicate the high electrochemical performance brought by the Co_3O_4-NF host. To further prove

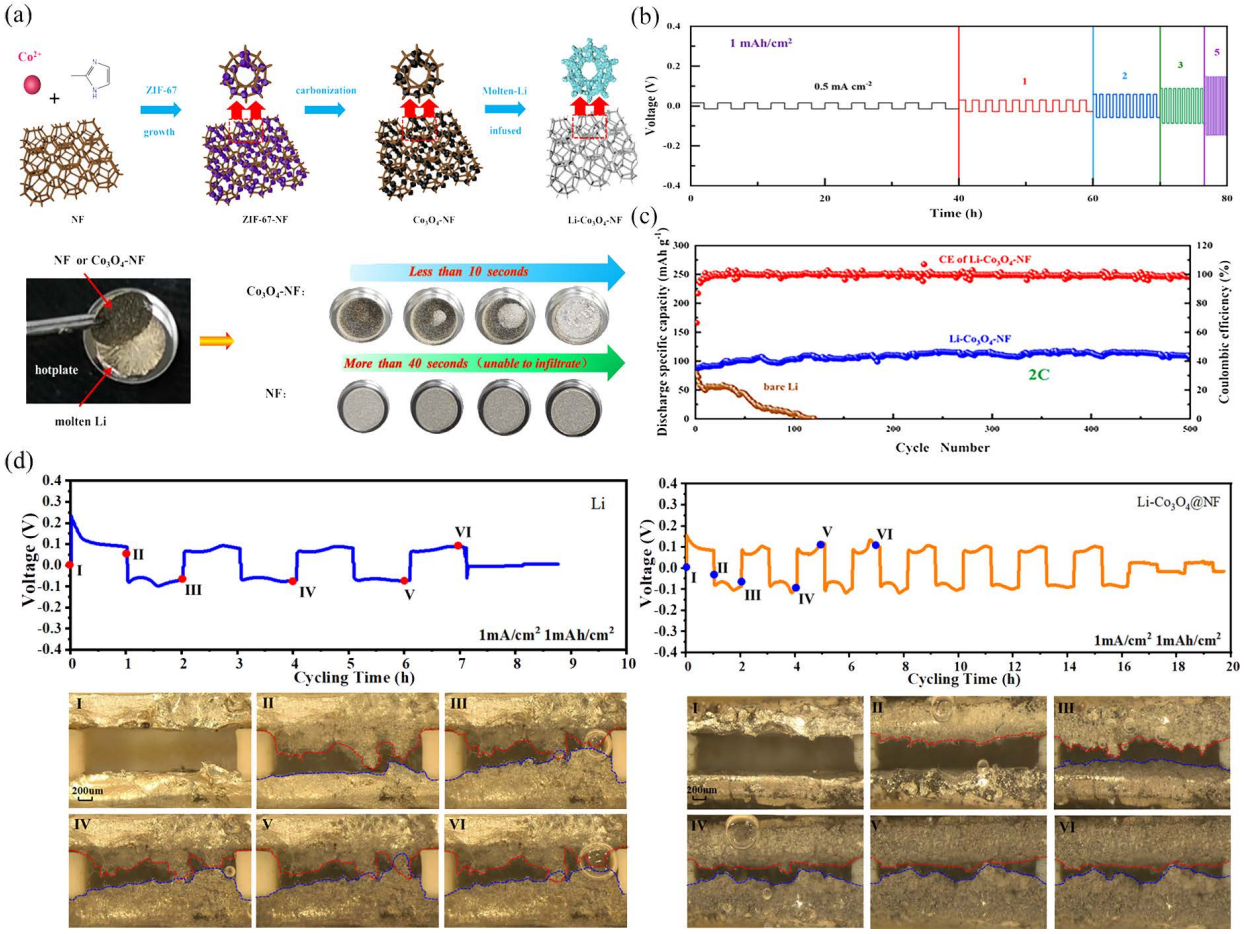


Figure 3 (a) Synthesis and molten Li infusion procedure of the Co_3O_4-NF electrode. (b) Rate capability of the symmetric cell with Li-Co_3O_4-NF electrode at different current densities (0.5—5 mA/cm^2). (c) Long-term cycling performance of the Li-Co_3O_4-NF|LFP and pure Li|LFP cells tested at 2 C. (d) Charge/discharge curves and *in situ* OM images of symmetric cells with pure Li and Li-Co_3O_4-NF anodes at a current density of 1 mA/cm^2 and an areal capacity of 1 mAh/cm^2.[92] Copyright 2022, Elsevier.

the advantages of this structure, the Li|LFP and Li-Co_3O_4-NF|LFP full cells were assembled and tested at 2 C, which is depicted in Figure 3c. These results reveal that the composite anode possesses an improved cycling and rate performance, which might be owing to the excellent Li stripping/plating behavior on the 3D lithiophilic host. What's more, the *in situ* optical microscopy (OM) images in Figure 3d also demonstrated that the Li-Co_3O_4-NF electeode could suppress the growth of lithium dendrites obviously.

3.1.2. MOF-derivation-modified copper foam (CF). In addition, CF has also been reported as an effective platform for the growth of active materials.[93] Compared to the above-mentioned 3D porous NF, 3D structural CF with a higher conductivity should be preferable as an anodic current collector.[94] Nevertheless, the corrosion resistance of CF is much poorer than NF, which might be the reason why NF is more attractive in the field of LMA. Moreover, CF has weak interaction with Li, thus inducing uneven Li deposition.[95-96] In the same way, uniform metallic lithium deposition and the prevention of Li dendrite formation in CF have also been the targets of intense research.[97]

In 2020, Liu *et al.*[98] showed a special free-standing lithiophilic vertical cactus-like framework (LVCF) made of coordination polymer (CP) that is employed as the current collector for reliable Li metal batteries. Different Li plating behaviors on bare CF and LVCF current collectors are demonstrated in Figure 4a. For the bare CF, the uneven Li nucleation was caused by the poor lithiophilicity of the skeleton, and then resulted in the growth of severe Li dendrite, which eventually led to a series of problems such as more electrolyte consumption, capacity loss, *etc*. On the contrary, there are many lithiophilic sites in LVCF like N-containing groups and ZnO quantum dots, which could enhance uniform Li^+ distribution and further guide even deposition of Li. As demonstrated in Figure 4b, the excellent CE performance of LVCF at 5 mA/cm^2 with an areal capacity of 1 mAh/cm^2 also showed a stable Li plating/stripping behavior that inhibited the growth of Li dendrites and prevented excessive electrolyte consumption. In contrast, the brittle SEI and the continuous growth of Li dendrites and "dead Li" resulted in the ultrashort cycles of the bare CF current collector. In order to determine the excellent cycling stability of LVCF@Li, symmetric cells with Li foil, CF@Li and LVCF@Li electrodes were assembled (Figures 4c and 4d). These results illustrated that the LVCF@Li electrode could suppress Li dendrites greatly and avoid extra electrolyte consumption, especially in the terms of rate performance and large cycling capacity. Full cells assembled with the $LiFePO_4$ (LFP) cathode and LVCF@Li anode were investigated, which are compared to Li|LFP and CF@Li|LFP cells. Figure 4e delivered an outstanding rate properties of LVCF@Li anode (132.2 mAh/g at 5 C).

By the end of this year, An *et al.*[95] put forward a new design of N/O dual-doped carbon array originated from ZIF-8, which was grown on CF tightly (NOCA@CF) as a durable scaffold for uniform Li deposition (Figure 4f). The electrochemical behavior of NOCA@CF in ether-based electrolyte was studied to demonstrate the universality of the NOCA@CF electrode in inhibiting the growth of Li dendrite (Figure 4g). The bare CF became rougher and looser due to the growth of lithium dendrite, which contributes to the uneven lithium deposition. Contrarily, the smooth surface of deposited Li on NOCA@CF is proven that no distinct

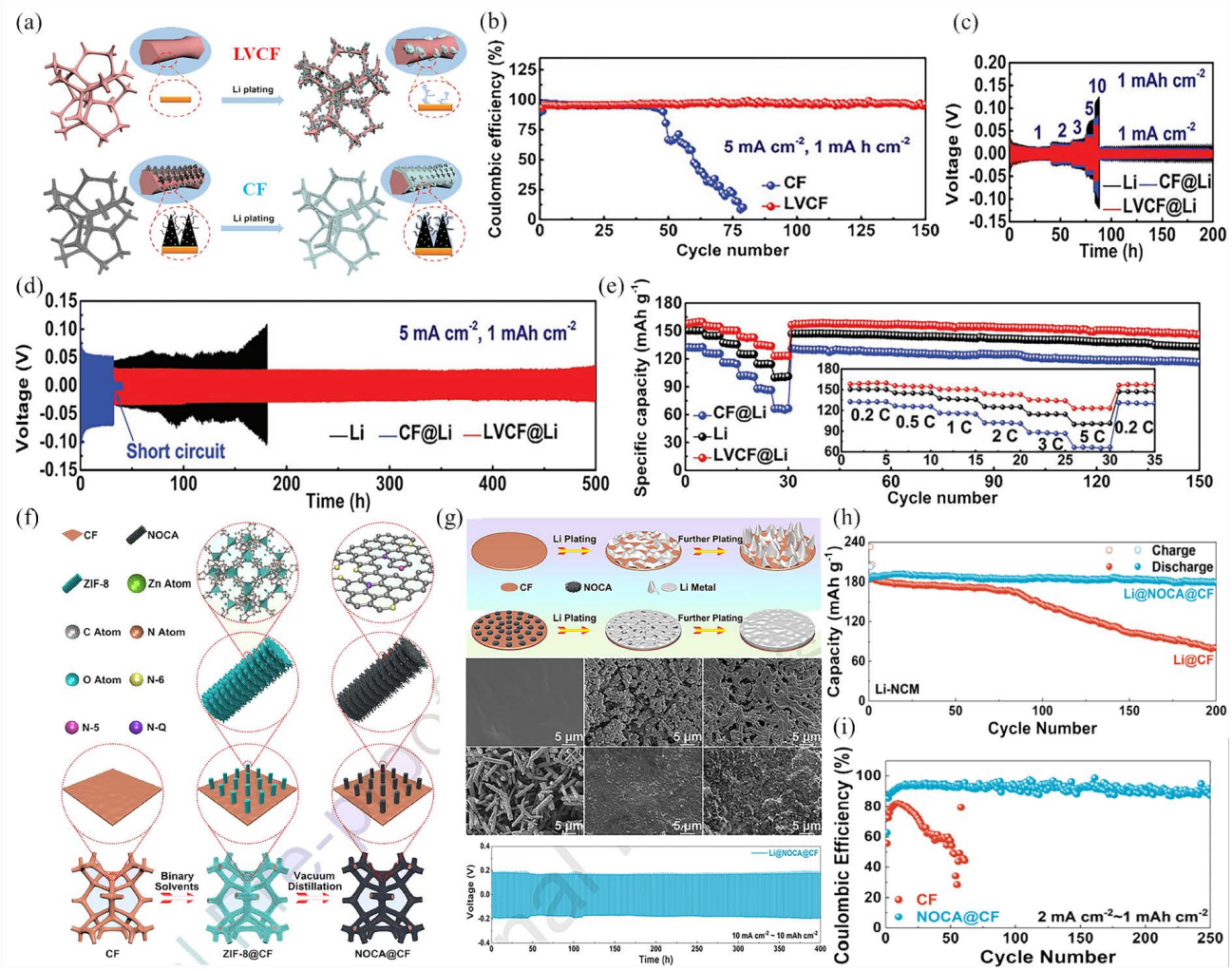


Figure 4 (a) Schematic diagrams for the comparison of Li deposition on bare CF and LVCF. (b) Coulombic efficiency of bare CF and LVCF composite electrode at 1 mA/cm^2 with the fixed capacity of 1 mAh/cm^2. (c) Rate performance of Li|Li, CF@Li|CF@Li and LVCF@Li|LVCF@Li cells (1—10 mA/cm^2). (d) Voltage-time profiles of galvanostatic Li plating/stripping process in symmetric cells (5 mA/cm^2, 1 mAh/cm^2). (e) Rate performance at various current densities from 0.2 to 5 C.[98] Copyright 2020, Wiley-VCH. (f) Schematic clarifying the fabrication of NOCA@CF product. (g) Schematic explaining Li deposition behavior on CF and NOCA@CF; SEM images of pristine CF, 100th Li plating, 200th Li plating at 1 mA/cm^2 (above); Cycling performance at 10 mA/cm^2 of symmetric cell with Li@NOCA@CF anode (below). (h) Cycling properties of Li|NCM cells based on Li@CF and Li@NOCA@CF anodes at 100 mA/g. (i) CEs of CF and NOCA@CF in carbonate-based electrolyte for LMA at 2 mA/cm^2 with a capacity of 1 mAh/cm^2.[95] Copyright 2020, Elsevier.

dendritic or mossy Li metal was found after cycling, which is beneficial to the cycle performance and lifespan of LMA. The cycling characteristics of a symmetric Li cell with a Li@NOCA@CF anode were also presented in Figure 4g. It could be clearly seen that the cell exhibited a low overpotential and stable cycling behavior over a 400-hour period under a high current density of 10 mA/cm^2. These improvements might be ascribed to the following explanations: (I) NOCA@CF can induce even electrochemical deposition due to its high specific surface area and good electric conductivity; (II) Volume change during Li plating/stripping processes can be alleviated by the hierarchical porous structure; (III) The NOCA layer grown on CF surface can play the role of guiding uniform distribution of Li^+ flux. Moreover, electrochemical performance presented in Figures 4h and 4i further demonstrates that the NOCA@CF anode has excellent electrochemical properties, which is ascribed to high porosity, large surface area and improved electronic conductivity of NOCA@CF anode and this can accelerate ion transport, facilitate electrolyte diffusion, and alleviate volume change during lithiation/delithiation process at the same time.

3.2. MOF-derivation-modified carbon cloth (CC)

However, the metal materials might lead to the reduction of the energy density of LMA because of its high mass density, which is unacceptable.[99-100] Fortunately, CC (or carbon fibers) can also be used as the 3D host.[101] Compared with any other porous carbon hosts for LMA, CC presents a more attractive practical prospect in LMA owing to its great flexibility, superior mechanical properties, strong electrical conductivity and chemical stability.[102] Nevertheless, the bare CC has a much poorer mechanical strength compared with NF and CF, which cannot inhibit the volume change effectively during cycling. Besides, CC also presents the same lithophobic characteristic, which means it also needs to be modified. Inspired by MOF-derivation-modified metal foams, relevant researchers utilized the idea into CC system and it also has acquired excellent performances.

Wang *et al.*[76] have proposed an optimum design of a carbonized MOF nanorod arrays modified carbon cloth (NRA-CC) as a support for LMA (Figure 5a) in 2020. Lithium plating on carbon-based electrodes is made easier by the cobalt and nitrogen atoms produced during the carbonization process of ZIF-67, which successfully changes the lithiophobic character of carbon materials into lithiophilicity. Long-term cycling properties of the Li|CC and Li|NRA-CC cells were conducted in Figure 5b, which indicates that the NRA-CC electrode is capable of stable and sustainable operation. To further study the cycling stability of Li@NRA-CC, the symmetric cells were tested at various rates, which is depicted in

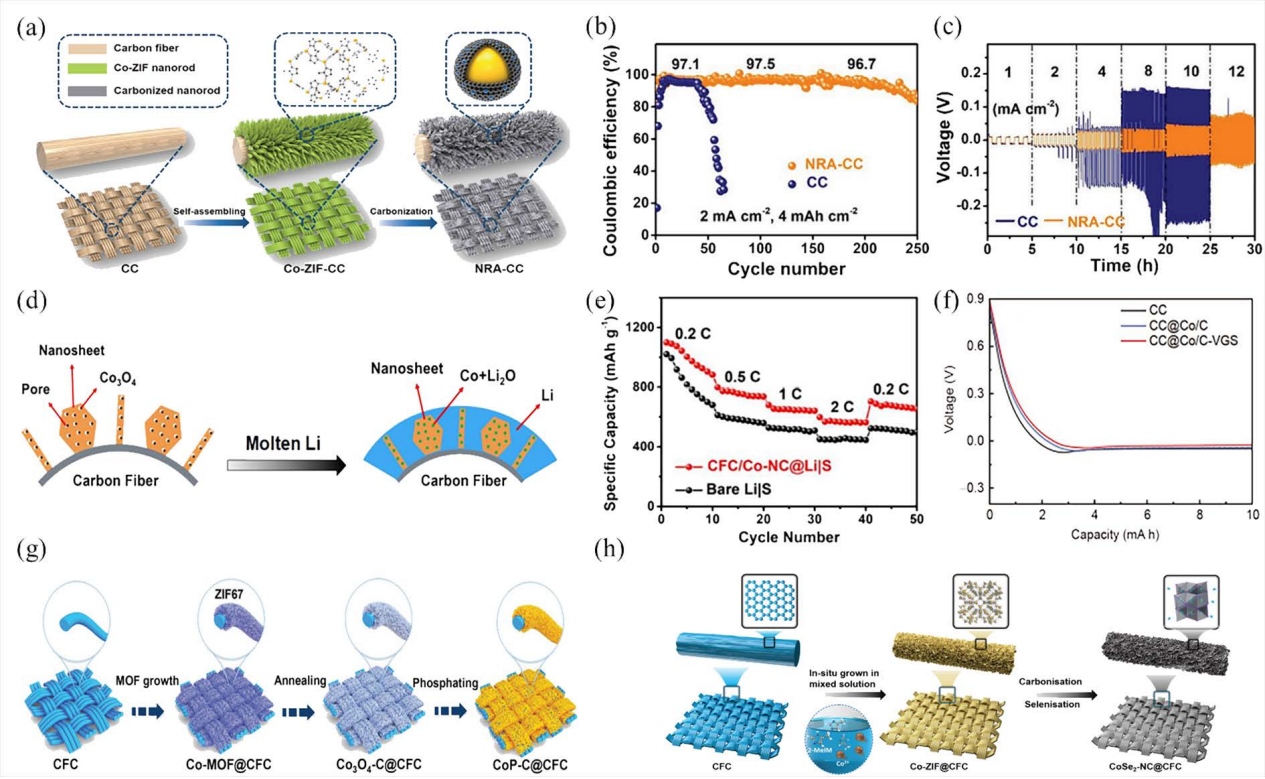


Figure 5 (a) Schematic illustration of the synthesis procedures of NRA-CC. (b) CE of the NRA-CC electrode with Li deposition amount of 4 mAh/cm^2 at a current rate of 2 mA/cm^2. (c) Rate performance of Li@NRA-CC|Li@NRA-CC symmetrical cells (1—12 mA/cm^2).[76] Copyright 2020, Wiley-VCH. (d) The schematic diagram of molten Li thermal infusion process. (e) Rate performances of CC/Co-NC@Li|S and Li|S cells (0.2—2 C).[103] Copyright 2019, Elsevier. (f) Nucleation overpotential of Li plating on bare CC, CC@Co-C, and CC@Co/C-VGS (1 mA/cm^2).[104] Copyright 2022, Springer Nature. (g) Schematic of the preparation process of CoP-C@CC.[105] Copyright 2022, Wiley-VCH. (h) Schematic diagram of preparation route for $CoSe_2$-NC@CC.[75] Copyright 2022, Wiley-VCH. The above-mentioned materials were all derived from ZIF-67.

Figure 5c. It can be obviously seen that the cells using Li@NRA-CC anode displayed a more consistent cycle stability and a less voltage hysteresis than the cells using Li@CC anode even at high current densities (10, 12 mA/cm^2), which might be ascribed to the cooperative effects of the lithiophilic NRAs and interconnected CC. Furthermore, a similar work, ZIF-67-derived Co_3O_4-embedded and nitrogen-doped porous carbon nanoflake arrays on carbon cloth (CC) (CC/Co_3O_4-NC) was reported by Jiang *et al.*,[103] which can act as a remarkably stable and wettable host to prestore molten lithium for dendrite-free LMAs (Figure 5d). The authors claimed that the superior lithiophilicity of CC/Co_3O_4-NC host is attributed to the cooperative effects of the porous nanostructures, the lithiophilic nitrogen dopants and the embedded Co_3O_4 nanoparticles. The carbon nanoflake array frameworks could not only be crucial for maintaining the structural stability of the CC/Co_3O_4-NC host during the rapid molten-Li infusion process, but they could also provide physical confinement for the deposited Li and ensure quick electron/ion transport during Li stripping/plating processes even in the fields of Li|S cells (Figure 5e). Recently, Shao *et al.*[104] have designed a special hierarchical carbon-based structure made up of ZIF-67-derived Co/C nanosheets and vertical graphene sheets (VGS), which were grown on flexible CC. In order to understand the variations in lithiophilicity performance between the anodes, the charge-discharge profiles were meticulously tested at 1 mA/cm^2 to investigate the nucleation overpotential, which was presented in Figure 5f. It suggests that Co/C nanosheets and VGS both have the ability to reduce deposition overpotential. The lithiophilicity of the hosts has been effectively improved by evenly dispersed Co nanoparticles and N-doped carbon, facilitating the smooth Li nucleation and uniform deposition processes. Li metal may be included in a bilayer carbon-based current collector to produce anodes that are dendrite-free, which consequently extends the life of symmetric and full cells.

Besides, Jiang *et al.*[105] constructed a MOF-derived porous carbon sheath with embedded CoP nanoparticles-modified CC (CoP-C@CC, Figure 5g), which was added a phosphating process. Significantly, the authors claimed that the outstanding electrochemical properties of CoP-C@CC are attributed to the reasons as follows: (I) weakly conductive porous carbon sheath derived by MOFs declined the space charge polarization in pure CC and provided a more uniform Li^+/electron flux in the entire 3D hosts; (II) fast ionic conductive Li_3P (10^{-4} S/cm) and electron-conductive Co nanoparticles can homogenize the Li^+ flux and charge distribution on the CoP-C@CC simultaneously during the constantly repeated Li plating/stripping processes; (III) the lithiophilic CoP and reversible Li_3P products effectively remove nucleation barriers and form a robust SEI layer, which are beneficial to achieve superior Li affinity and obtain a smooth Li layer with good reversibility; (IV) a robust and adaptable carbon scaffold can withstand extreme pressure changes and reduce the volume effect. In the same year, Cao *et al.*[75] created a 3D hierarchical structure with $CoSe_2$ nanoparticle modified nitrogen-doped carbon ($CoSe_2$-NC) nanoflake arrays grown on a CC as a flexible host for dendrite-free LMAs (Figure 5h), which was added a selenidation process. Compared to the above study, the $CoSe_2$-NC@CC offers the advantages as follows: (I) the uniformly distribution of $CoSe_2$ nanoparticles and doped N-bonding configurations entrasted CC to excellent lithiophilicity, which facilitates uniform Li deposition behaviour with a relatively low overpotential; (II) $CoSe_2$ nanoparticles are transformed into Li_2Se/Co nanoparticles during the early Li nucleation process owing to the high reducibility, which can act as ordered Li nucleation sites to regulate Li deposition behaviour; (III) *in situ*-generated Li_2Se-enriched SEI has the advantages of high ionic conductivity, good chemical stability and large mechanical strength, which are favourable for fast Li^+ transport and electrode/electrolyte interface stability.

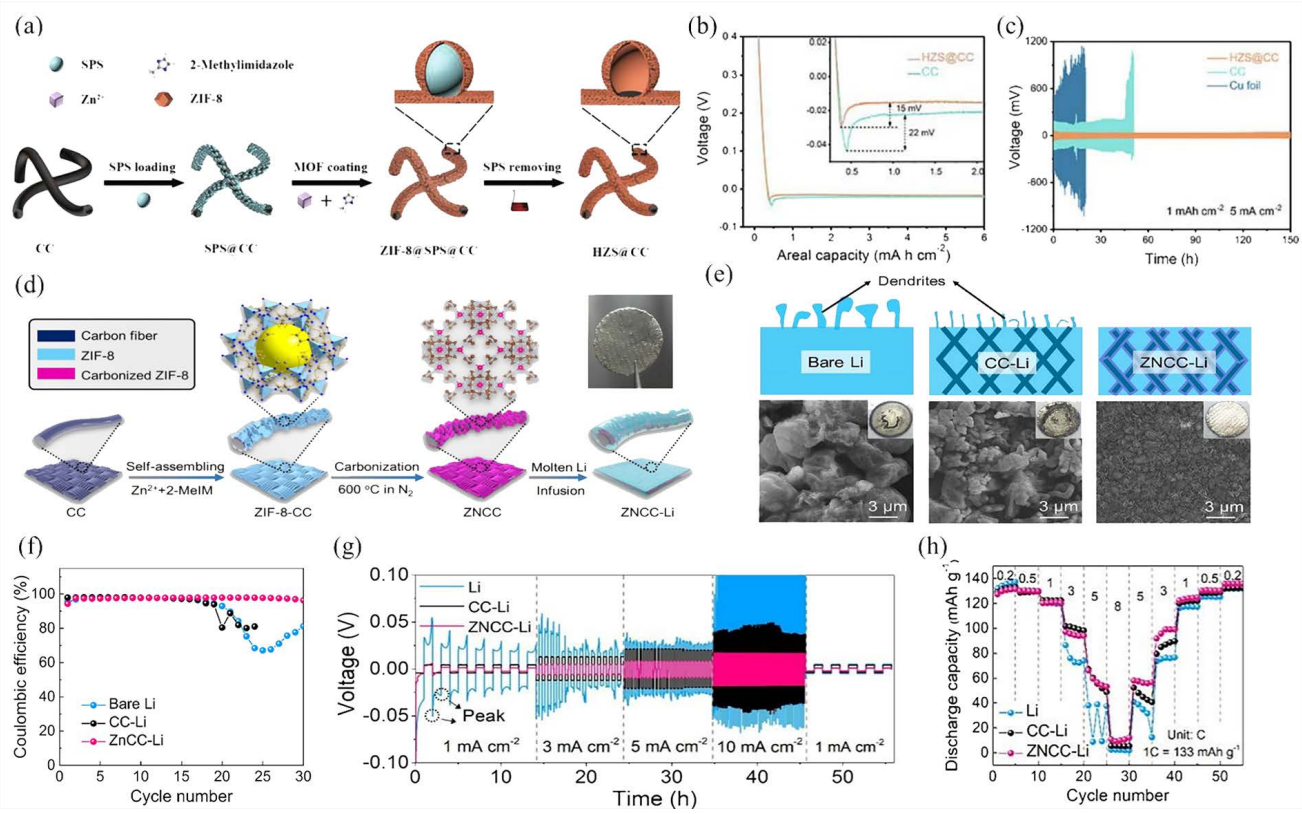


Figure 6 (a) Schematic illustration of the preparation of HZS@CC. (b) Initial discharge profiles at 1 mA/cm^2 of HZS@CC and CC. (c) Long-term cycling performance with a cycling capacity of 1 mAh/cm^2 at 5 mA/cm^2.[106] Copyright 2020, Elsevier. (d) Scheme of the synthetic procedure of ZNCC and ZNCC-Li. (e) Li dendrite morphology after 500 cycles at 5 mA/cm^2. (f) Coulombic efficiency of Li, CC-Li and ZNCC-Li obtained from Li-Cu cells (discharge: 0.5 mA/cm^2, charge: 1 mA/cm^2). (g) Voltage profiles of Li, CC-Li, ZNCC-Li at different current densities (1—10 mA/cm^2). (h) Rate performance of ZNCC|LMNO ($LiNi_{0.5}Mn_{1.5}O_4$) full cells (0.2—8 C).[107] Copyright 2021, Royal Society of Chemistry. The above-mentioned materials were all derived from ZIF-8.

Furthermore, Fang *et al*.[106] prepared a novel structure — a compact ZIF-8 shell with hollow vesicle-shaped humps was built on carbon cloth (HZS@CC) as a 3D LMA host (Figure 6a), a well-distributed Li-ions flow is successfully realized during Li deposition by the numerous functional groups and organized channels of ZIF-8. In addition, the improvement in the nucleation overpotential of HZS@CC (Figure 6b) could be attributed to the larger space for Li-ions nucleation on fiber surface. At the same time, another important factor in the homogeneous multi-site nucleation is the regular Li-ions passageways on channel-rich MOF particles. As presented in Figure 6c, the HZS@CC electrode can still maintain a consistent lithium round-trip conversion after more than 150 cycles, which is superior to the majority of reported LMA hosts. Subsequently, Zhuang *et al*.[107] created a novel carbon-nitride-like material by carbonizing ZIF-8 and producing MOF-derived Zn-C_xN_y nanoparticle arrays on a CC (Figure 6d). The authors claimed that the homogeneous Li plating/stripping process can be accelerated and the growth of lithium dendrites can be suppressed by N and Zn species, which act as lithiophilic sites. These results presented in Figures 6e—h indicate that the uneven Li plating/stripping process might be delayed by using the CC scaffold and Zn-C_xN_y nanoparticle arrays, which is capable to increase the surface area of the Li anode and decrease the current density. On one hand, the CC host could act as electrically conductive scaffolds owing to its high specific surface area. On the other hand, the nanoparticle arrays impregnated with Zn and N species render double lithiophilic spots that could promote the even Li plating/stripping process. Furthermore, the interaction between Zn and N species and Li-ions could potentially improve the reaction kinetics of Li-ion desolvation.

Besides, Zhou *et al*.[77] proposed a facile method for Co nanoparticles anchored in N-doped carbon (CN-Co) nanosheet arrays on CC (Figure 7a) in 2020, which was derived from ZIF-L. This unique skeleton has the following benefits: (I) the lithiophobic CC is converted to a lithiophilic framework by the uniformly distributed N-containing functional groups and embedded CN-Co nanosheet arrays, which then directs uniform Li deposition with low nucleation overpotential. These Co nanoparticles might offer useful sites to guide Li deposition; (II) the uniform distribution of Li ionic flux can be facilitated by the well-defined array structure, which can also increase the active surface area and improve the electrolyte absorption capacity during circulation in the meantime. The improved electrochemical performance of the CC@CN-Co host presented in Figure 7b further illustrates the superior properties of CC@CN-Co for LMA.

In 2021, Zeng *et al*.[108] synthesized CC modified with ZnO nanosheets derived from ZIF-L and nitrogen-doped carbon (CC@ZnO/NC) as a 3D lithiophilic skeleton to accommodate molten Li for dendrite-free LMAs (Figure 7c). The CC@ZnO/NC skeleton had a high Li affinity to actualize the quick infusion of molten Li into the host, generating a dense CC@ZnO/NC@Li composite anode. Figure 7d illustrates the morphology of CC@ZnO/NC@Li composite anode, which is apparently smooth. Figures 7e and 7f imply that the stabilization of the Li plating/stripping process and increased cycle reversibility can both be achieved by the CC@ZnO/NC skeleton. In order to verify the viability of composite lithium anodes for commercial use, the full cells with LFP cathode were assembled and measured (Figure 7g), which further demonstrates the feasibility and superiority of the MOF-derived-modified 3D CC host in the practical applications for LMA.

In conclusion, previous studies have proven that MOF-derivations can be easily deposited on the 3D porous conductive frameworks through a simple solvothermal method and outstanding electrochemical properties can be achieved (Table 2).

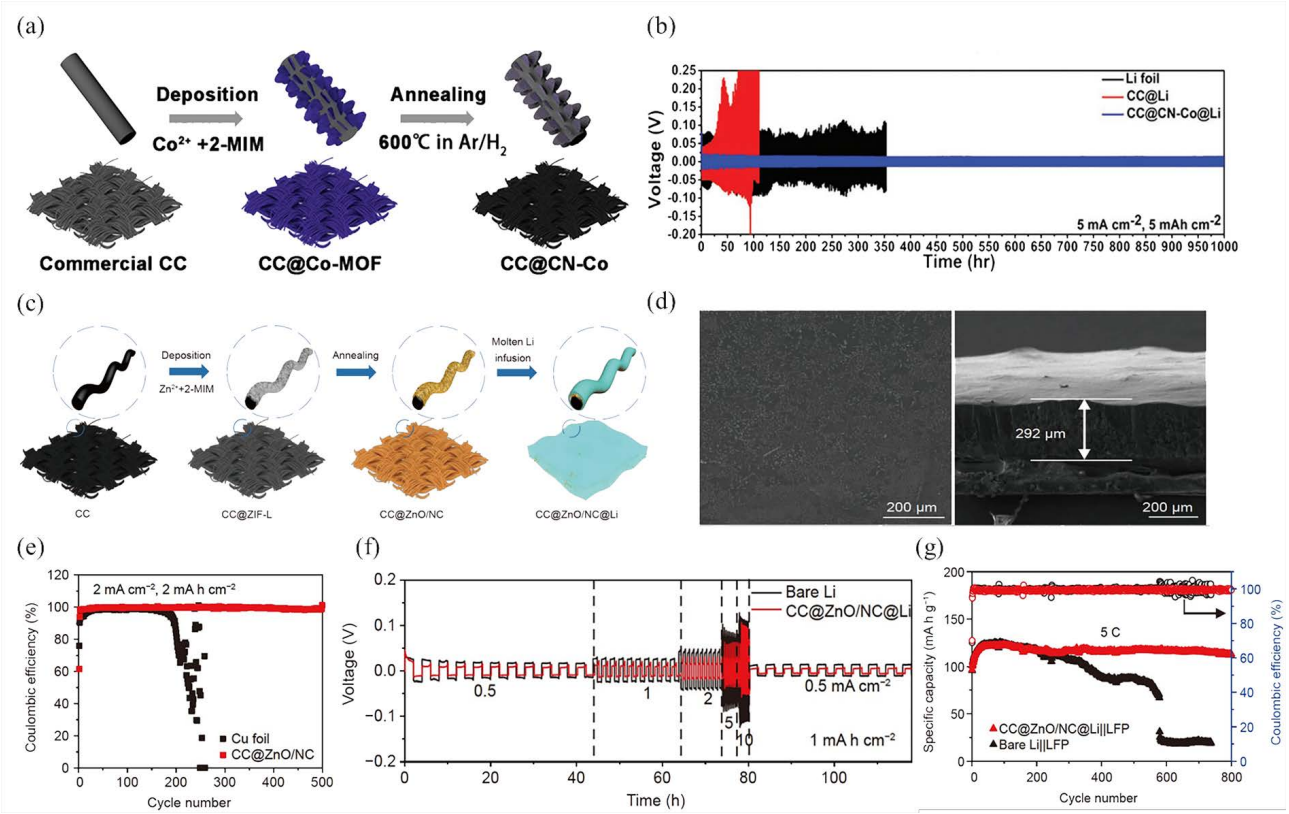


Figure 7 (a) Schematic illustration for the growth of MOF-derived CN-Co nanosheet arrays on CC substrate. (b) Voltage profiles of metal Li plating/stripping in symmetric cells at 5 mA/cm^2 for 5 mAh/cm^2.[77] Copyright 2020, Wiley-VCH. (c) Flow chart for the preparation of CC@ZnO/NC@Li composite anode. (d) SEM images of CC@ZnO/NC@Li. (e) CEs of CC@ZnO/NC and Cu foil cycled at 2 mA/cm^2 and 2 mAh/cm^2. (f) Rate performances at different current densities (0.5—10 mA/cm^2). (g) Cycling performances of CC@ZnO/NC@Li|LFP and Li|LFP full cells at 5 C.[108] Copyright 2021, Springer Nature. The above-mentioned materials were all derived from ZIF-L.

Table 2 Electrochemical properties comparison of different MOF-derivations-modified 3D hosts

MOF-derivation-modified-host	MOF-derivation	3D hosts	Current density/(mA·cm^{-2})	Capacity/(mAh·cm^{-2})	Cycling times/h	Ref.
LZNF (ZIF-L (Zn))	ZnO	NF	5	1	100	[74]
CNS@NF (Ni-MOF)	NiO	NF	5	5	1000	[91]
Co_3O_4-NF (ZIF-67)	Co_3O_4	NF	3	1.5	500	[92]
LVCF (ZIF-8)	ZnO	CF	5	1	60	[98]
NOCA@CF (ZIF-8)	N/O-doped carbon array	CF	10	10	400	[95]
CC@CN-Co (ZIF-L (Co))	Co/N-C	CC	5	5	1000	[77]
CFC/Co_3O_4-NC (ZIF-67)	Co_3O_4/N-C	CC	5	10	~200	[103]
NRA-CC (ZIF-67)	Co/N-C	CC	12	12	200	[76]
HZS@CC (ZIF-8)	Hollow ZIF-8 shell	CC	5	1	100	[106]
ZNCC (ZIF-8)	Zn/N-C	CC	10	1	~2300	[107]
CoP-C@CFC (ZIF-67)	CoP	CC	2	1	~550	[105]
CC@Co/C (ZIF-67)	Co/C	CC	10	10	800	[104]
$CoSe_2$-NC@CFC (ZIF-67)	$CoSe_2$-N-C	CC	10	20	280	[75]
CC@ZnO/NC (ZIF-L)	ZnO-N-C	CC	10	1	~180	[108]

4. Summary and Outlook

Due to the synergetic effects of the MOF-derived lithiophilic materials and the 3D hosts, the development of MOF-derived materials modified lithiophilic 3D hosts has gained valuable progresses for LMA. Firstly, the lithiophilic materials can be obtained easily by the facile method of *in-situ* growth of the nano-MOFs on the hosts (use the MOF directly or MOF-derived materials); Secondly, the lithiophilic materials obtained from MOFs can inherit the advantages of the MOFs, such as high surface area, adjustable pore spaces, well-organized open channels, and so on. What's more, different metal ions or organic functionality enriched the variety of choices of MOFs (>20 000 species); Thirdly, the porous 3D conductive hosts can alleviate the volume expansion during charging/discharging and decrease local current density so as to restrain the Li dendrites growth. And this technology could also be applied in lithium/sodium/potassium metal batteries or Li-S/air batteries.

However, we believe that many problems and challenges are remain to be addressed. Here, we outline several possible direc-

tions for future Li anode research, which may lead to pathways for the future research and development of LMA:

(1) Development of the MOFs. From Table 2, it can be concluded that the most used MOFs are Zn-MOFs or Co-MOFs (ZIF-8 and ZIF-67), and from Table 1, MnO_2 has the lowest ΔG, which means Mn-MOFs might be the best MOFs to be used in this area compared with others. Bimetallic oxides may also show a better performance, such as bimetallic-MOFs and polyoxometalates-based MOFs (POMOFs). What's more, combined with phosphating and selenidation processes or doped by N/F element, the advantages of the diversity of MOFs or MOF-derived materials allow researchers to have plenty of choices.

(2) Advanced characterization techniques on Li metal. Different techniques can mainly be divided into two categories that have been applied to study Li metal anodes. For example, SEM, TEM, optical microscopy and atomic force microscopy (AFM) to study the microstructures, X-ray photoelectron spectroscopy (XPS) and Fourier transform infrared spectroscopy (FTIR) for surface chemistry analysis. However, we have not yet understood the lithium nucleation, growth, stripping and chemical reactivity clearly, for the reason that most of the reported characterizations were done *ex situ* or *in situ* under static conditions. Applying *in situ* diagnostics in a real working environment can provide more meaningful knowledge on the dynamics of LMA. We believe that a combination of characterization techniques, including both *in situ* and *ex situ* techniques, is helpful to understand the dynamic behaviour of Li in-depth.

(3) Design 3D hosts with gradient. During infusion of molten Li or cycling, Li prefers being deposited on top of the 3D host or the side near the electrolyte due to the negligible difference in the 3D structure. And this will lead to the Li accumulation on the surface of the 3D host during long cycling or high deposition rate. Thus, designing 3D hosts with gradient in which Li deposits more easily at the bottom but harder on the top is effective. What's more, the maximum permitted load of Li into the 3D host should also be pay attention to.

(4) 3D ceramic solid electrolyte. One of the main challenges still exists in the design of the Li metal full cell even all problems of LMA are solved, namely the unwanted shuttling of cathode species to the anode as in Li-S and Li-air cells. Solid electrolyte coupled with a MOF-modified 3D LMA may be one of the best ways to solve this issue talked above. Such as a 3D host based on a porous-dense-porous trilayer ceramic electrolyte.

In a word, it is impossible that a single strategy can solve all the problems of Li metal anodes. Nevertheless, more efforts have been put on the way to revive the Li metal chemistry and promote the applications of LMBs.

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