Photo-assisted non-aqueous lithium-oxygen batteries: Preparation and prospect of photocathode materials

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Abstract：Photo-assisted Li-O$_2$ batteries（LOBs）have remained a prominent growing field over the past several years. However，the presence of slow oxygen reduction reaction（ORR）and oxygen evolution reaction（OER），large charging and discharging overpotentials，and unstable cycle life lead to low energy efficiency，thus limiting their commercial application. The rational design and synthesis of photocathode materials is an effective way to solve the above existing problems of photo-assisted LOB systems. Herein，the recent advances in the design and preparation of photocathode materials for photo-assisted LOBs were summarized in this review. First，we summarize the basic principles and comprehension of the reaction mechanism for photo-assisted LOBs. The second part introduces the latest research progress on photocathode materials. The third section describes the relationship between the structure-properties and electrochemistry of different photocathodes. In addition，attempts to construct efficient photocathode materials for photo-assisted LOBs through vacancy engineering，localized surface plasmon resonance（LSPR），and heterojunction engineering are mainly discussed. Finally，a discussion of attempts to construct efficient photocathode materials using other approaches is also presented. This work will motivate the preparation of photocathode materials with stability and efficient photo-assisted LOBs and aims to promote the commercial application of rechargeable photo-assisted LOBs energy storage.

Key words：Photo-assisted；Lithium-oxygen battery；Photocatalysis；Electrode design

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光辅助非水系锂氧气电池：光电阴极的制备与展望

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摘要：在过去的几年里,光辅助锂氧气电池(LOBs)一直是一个突出的发展领域。然而,由于存在氧还原反应(ORR)和析氧反应(OER)缓慢、充放电过电位较大、循环寿命不稳定等问题,导致能量效率较低,从而限制了其商业化应用。光电阴极材料的合理设计和合成是解决光辅助 LOBs 系统上述存在问题的有效途径。本文综述了近年来光辅助 LOBs 光电阴极材料的设计和制备的研究进展。首先,综述了光辅助 LOBs 的基本原理和反应机理。第二部分介绍了光电阴极材料的最新研究进展。第三部分介绍了不同光电阴极结构−性能与电化学的关系。此外,主要讨论了通过空位工程、局域表面等离子体共振(LSPR)和异质结工程来构建高效光辅助 LOBs 光电阴极材料的尝试。最后,还讨论了采用其他的方法来构建高效光电阴极材料的尝试。这项工作将

为制备具有稳定和高效的光辅助LOBs光电阴极材料提供动力，并旨在促进可充电光辅助LOBs储能的商业应用。

关　键　词：光辅助；锂氧气电池；光催化；电极设计

1　Introduction

During the past decades, Li – O$_2$ batteries (LOBs) have been researched as an effective strategy for replacing fossil fuels. The LOBs consist of a lithium anode, oxygen cathode, and electrolyte, which have drawn interest continuously due to the superiority of their ultra-high theoretical energy density ($3500Wh \cdot kg^{-1}$), which has a comparable effect to that of fossil fuels. Among them, the oxygen cathode is a porous structure divided into a substrate and a catalyst, which is mainly involved in the reaction through the O_2(oxygen) in the environment. Nevertheless, the practical applications of LOBs have been restricted by many problems, including high overpotential caused by sluggish reaction kinetics and electrolyte decomposition under high voltage. Designing novel photocathode can guarantee a high discharge ability and long-term stability. According to the basic tenable storage mechanism of O_2 cathode ($2Li^+ + O_2 + 2e^- \rightarrow Li_2O_2, E_\theta = 2.96V \ versus \ Li^+/Li$), displaying great prospects in the fields of energy storage. The discharge and charge processes in LOBs are related to the oxygen reduction reaction (ORR) and the oxygen evolution reaction (OER), respectively. In the ORR reaction, the lithium metal on the negative electrode loses electrons into Li^+ (lithium ions), which are transferred to the porous positive electrode side through the electrolyte and react with O_2 in the air, thus generating the discharge product Li_2O_2(lithium peroxide). Due to the insolubility and insulating nature of the discharge product, a very small amount of Li_2O_2 is dissolved in the electrolyte, while most of Li_2O_2 accumulates in the active sites of the porous positive electrode, blocking the porous positive electrode, thus affecting further reactions; while the OER reaction is the reverse reaction of the ORR reaction, in which the generated Li_2O_2, a discharge product, is decomposed into Li^+, O_2 and e^-. Due to the insulating nature of the discharge product, the voltage required for its decomposition tends to be large, and thus phenomena such as electrolyte decomposition usually occur, resulting in the generation of by-products and affecting the performance of the battery.

Different from conventional LOBs, Photo-assisted LOBs also need an additional photoelectrode to absorb light energy and generate photogenerated carrier pairs. Therefore, the early photo-assisted LOB is a three-electrode structure, but due to the complicated structure of the three-electrode structure, the photoelectrode cannot participate in the reaction at discharge process, and the high cost, the current photo-assisted LOB is a two-electrode structure, and the photoelectrode and oxygen electrode are integrated together, also known as the photocathode. The charge and discharge mechanism of the photo-assisted LOB with three electrodes and two electrodes is shown in **Fig 1**. Where C_R is the reduction product, i. e. , Li$_2$O$_2$; C_O is the oxidation product, i. e. , O$_2$; M is Li and M^{n+} is the Li^+; M_{red} and M_{ox} are the reduced and oxidised states of the redox medium. Light is involved in the charge/discharge process as follows: During the discharge process of photo-assisted LOBs, the photoelectrode is excited by light to generate electrons, which can induce a reduction transition from O_2 to Li_2O_2($2Li^+ + O_2 + 2e^-$ (*Conduction Band*, CB) $\rightarrow$ Li$_2$O$_2$), while the photoexcited holes residing in the valence band (VB) are reduced by the electrons from the external circuit. On the other hand, the photo-assisted charging is a reversible process relative to the photo-assisted discharging. An oxidation reaction ($2h^+$(VB) $+Li_2O_2 \rightarrow 2Li^+ + O_2$) occurs on the photoelectrode, while the photoexcited electrons transfer to the Li anode by the external circuit (**Fig. 2**). [1-7]Therefore, the photo-assisted discharge and charge potential of the assembled LOBs will be de-

termined and influenced by the CB and VB values (*vs*. Li⁺/Li) of unique photocatalysts under illumination. To date, researchers have invested significant efforts and made remarkable progress in the field of photo-assisted LOBs. **Table S1** lists the performance of some photo-assisted LOBs using different photocathode preparation methods to compare the differences in these design approaches. In addition,

other photovoltaic devices have shown promising results[8-17], these photovoltaic device studies also provide insights into the design of novel photo-assisted LOBs. In general, research on photocathodes has focused on substrate-loaded catalysts. Therefore, the subsequent discussion of photocathodes in this paper also focuses on the construction of efficient catalysts.
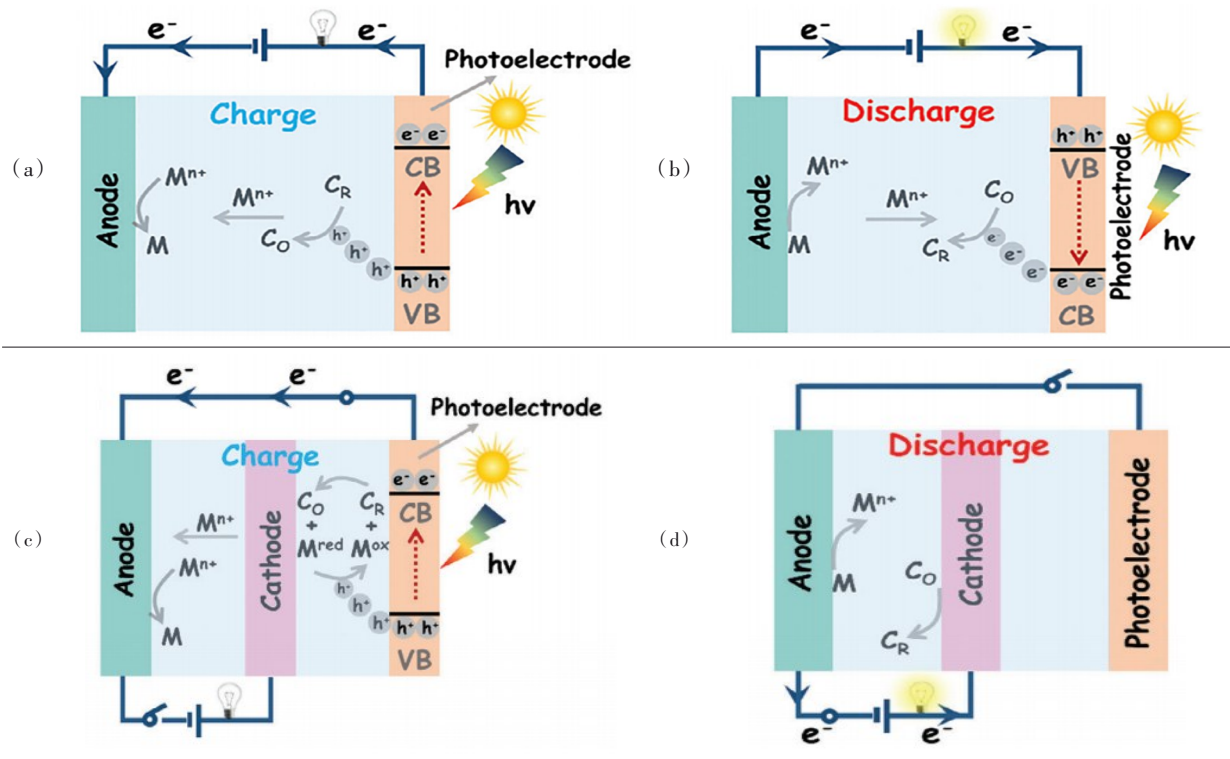

Fig. 1　Working principle of the photo-assisted energy storage device：a，b）The charging and discharging process of a two-electrode device. c，d）The charging and discharging process of a three-electrode device. Note：The direction of the blue arrow represents the flow of electrons. Reproduced with permission from Ref. ［8］，Copyright（2024）Advanced Science.

Discharging process:
Photoelectrode:
$$Photoelectrode \xrightarrow{h\nu} e_{CB}^- + h_{VB}^+$$
$$O_2 + e_{CB}^- + Li^+ \rightarrow LiO_2$$
$$LiO_2 + e_{CB}^- + Li^+ \rightarrow Li_2O_2$$
Anode : $Li - e^- \rightarrow Li^+$
Charging process:
Photoelectrode:
$$Photoelectrode \xrightarrow{h\nu} e_{CB}^- + h_{VB}^+$$
$$Li_2O_2 + 2h_{VB}^+ \rightarrow 2Li^+ + O_2$$
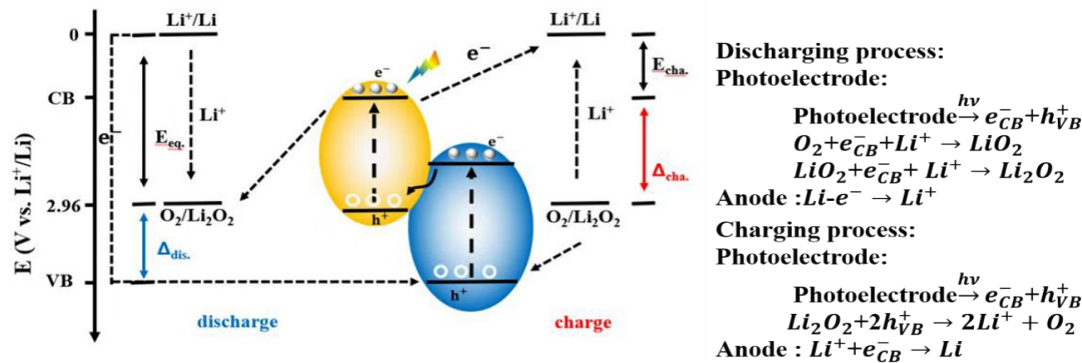**Anode :** $Li^+ + e_{CB}^- \rightarrow Li$

Fig.2　Mechanism diagram of photo-assisted lithium-oxygen battery

　　In this paper, we summarise the methods used to design photocathode for photo-assisted LOBs that aim to design efficient and stable photocathode. Significant research has been conducted with the aim of modifying the properties of photocathode. This mainly includes studies of vacancy engineering, localised

surface plasmon resonance (LSPR), doping and heterojunctions[18] Subsequently, the application of the above semiconductors' modification technology in non-aqueous LOBs is introduced. It is anticipated that this review will provide new insights into the design of photo-assisted LOBs photocathode and serve as a foundation for further investigations and significant advancements in next-generation photocathode design. [19-24]

2   Research progress of semiconductor photocathode of photo-assisted LOBs

Photocathodes for photo-assisted LOBs can be classified into the following three categories according to the type of catalyst: precious metals and their alloys, transition metal oxides, and non-metallic catalysts. Metal oxides and transition metal oxides have been proposed and extensively studied, such as Co_3O_4、Fe_2O_3、ZnO、TiO_2、WO_3、$BiVO_4$. [25] In the reported literature, to ensure the high capacity of photo-assisted LOBs, the lithium sheet is directly used as the negative electrode. All the experimental results demonstrate that the lithium sheet in the oxygen-rich state is easy to corrode, and its discharge capacity and cycle times decrease with the reduction of metallic lithium. In addition, when the photo-assisted LOBs are working, the lithium dendrites generated by the negative electrode will pierce the film and contact the positive electrode, resulting in a short circuit of the batteries. [4,26-30] Meanwhile, the photo/oxygen cathode, the electrolyte, and photocatalytic materials of the photo-assisted LOBs are also very important, especially photocathodes, which is the key to achieving the superior electrochemical performance of the photo-assisted LOBs. The photocathodes, as the place where the electrochemical reactions occur directly, has an extremely large impact on the formation of Li_2O_2. The absorption of O_2 and reaction between O_2 and Li^+ are all proceed with photocathodes catalyst sites. An ideal photocathode should have good catalytic activity, enough space to store discharge products, and sufficient oxygen and charge transport channels. Therefore, the rational design and construction of photocathodes materials play a crucial role in improving the electrochemical performance of photo-assisted LOBs and promoting their commercial application. This section describes the light-responsive semiconductor photocatalytic materials and their work mechanisms, as well as the tremendous efforts to improve their electrochemical properties. At the same time, several methods for improving the properties of existing materials are presented, including vacancy engineering, localised surface plasmon resonance (LSPR), heterojunction engineering, and others, and a detailed review of the various improvement methods is given. Those ideas opened an avenue for overcoming the inherent defects of photosensitive semiconducting materials to further advance the performance of photo-assisted LOBs [30-34].

Semiconductor materials have attracted a lot of attention from researchers because of their unique electronic properties and the band gap between conductors $(10^4—10^6 \Omega \cdot cm^{-1})$ and insulators$(10^{-10}\Omega \cdot cm^{-1})$(**Fig. 3a**). The energy band of a semiconductor consists of a VB filled with electrons and an empty high-energy CB. The gap between the top of the VB and the bottom of the CB is the forbidden band (E_g). The highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) interact to form. [35-37] According to the distribution of carriers, semiconductors can be divided into n-type semiconductors, and p-type semiconductors. Intrinsic semiconductors have no impurities and no defects, the electrical conductivity is mainly determined by the intrinsic excitation of the material, and the Fermi level (E_f) is located between the bottom of the CB and the top of the VB. Correspondingly, for n-type semiconductors, which contain donor impurities, its E_f is close to the bottom of the CB because most carriers are electrons, while for p-type semiconductors, due to hole carriers existing, the E_f moves towards the top of the VB (**Fig. 3b**) . [35,38] When light propagates in a conductive medium, there is an attenuation phenomenon, known as light absorption. Semiconductor materials are usually highly absor-

bent. When a certain wavelength of light (electromagnetic radiation) irradiates the semiconductor material, electrons absorb enough energy to transition from the VB to the CB. At the same time, light absorption leads to the formation of non-equilibrium carriers in the semiconductor, and the increase of the carrier concentration improves the material conductivity. The unique electronic properties of semiconductors have important reference significance for photocatalytic LOBs. [4,37,39] The n-type semiconductor is favorable for the oxidation reaction, and the p-type semiconductor is favorable for the reduction reaction. The light absorption properties of semiconductors mainly depend on the wavelength (wavelengths of ultraviolet light: 200nm-380nm; wavelengths of visible light: 380-760nm; wavelengths of infrared light: 760nm-2500nm). When exposed to light, semiconductor materials usually capture photons with higher energy than E_g to generate e^- and h^+ are coupled via direct or spaced electron transitions: e^- is excited from VB to CB, while h^+ remains in VB. According to classical semiconductor band theory, when the energy of the incident light (h_v) is equal or greater than the band E_g of the semiconductor, electrons (e^-) are excited from the VB into the CB, leaving an equal number of h^+ at VB. That is, the photogenerated electron-hole pairs are produced. It is evident that the isolated e^- acts mainly in the reduction reaction and h^+ acts mainly in the oxidation reaction.
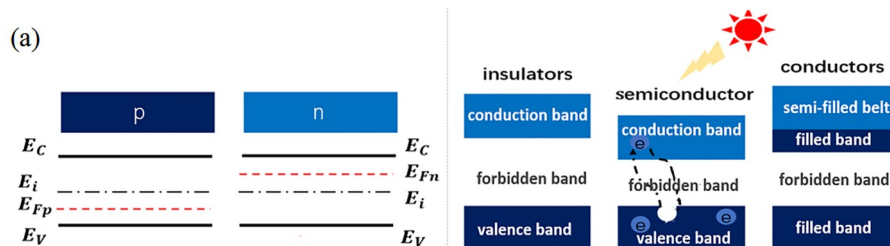
(a)

Fig. 3　（a）Energy band of n-type and p-type semiconductors. （b）Energy band diagrams of insulators, semiconductors, and conductors.

In order to reduce the charging overpotential and prolong the cycle life of the battery, researchers have investigated $CeVO_4$ photocathodes (**Fig. S1–S2**). [40] The results show that the photo-assisted LOB composed of this photocathode exhibits a better performance. This provides a reference for designing efficient photocathode materials. In recent years, perovskite, graphene, and metal-organic framework (MOF) materials have attracted much attention, making them promising candidates for photocathodes in photo-assisted LOBs due to their unique photovoltaic properties. They also showed the desired results in photo-assisted LOBs. For example: Reduced overpotential, extended cycle life, improved rate performance, and so on [41-43]. However, only the carriers at the surface of the semiconductor can play the role of redox reaction. [44] In fact, only a small number of photogenerated carriers can actually participate in the reaction due to their high rate of complexation (~ 90%). Therefore, inhibiting the recombination of photogenerated carriers is the primary direction to improve the performance of photo-assisted LOBs.

2.1　Construction of the heterojunction

The high carrier recombination rate and limited catalytic ability of a single semiconductor material have severely limited the performance of photo-assisted LOBs. Therefore, there is an urgent need for a method to construct efficient photocathodes to improve the performance of photo-assisted LOBs. The current popular method to construct an efficient photocathode is to construct a heterostructure consisting of two semiconductors. According to previous works, through a built-in electric field was induced at the junction of the heterojunction, facilitating the separation of photoexcited carriers, superior light harvesting capability and high electronic conductivity. Thus, the heterojunction photocatalyst has become an inevitable choice and has attracted an increasing attention due to its wide spectral response and high separation efficiency of the photogenerated charge

carriers.

The common types of heterostructures in photocatalytic LOBs are type II heterojunction, Z - scheme, S - scheme(**Fig. 4**) (For a detailed description of **Fig. 4**, see Supporting Information). Several researchers have improved the performance of photo-assisted LOBs by constructing a type II heterostructure photocathode. The construction of the type II heterojunction can effectively inhibit the compounding of photogenerated carriers, thus accelerating the cathode reaction kinetics (**Fig. S3−Fig. S5**). In order to improve the redox capacity and increase the photocatalytic activity, the researchers further constructed a Z-type heterostructure photocathode in a photo-assisted LOB (**Fig. S6−Fig. S7**). Researchers have also investigated S-type heterostructure photocathodes. Photo-assisted LOBs consisting of this heterostructure demonstrated multiplicative performance and cycling stability. [45]
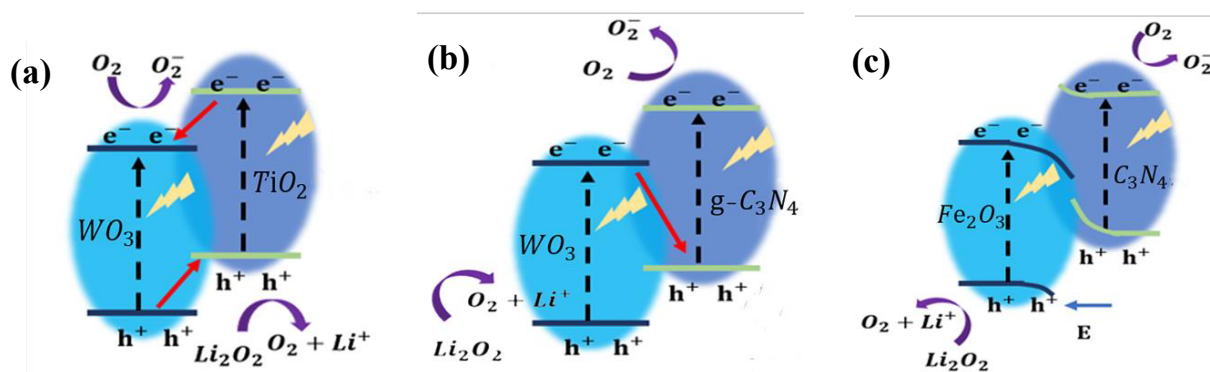

Fig.4　Schematic diagram of photoexcited electron-hole transfer process：（a）II type heterojunction and（b）Z-scheme transfer （c）S-scheme transfer.

2.2 Localized surface plasmon resonance

Based on physical mechanisms, LSPR-assisted electrochemistry can be usually classified into three types: photothermal, enhanced electric field, and hot electrons, as shown in **Fig. 5**. In mechanism A, a high lattice temperature of the plasmonic material is induced by the relaxation of hot carriers via electron – electron and electron – phonon scattering. Mechanism B involves a highly enhanced electromagnetic field, which could activate nearby species or generate vacancies in the CB of adjacent semiconductors. In mechanism C, the transfer of hot carriers occurs from the plasmonic nanostructure to an adjacent species, which is critical for direct plasmon-driven electrocatalysis over pure plasmonic electro-catalysts and indirect plasmon-assisted electrocatalysis over electroactive species or semiconductors.
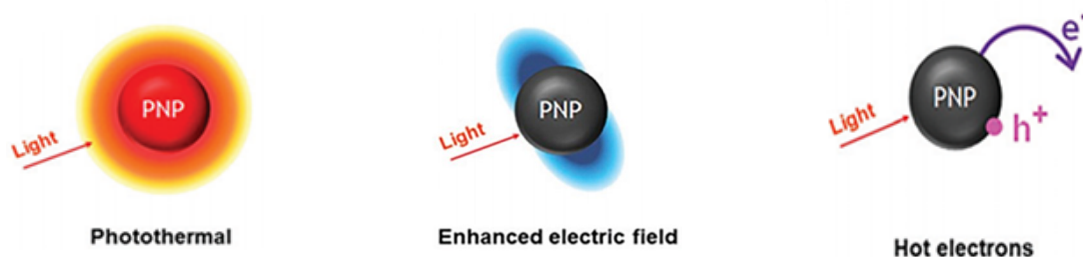

Fig.5　Main physical mechanisms on plasmonic metallic nanostructures arising from LSPR（a-c）.（a）Photo-induced heating of plasmonic materials.（b）Enhancement of the electromagnetic field.（c）Photo-induced electron-hole pair generation and hot electron injection Reproduced with permission from Ref.［46］，Copyright（2020）American Chemical Society.

Many transfer paths of hot carriers have been widely investigated. The electron transfer process at the interface between a plasmonic material and semiconductors, as shown in **Fig. 6**, including the indi-

rect and direct pathways. The hot electrons are transferred to or are generated in the CB of semiconductors to participate in reduction reactions, or are transferred to the external circuit, producing a photocurrent and increasing the separated holes for oxidation reactions. The Schottky barrier formed at the interface of a metal and a semiconductor could act as an efficient electron trap to prevent the $e^- - h^+$ recombination. While the electrons transfer to the reactant species upon plasmon decay of p-type semiconductors due to the presence of most hole carriers. The holes transfer to the VB of the semiconductor and then to the external circuit. [46-49]. Au nanoparticles are a common plasmonic material that has been extensively studied(**Fig. S8**). [50] Photo-assisted LOBs employing this plasma material have also demonstrated promising performance, which provides a new direction for the design of efficient photocathodes.

2. 3 Vacancy engineering

Defects can be divided into three types: point defects (vacancies, interstitial atoms), line defects (dislocations), planar defects (stacking faults, twin boundaries in polycrystals). As one of the most prevalent point defects, vacancies are formed when atoms escape from the lattice. [51-53] This process would change the electronic and geometric structures of materials, offering a potential opportunity to optimize the photocatalytic performance of materials. In general, the vacancy engineering largely via three strategies, that is, extending the light absorption range, improving photo-generated charge separation and transfer and optimizing photocatalytic reactions at the surface of catalysts. Therefore, focused on the roles and mechanisms of vacancies in enhancing light absorption, promoting charge separation and tuning surface catalytic reactions is important.

Vacancy engineering of photocatalysts improves photoreaction kinetics due to the following reasons: (1) uncoordinated active sites induced during the formation process of vacancies are benevial to reactant adsorption and activation; (2) it regulates the surface reaction pathway and the selectivity of photocatalytic reactions by different adsorption and
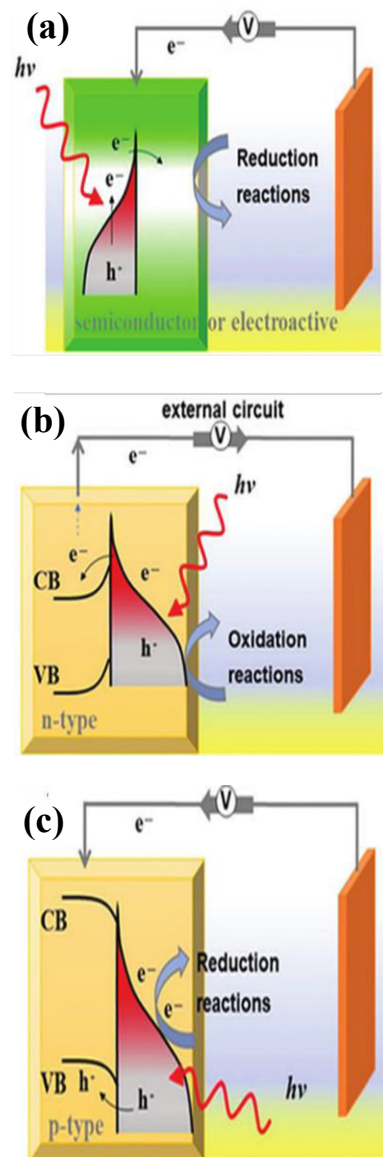

Fig.6 （a）Hot electrons transfer to semiconductors for reduction reactions. （b）Hot electron-injection to an n-type semiconductor facilitates oxidation reactions over plasmonic materials and/or n-type semiconductors. （c）Hole transfer to a p-type semiconductor with a wide band gap leaving electrons for reduction reactions over plasmonic materials. Reproduced with permission from Ref.［47］, Copyright（2021）Royal Society of Chemistry.

dissociation states of reaction intermediates on vacancy sites. [54-55] Although the presence of vacancies improves the photoelectrochemical performance of the photocathode, how to detect the presence of vacancies remains a problem. The researchers proved the existence of vacancies using XPS and XRD, respectively (**Fig. S9−Fig. S11**). Photocathodes with

oxygen vacancies have been investigated by experimentalists due to their easily constructible characteristics. This photocathode with oxygen vacancies has demonstrated good performance in photo-assisted LOBs, which also provides a new idea for constructing efficient photocathodes(**Fig. S12–Fig. S14**).

2.4 Other methods

In addition to the above methods, there are other methods for constructing an ideal photocathode for application in photo-assisted LOBs, including: controlling the size of the catalyst on the photocathode, adjusting the major crystalline surfaces of the catalyst on the photocathode, and constructing a photocathode material with photomagnetic properties (**Fig. S15–Fig. S17**). The photocathodes prepared by these methods have demonstrated good performance in photo-assisted LOBs. Obviously, these methods open up a new path for the construction of highly efficient photo-assisted LOBs.

3 Conclusions and Outlook

In conclusion, photo-assisted non-aqueous LOBs have received extensive attention recently due to their high energy density and round-trip efficiency. Meanwhile, researchers have improved semiconductor materials using vacancy engineering, plasma resonance excitation, and heterojunction engineering, and have already made significant breakthroughs in promoting redox kinetics and reducing overpotentials in photo-assisted LOBs. Therefore, the concept of photocatalysis can be extended to other metal-air batteries. Although the development of

photo-assisted LOBs is at a rapid stage, their photocatalytic mechanism still needs a lot of effort. As shown in **Fig S18**, the predicament of the current photo-assisted LOB and the direction of future research are discussed. At present, there are many problems in the operation of photo-assisted LOBs, including high photo-generated carrier recombination rate, decomposition of electrolyte caused by long-term light, premature failure of lithium anode, lack of photocatalyst excited by long-wave light, and unsatisfactory cycling performance, rate performance and discharge capacity. Therefore, it is of utmost importance that we dedicate concerted efforts towards advancing this domain by focusing on the development of stable electrolytes, or alternatively, exploring the utilization of gel or solid electrolytes. Furthermore, there is a pressing need to design photocatalysts capable of being efficiently excited by long-wavelength light while maintaining high performance. Additionally, the incorporation of alloy anode materials and the implementation of innovative approaches beyond traditional boundaries are essential. Such comprehensive strategies are crucial for obtaining better results in this research endeavor. For the study of photocatalytic LOBs, this will involve an interdisciplinary knowledge of photochemistry, materials chemistry, electrochemistry, computation, spectroscopy, and surface science. The collaboration between researchers from different backgrounds is expected to overcome key scientific and technical issues for the practical application of photovoltaic cells in the future.

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