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Recent progress on electrodeposition of metal/alloy films or coatings in deep eutectic solvents

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Abstract: The development of low-energy consumption and environmentally friendly electrodeposition of metal/alloy films or coatings is presently one of the primary topics for the research community. For this purpose, deep eutectic solvents (DESs) are valued as electrolytes for their advantages of low operating temperature and wide electrochemical windows. At present, there is large amount of literature on this emerging field, but there are no specialized reviews of these studies. Here, after a brief introduction of DESs' concept and history, we comprehensively reviewed the lastest progress on the metal/alloy electrodeposition in DESs. Additionally, we discussed the key influence factors of the electrodeposition process and analyzed the corresponding mechanisms. Based on these, we emphasized the importance of the establishment of predictive models for dealing with the challenges in large-scale applications.

Key words: recent progress; electrodeposition; metal/alloy films; deep eutectic solvents; electrode process

1 Introduction

Environmental conservation is one of the hottest topics in recent years, which prompts the upgrading of metallurgical industry from the traditional energy-intensive production to a green and energy-efficient direction. One of the available ways for this goal is the near-room temperature electrodeposition drove by the "green electric energy" generated from the sustainable energies such as solar energy and wind energy. The discovery of the deep eutectic solvents (DESs) is a significant breakthrough in this field because they are ideal electrolytes for the low-temperature electrodeposition of metals and alloys.

DESs are typically defined as the mixtures combining metal salts or hydrogen bond donors (HBD) with hydrogen bond acceptors (HBA) in certain molar ratios mainly through hydrogen bonding [1,2]. The properties of DESs can be easily adjusted by changing HBA and HBD (Fig. 1(a)), which greatly extends their application prospects, including electrodeposition [3−5], extraction and separation [6−8], battery technology [9−11], and pharmaceuticals research [12−14]. As shown in Fig. 1(b), DESs were discovered by ABBOTT et al [15,16] about 20 years ago. They found that a mixture of choline chloride (ChCl) and zinc chloride in a molar ratio of 1:2 had a very low-melting point (23−25 °C). Since then, the unique characteristics of DESs attracted widespread

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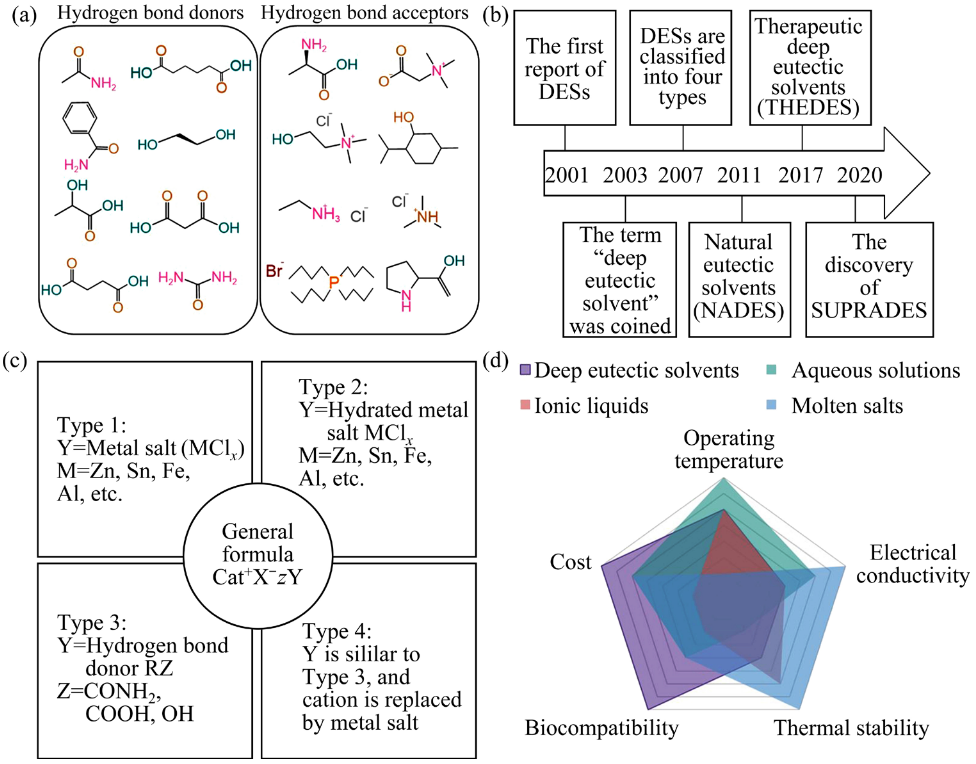

Fig. 1 Overview of DESs: (a) Several common hydrogen bond donors and acceptors; (b) Milestones of history of DESs; (c) General formula of DESs and four classifications; (d) Performance comparison of deep eutectic solvents, aqueous solutions, ionic liquids, and molten salts (The inward direction represents a high operating temperature)

attention and a series of mixtures consisting of salts and HBDs were developed [16]. In 2018, a new class of DESs that is insoluble in both water and lipid phases within cells was discovered and named as "natural deep eutectic solvents" (NADES). This is an important natural compound extractor [17]. In 2015, AROSO et al [18] introduced the concept of "therapeutic deep eutectic solvents" (THEDES) and attempted to combine three active pharmaceutical ingredients. It is worth mentioning that in 1998, STOTT et al [19] reported a similar synthetic method. WANG et al [20] further confirmed that THEDES could enhance permeability, enabling the development of new transdermal delivery systems. In recent years, another new concept of DESs named "supramolecular deep eutectic solvents" (SUPRADES) was introduced that employs cyclodextrins and their derivatives as HBA [21]. Up to date, research on this area is rapidly developing [22−25].

Among many applications of DESs, electrodeposition is one of the earliest and most important applications. ABBOTT et al [15,16] deposited zinc in the eutectic mixture of $ZnCl_2$ and ChCl. Subsequent research found that only metal salts with lower melting points could form DESs, which limited the further applications of DESs. In order to enable more metals to electrodeposit in DESs, a series of hydrates were introduced. The result showed that the melting point of the mixture decreased, but only cobalt and chromium were electrodeposited from them. In subsequent studies, HBD and HBA were introduced. The DESs have excellent adjustability and solubility for a variety of metal salts and oxides, enabling the electrodeposition of multiple metals. As of now, multiple metals can be electrodeposited in DESs.

Although, over the years of development, the variety of DESs has significantly expanded, all of them can be described by a general formula: $\mathrm{Cat}^+\mathrm{X}^-z\mathrm{Y}$, where Cat is an ammonium, phosphonium, or sulfonium, X is a Lewis base, usually a halide anion, Y is a Lewis acid or Brønsted acid, and z is its number. By specifying these components, we can obtain the four types of DESs shown in Fig. 1(c) [26]. Type 1 DESs are similar to the widely known ionic liquids formed by metal halides and imidazole. In Type 1, Y is MCl$_x$, M represents metal, and x is the number of Cl ions. This type is limited by the number of anhydrous metal halides with a low melting point. Type 2 DESs decrease the melting point and increase the number of metals by adding hydrated metal halides and choline chloride, but water hinders the deposition of active metals. Type 3 DESs are a group of tunable DESs and are also the most concerned type of DESs. They exhibit good controllability and solubility. Type 4 DESs are composed of metal halide hydrates and HBD, and cationic complexes are generated in such DESs, which increase the concentration of metal ions on the electrode surface. Therefore, metal electrodeposition occurs more easily [27,28].

As electrolyte used in the electrodeposition process, DESs’ structure results in lower lattice energies, hence they have lower melting points. Some common components of DESs, such as choline and urea, are widely found in nature or have already been produced extensively for industrial and agricultural purposes. These common constituents have some advantages such as low cost and biodegradability. Additionally, DESs have high solubility for metal salts and wide electrochemical window. As shown in Fig. 1(d), in comparison to other electrolytes commonly used for electrodeposition, DESs exhibit notably superior performance. However, this field has only received relatively brief descriptions in some review chapters [2,17,29], making it essential to provide a dedicated summary and discussion.

Here, we systematically surveyed the literature on the electrodeposition of metals and their alloys, such as typical transition metal elements, rare earth elements, and precious metal elements, in DESs. Meanwhile, some classical metal electrodeposition processes and their mechanisms were discussed in detail. Then, the important influence factors, including water content, temperature, current mode, and additives, on metals electrodeposition were analyzed and the action rule to the morphology, current efficiency, adhesive strength of deposits and the corresponding mechanisms were summarized. Besides, the bottlenecks that limit the advance of this emerging field were proposed and the possible solutions were also discussed from the perspectives of the basic research and industrial application on the metal electrodeposition in DESs. We believe that this review will be instructive for the advance of this emerging field.

2 Electrodeposition in deep eutectic solvents

In 2008, ABBOTT et al [30] firstly characterized the physical properties of numerous DESs, such as viscosity, conductivity, and solubility of various metal species in them, and proposed the possibility of electrodeposition in these DESs. Up to now, there are a lot of reports on the metal electrodeposition in DESs.

2.1 Typical transition metals and their alloys

2.1.1 Nickel and its alloys

Nickel and its alloys are important engineering materials for their high hardness and good corrosion resistance. In 2006, ABBOTT and MCKENZIE [29] reported nickel electrodeposition in ChCl/urea and ChCl/ ethylene glycol (EG) DESs at a 1:2 molar ratio and obtained bright nickel metal coatings on copper and aluminum substrates. In 2010, FLOREA et al [31] continued the research of ABBOTT and MCKENZIE [29], using various DESs to deposit nickel on substrates such as copper and magnesium. The coating on the copper substrate is relatively uniform and dense, while the coating on the magnesium substrate is slightly porous. This provides a new coating method for active metals that tend to passivate in water. For the first time, they reported the electrodeposition of Ni in a DES containing $NiSO_4$. It is worth noting that they also investigated two different alloys, Ni−Mo and Ni−Sn, and observed their deposition behavior in different DESs and at different current densities. It was found that the content of Mo decreased with increasing current density, while the content of Sn remained relatively stable. Similarly, in DES with propylene glycol as HBD, the phenomenon of Mo content changing with cathode potential was also observed. This is explained as the increase in the rate of intermediate reactions inhibiting the reduction of Mo [32]. Apart from these alloys, GENG et al [33] extracted a Cu−Ni alloy from nickel matte using a two-step process. They first conducted chlorination roasting and then used DESs for leaching, followed by an electrodeposition method.

The study of nickel-containing ternary alloys electrodeposited in DESs is noteworthy. For instance, in 2016, YANAI et al [34] deposited

Fe−Co−Ni alloy from a mixture of hydrated chlorides of respective elements, potassium chloride, and ethylene glycol, and investigated the influence of elemental molar ratios on the current efficiency, coercivity, and saturation magnetization. Subsequent research [35] within this system led to the synthesis of Fe−Co−Ni−NS nitrosulfide ternary metal materials, serving as stable and efficient catalysts for the oxygen evolution reaction. In similar DESs, successful deposition of Fe−Ni−Cr ternary alloys with nickel content ranging from 21 at.% to 32 at.% was achieved on SA-106 carbon steel [36]. Additionally, in ChCl/EG DES, direct deposition of Fe−Co−Ni thin films was possible, where deposition potential could control the elemental composition, corrosion resistance, and magnetism, albeit with a narrower range of compositional control compared to previous systems. In Ref. [37], Ni−Mo−Cu ternary alloy was electrodeposited from nickel matte in ChCl/EG DES, and further investigation revealed its potential as a catalyst for the hydrogen evolution reaction in alkaline aqueous solutions.

In addition to metals and their alloys, compound material coatings have also been studied. In 2015, LI et al [38] electrodeposited nickel coatings containing micrometer and nanometer-sized SiC particles using ChCl/EG DES as the electrolyte. Compared to pure nickel, these coatings exhibited superior tribological properties. In 2022, PROTSENKO et al [39] investigated Ni−TiO_2 coatings electrodeposited in DESs, and the results indicated that not only the microhardness and corrosion resistance were improved, but also the coatings exhibited photocatalytic activity for the photochemical degradation of methylene blue organic dye in aqueous solution. These studies have expanded the application scope of nickel electrodeposition technology in DESs.

Comparative studies of nickel electrodeposition in DESs and aqueous solutions have been conducted [40]. The nickel deposition process exhibited significant differences between DESs and aqueous solutions. For example, although the deposition rates and corrosion resistance of the films obtained from two electrolytes were similar, the surface morphology of the deposits was entirely different. Moreover, SRIVASTAVA et al [41] found that the films deposited from DESs exhibited higher microhardness than those obtained in aqueous solutions. GU et al [42] investigated the relationship between the applied voltage and the morphology of deposits. They prepared fully dense nanocrystalline nickel films under a voltage of 1 V on brass substrates in ChCl/EG-based DESs. The nanocrystalline nickel films demonstrated high strain sensitivity and improved hardness. Additionally, as an extension of Abbott's research, various additives, such as nicotinic acid, methyl nicotinate, boric acid [43], and water [44], were introduced into the system, resulting in bright nickel coatings in these studies.

There is a lot of research related to nickel electrodeposition in DESs, ranging from metals to composite materials. The products of deposition have improved physical properties compared to the products in aqueous solution systems, and there are also changes in function. This adjustable performance well reflects the potential of DESs.

2.1.2 Zinc and its alloys

Zinc is commonly used for the corrosion protection of steel. The electrodeposition of zinc and zinc alloys in aqueous solution, typically involves toxic and corrosive cyanide acid baths, non-alkaline cyanide baths, and acyl chloride baths. These systems often exhibit hydrogen evolution phenomena. For instance, a study [45] revealed that zinc coatings deposited via electroplating contain approximately 5.79×10^{-6} (mass fraction) of hydrogen, thereby affecting the performance of the zinc plating. However, in DESs, such occurrences can be circumvented as DESs theoretically can be water-free.

In 2011, ABBOTT et al [46] conducted deposition of zinc, tin, and Zn−Sn alloys in ChCl with EG and urea, respectively. It was found that the zinc electrolyte contained various active zinc species, including $ZnCl_3^-$, $Zn_2Cl_5^-$, and $Zn_3Cl_7^-$. ABBOTT et al [47] further explored the differences in the deposition processes in these two DESs. Double-layer capacitance studies indicated that the adsorption of chloride ions on the electrode's surface was the origin of the difference. They also investigated the effects of ethylenediamine, ammonia, and acetonitrile on zinc deposition, resulting in a bright zinc film.

Subsequently, SONG et al [48] presented a different perspective, suggesting that in the presence of ethylenediamine and ammonia, concentrations of lower-coordinated active species, which are easier to nucleate and grow, increase. VIEIRA et al [49] discovered that the zinc electrodeposition process on different electrode materials in a system containing ethylene glycol varies. This may be because zinc electrodeposition occurs through the reduction of zinc-containing intermediates. More recently, CIHANGIR et al [50] have studied zinc electrodeposition mechanism in different DESs (Fig. 2(a)). This research revealed the deposition mechanism of zinc in ChCl/EG. In this electrolyte, there are competitive reduction reactions between EG and zinc ions. The reduction products of EG can further react with zinc ions to generate ethyl glycolate attached to the electrode surface, and subsequently reduce to form zinc films.

In addition to depositing zinc in solutions containing chloride salts, electroplating using oxides as raw materials has also been attempted. As shown in Fig. 2(b), XIE et al [51] successfully prepared Cu−Zn alloy through electroplating using ZnO and CuO dissolved in a DESs as raw materials. Subsequent studies [52−54] have elucidated the mechanism of the reaction. Oxides dissolve in DES and form complex species $[\mathrm{CuO{\cdot}Cl{\cdot}urea}]^-$ and $[\mathrm{ZnO{\cdot}Cl{\cdot}urea}]^-$, which deposit on the cathode to generate alloys and $[\mathrm{ChCl{\cdot}O{\cdot}urea}]^{2-}$, the latter generates oxygen in the anode reaction. On the cathode, chlorine gas is only produced when the voltage is lower than the decomposition potential of the electrolyte.

Two years later, this approach was further developed by BAKKAR and NEUBERT [53]. They used a DES with a ChCl/urea/EG molar ratio of 1:1.5:0.5 to selectively leach zinc species from the cupola furnace dust containing various metal oxides. As implied by the cyclic voltammetry plot in Fig. 2(c), it is possible to obtain relatively pure zinc through electrochemical methods. In industrial zinc smelting, sphalerite (predominantly ZnS) serves as the primary zinc source, contrasting with ZnO-derived feedstocks. Recently, WANG et al [54] achieved the dissolution of ZnS in a DES and electroplated pure zinc by adding succinic acid (Fig. 2(d)). Although many parameters should be optimized further, this method holds the potential to simplify and reduce energy consumption in industrial zinc extraction.

In the case of zinc, DESs once again demonstrated excellent solubility for metal species. For zinc electrodeposition, this characteristic helps to find a lower-cost and shorter process.

2.1.3 Cobalt and its alloys

Cobalt exhibits strong ferromagnetism, and cobalt alloys have superior magnetic properties, making them an ideal choice for high-performance magnetic recording applications. Some cobalt

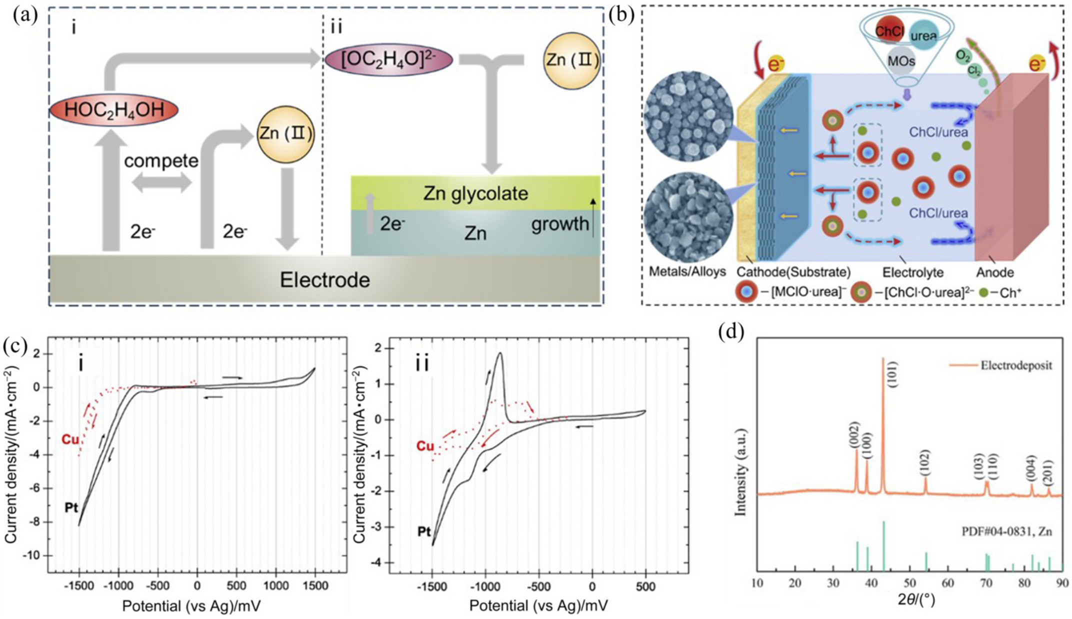

Fig. 2 (a) Schematic diagram of electrodeposition mechanism of zinc in ChCl/EG DES (i) before and (ii) after electrode is covered with zinc [50]; (b) Schematic illustration of reduction of metal oxides to metal in DES [51]; (c) Cyclic voltammetry of platinum and copper electrodes in: (i) blank DES and (ii) DES with cupola furnace dust [52]; (d) X-ray diffraction spectrum of zinc obtained by direct electroplating of ZnS [54]

alloys, such as Sm−Co alloys, can be electrodeposited from aqueous solutions. However, hydrogen evolution occurs during electrodepositon due to the limited electrochemical window of water, resulting in low current efficiency (below 20%) [55]. This situation can be avoided in DESs, as they can be anhydrous. Therefore, DESs have emerged as new supporting electrolytes, offering potential solutions to deal with this challenge.

Reports on the electrodeposition of cobalt in DESs have a shorter history compared to the reports on nickel and zinc. In 2011, GÓMEZ et al [56] reported the electrodeposition of cobalt and samarium in a ChCl/urea-based DES. They suggested that cobalt electrodeposition follows the typical nucleation and three-dimensional growth process. In 2014, LI et al [57] conducted a more detailed investigation on cobalt electrodeposition, including the effects of electrode potential and temperature on the electrodeposition process. They found that lower temperatures and lower overpotentials favored the deposition of uniform and dense coatings. In 2015, COJOCARU et al [58] examined the cathodic processes of Co^{2+} ions in four ChCl-based DESs with different hydrogen bond donors. They qualitatively compared the electrochemical behaviors of these DESs and suggested that hexahydrated cobalt chloride might have better electroplating performance compared to cobalt chloride as the cobalt source.

Reports on the addition of water have demonstrated its beneficial effects. In 2020, AL-MURSHEDI et al [59] reported the electrodeposition of cobalt in a ChCl/EG with water. They found that the inclusion of water in the electrolyte led to larger peak currents in the redox process, which shifted in the positive direction. Research on cobalt electrodeposition continues to advance, exploring factors such as substrate properties, potential ranges [60], and the influence of organic additives [61].

2.1.4 Chromium and its alloys

Chromium is widely used in various industrial applications due to its strong corrosion resistance, wear resistance, and hardness. The commonly used electroplating process for chromium involves depositing Cr(VI) from aqueous solutions. However, this process faces issues such as low current efficiency and hydrogen embrittlement due to the hydrogen evolution reaction. Additionally, Cr(VI) is highly toxic to humans. Therefore, researchers have sought alternatives, such as Cr(III), to address these concerns. However, the reduction potential of Cr(III) is more negative than that of hydrogen ions, leading to reduced current efficiency in electroplating in aqueous solutions due to hydrogen evolution. Moreover, the aggregation of Cr(III) species also inhibits the progress of the reduction reaction [62]. In DESs, electrodeposition can avoid hydrogen evolution side reactions. Also, no aggregation phenomenon has been observed in DESs.

The electrodeposition of Cr(III) in DESs was reported by ABBOTT et al [63]. Chromium deposition was achieved in two DESs: ChCl+$CrCl_3\cdot 6H_2O$ and $CrCl_3\cdot$ChCl/urea+$CrCl_3\cdot 6H_2O$. The obtained products were amorphous and crack-free, but the wear resistance was not ideal. In 2016, AL-BARZINJY [64] obtained deposits with hardness close to that of chromium in a mixture of urea and $CrCl_3\cdot 6H_2O$. In these studies, the combination with water seemed to positively influence the deposition performance. MCCALMAN et al [65] reported the influence of water content on the solution. Surface analysis indicated that the reduction current was primarily affected by the physical properties of the solution, such as conductivity, rather than the morphology of chromium species. In later research, BOBROVA et al [66] found that high water content led to an increased hydrogen evolution reaction. Therefore, under a moderate water content and temperature, a gray chromium deposition layer was obtained. Similar conclusions were reached by NABLO and DELA PENA [67], who achieved the best black deposition layer with a maximum of 45% current efficiency in a solution with a $CrCl_3/H_2O$ mass ratio of 1:6.

In ChCl/EG DES, chromium electrodeposition was also performed. A study [68] found that crack-free Ni−Cr alloys were obtained in this system and discovered that the chromium content in the alloy could be controlled by the electrolysis time. Interestingly, the current efficiency was determined to be 78% through electrochemical quartz crystal microbalance, which is significantly higher than other systems. In the same solvent, Fe−Cr nanocrystalline alloys could also be prepared by electrodeposition, and iron promoted the reduction of chromium. This is a simple path to obtain corrosion-resistant coatings [69].

2.1.5 Copper and its alloys

Unlike many metals mentioned earlier, copper can be electrodeposited in aqueous solution. Conventional copper electroplating processes typically involve highly corrosive electrolytes ($pH<1$), posing challenges such as increased energy consumption and environmental pollution issues. DESs, which are more environmentally friendly, are thus being looked upon to address this concern.

In 2009, ABBOTT et al [70] conducted the first attempts on electrodeposition of copper and its alloys in DESs. The research revealed that progressive nucleation resulted in smooth deposits, while lower copper concentrations led to rougher deposits due to instantaneous nucleation. This is also the first reported instance of electrodeposited copper composite materials in DESs. They found that this may be due to the higher viscosity, which results in a more stable suspension of composite materials such as Al_2O_3 and SiC, thus providing an advantage to DESs. They successfully prepared copper-based composite materials containing Al_2O_3 or SiC in DESs. Subsequently, POPESCU et al [71], and GHOSH and ROY [72] separately achieved copper deposition in different DESs and characterized the morphology of the obtained products. In 2014, SEBASTIÁN et al [73] reported that Cu(I) could be directly electrodeposited from the solution, while Cu(II) needed to be reduced to Cu(I) firstly.

In 2019, ANGGARA et al [74] reported the direct extraction of copper from copper sulfide minerals using DES electrolytes. Both ChCl/urea and ChCl/EG-based DESs were tested, and the morphological forms of Cu active species in the solutions were analyzed. It is worth noting that iron and copper can dissolve together, but the deposition process exhibits selectivity.

2.2 Precious metals and their alloys

Precious metals have irreplaceable industrial applications. Methods for the extraction, refining, and electroplating of precious metals in DESs have been extensively studied.

ABBOTT et al [75] deposited silver coatings and silver composite coatings with SiC or Al_2O_3 in ChCl/EG-based DES. Subsequently, more precious metals have been electrodeposited in DESs. For example, in 2013, LANZINGER et al [76] studied the electrodeposition of palladium in DES. It was reported that dense palladium was obtained through a constant current electrolysis in ChCl/urea. BERNASCONI et al [77] reported the electroplating of ruthenium in ChCl/EG with the addition of sulfamic acid in 2018.

Extracting precious metals from ores and E-waste is a difficult process, but the great industrial demand for them continues to drive efforts to extract these metals. Gold extraction typically involves processes with significant pollution, such as cyanide. To address this issue, in 2015, ABBOTT et al [78] reported a process (Fig. 3) for extracting gold and silver from sulfide ores using the oxidative properties of iodine in DES systems.

One feature of precious metals is their wide-ranging applications in the field of catalysis. Catalytic performance is closely related to the microstructure, making the controlled synthesis of nanomaterial shapes in DESs a research focus. In 2013, WEI et al [79] deposited triambic icosahedral platinum nanocrystals using square wave potential.

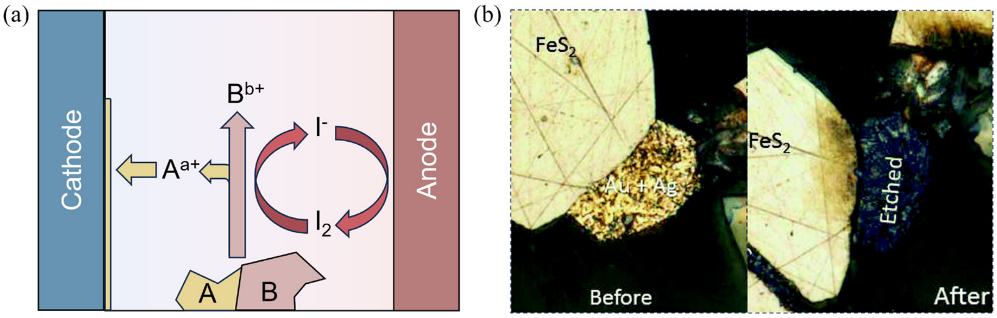

Fig. 3 Recovering precious metals using DESs: (a) Schematic diagram of separation and recovery of elements A and B in DESs using iodine as electrocatalytic oxidant; (b) Optical microscopy of sulfide ores before and after recovery of precious metals [78]

LIAO et al [80] obtained star-shaped gold nanoparticles by reducing $HAuCl_4$ (Fig. 4(a)). It was discovered that the shape of nanoparticles could be modified by altering the water content slightly. As shown in Fig. 4(b), LI et al [81] expanded the range of water content, resulting in different morphologies of gold nanocrystals, such as Au nanostars, Au nanodentists, and Au hierarchical nanoflowers. In these experiments, Au nanocrystals exhibited high electrocatalytic activity.

2.3 Rare-scattered metals and their alloys

Rare-scattered metals, such as tin, indium, lead, bismuth, and germanium, are characterized by their low abundance in the Earth's crust, non-concentrated distribution, or difficulties in extraction. This paper selects the representative metal tin and describes the research progress in its electrodeposition and electroplating.

Tin is widely used in industries due to its excellent wear resistance, ease of soldering, and corrosion resistance. Past research found that tin can be electrodeposited in various aqueous electrolytes [82]. However, to enhance the performance of the coatings, additives such as cyanides and organic compounds are introduced into the electrolyte [83], which are toxic. Because it is safe and eco-friendly, electroplating in DESs has also garnered attention. Research on the electroplating of tin in DESs began to deposit tin, zinc, and Zn−Sn alloys in ChCl/EG-based DES [84]. In 2012, PEREIRA et al [85] introduced additives such as EDTA, HEDTA, and Idranal VII into the system which had distinct effects on the film's performance. In 2013, SALOMÉ et al [86] studied the influence of different HBDs on the deposition performance. In 2020, CAO et al [87] obtained dense Sn films in ChCl/urea-based DES and further analyzed the electrochemical behavior of Sn. They obtained a conclusion that the electrodeposition of Sn(II) was a quasi-reversible and one-step reduction process.

Sn−Bi and Sn−In are widely studied alternatives to Sn−Pb solder materials with low melting point. In 2014, GAO et al [88] reported the co-deposition of Sn−Bi alloy by adding $BiCl_3$, $SnCl_2$, and H_3BO_3 in a ChCl/EG-based DES. This study demonstrated the feasibility of electro-depositing Sn−Bi alloys near the eutectic composition. In 2019, ANICAI et al [89] reported the uniform deposition of Sn−In alloy on copper using a ChCl/EG electrolyte. The content of indium increased with increase of temperature and achieved a maximum indium content of 65%.

Some ternary alloys can also serve as lead-free solders, such as Sn−Cu−Ni and Sn−Ag−Cu. In 2021, ROSOIU et al [90] obtained Sn−Cu−Ni alloy through electrodeposition in two different types of DESs, namely ChCl/EG and ChCl/urea. Experiments demonstrated that it possesses good corrosion resistance and solderability. Inspired by this research, HUANG et al [91] used a similar method to obtain Sn−Ag−Cu alloy and explored the effect of potential on the deposition products, obtained a solderable coating of Sn−Ag−Cu ternary alloy. This method has shown excellent potential in the preparation of ternary lead-free solders.

2.4 Rare earth metals and their alloys

Rare earth elements are in high demand in high-tech industry and are considered among the most important raw materials. Some important functional materials can be obtained through the electrodeposition of rare earth elements. However,

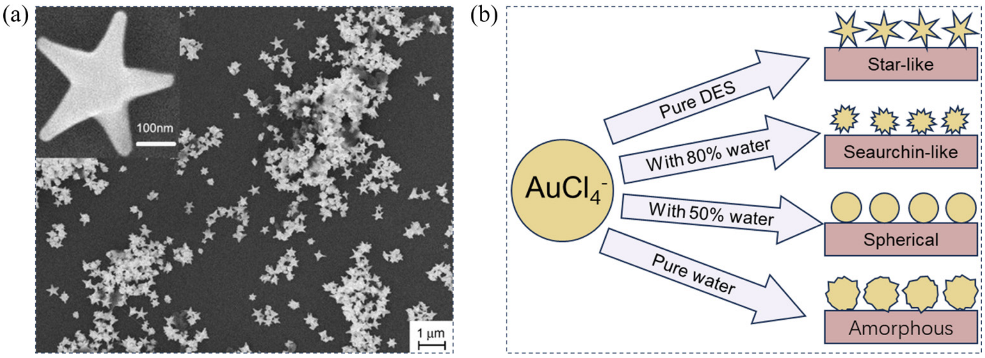

Fig. 4 Morphology control of Au particles: (a) SEM image of star-shaped gold nanoparticles [80]; (b) Schematic diagram of relationship between water content and nanoparticle shape [81]

there have been relatively few reports on the electrodeposition in DESs primarily because the decomposition reaction of common DESs is prior to the electrodeposition of most rare earth elements.

At present, there are few successful cases on the electrodeposition of rare earth elements in urea-based DES at elevated temperatures. In 2011, GÓMEZ et al [56] studied the electrodeposition of cobalt, samarium, and CoSm in ChCl/urea-based DES. As the overpotential increased, the proportion of samarium in the co-deposited product also increased and reached to 70 wt.% ultimately. This study has demonstrated the feasibility of depositing high-concentration samarium alloys in DESs. LIU et al [92] electrodeposited a Co−Nd alloy in a ChCl/urea-based DES and obtained a Co−Nd alloy coating with a neodymium content of 3.74 wt.% on copper cathode. However, they pointed out that it is not possible to achieve the pure neodymium in ChCl/urea-based DES based on cyclic voltammetry data.

PANZERI et al [93] introduced the electrodeposition of Sm−Co alloy in another DES system (ChCl/EG). They discovered that process parameters had a significant impact on the composition of the products. Adding ChCl or glycine to the mixture increased the samarium content in the co-deposited product, which results in the formation of ferromagnetic Sm−Co films with samarium content ranging from 0 to 50 wt.%. KOPCZYŃSKI and LOTA [94] reported the electrodeposition of Ce−Ni alloy coatings in ChCl/EG. In addition to the chlorides of cerium and nickel, ammonium chloride and boric acid were also added, resulting in coatings with a cerium content of up to 85 wt.%. The experiments proved that these coatings exhibited excellent electrochemical performance in the hydrogen evolution reaction.

2.5 Alkali metals

DESs have widely used in alkali metal recovery [95,96] and energy-related applications [97,98]. However, there have been relatively few studies in the realm of electrodeposition because the components in the DES tend to be decomposed before the alkali metals reduction takes place [99,100].

However, LI et al [101] reported the addition of LiTFSI-urea DES as an additive to commonly used carbonate-based electrolytes in lithium metal batteries, and they discovered excellent cycling stability. Further investigations revealed that the solid electrolyte interface (SEI) layer was modulated by the DES additive in terms of its composition. Additionally, the energy barrier for lithium desolvation was reduced. These findings provide inspiration for future research.

3 Key influential factors of metal electrodeposition in DESs

3.1 Water content

Water is everywhere on the earth and it is difficult to completely removal from the DESs. Therefore, in many references, the water content in DESs has been studied and found that it significantly affected the electrochemical behavior of active ions as well as the performance of electrodeposited products [59,80,102]. Especially for hygroscopic DESs, conducting a quantitative study on the influence of water on electrodeposition is highly important.

In some applications, the hygroscopic nature of DESs is considered negative. However, in the study of chromium metal deposition, it has been found that the electrodeposition in a DES with water 20 wt.% led to a bright chromium plating [103]. In general, water has a notable impact on the physical properties of DESs, such as adjusting viscosity [103], promoting the dissociation and conductivity of DESs [43]. In a nickel electrodeposition experiment, the addition of water led to an outstanding improvement in current efficiency and densification of the deposited product.

MERNISSI CHERIGUI et al [104] found a new influence mechanism of water on deposited products. During the nickel electrodeposition process, water in DES formed a matrix of $Ni/Ni(OH)_2$ on the surface of the deposited material, where hydroxides could form hydrogen bonding with DES molecules. This quenched the growth of nickel. This phenomenon explains why the absorption of water results in nickel electrodeposition products that exhibit a uniform and compact appearance with nanoscale particles. LI et al [105] used a similar phenomenon by adjusting the water content in DESs to regulate the formation of $Zn(OH)_2$. They succeed in controlling the nickel content in Zn−Ni alloys. PHUONG et al [106] reported the copper deposition in ChCl/EG DES and also returned similar results, including the increased mass diffusion rates and improved conductivity.

In summary, the influence of water content on metal electrodeposition in DESs mainly involves three aspects: reducing the viscosity of DESs, promoting the dissociation of DESs by changing hydrogen bonding, and affecting the water decomposition products, such as hydroxides.

3.2 Temperature

From early stage, the variable temperature studies of electrodeposition in DESs have been performed, which confirms the significant effect of temperature on the properties of DESs [106−108]. The most intuitive phenomenon showed in the tin electrodeposition in ChCl/EG DES [109] is that the increase of temperature will improve the mobility and electrical conductivity of DESs, which is similar to the influence of water content.

Additionally, elevated temperature could facilitate the thermal motion and kinetic energy of electroactive species, which generally results in an extensive influence on electrodeposition. For example, it not only decreases the electrochemical polarization of cathodic process, but also increases current efficiency. Moreover, some experimental findings show that increasing temperature promotes the growth of tin grains, leading to the formation of larger multi-layer pagoda-shaped crystals. Similar examples can be observed in other studies, such as in the research of depositing nickel on copper [110], it was found that with the increase of temperature, grain coarsening occurred, and the average surface roughness also increased with the rising temperature.

Overall, the effect of temperature is similar to that of water content, such as reducing viscosity and increasing electrical conductivity, but there are also some exceptions. For example, the addition of water leads to a decrease in the size of deposited grains, while the increase of temperature has the opposite effect.

3.3 Additives

The additives of the electrodeposition in DESs were also widely studied for various aims [61,101,111]. In most reports, it has been observed that additives lead to dense and bright deposits, which is crucial in practical applications because a dense deposit deliveries high strength and excellent corrosion resistance.

In Fig. 5(a), the mechanisms of different additives effect on Zn metal electrodeposition summarized from various studies are listed. As examples of these studies, ALESARY et al [112] reported the influence of adding nicotinic acid, boric acid, and benzoquinone on the electrodeposition in ChCl/EG DES. The additives significantly decreased the reduction peak current density of Zn^{2+} compared to the blank control. In the groups with nicotinic acid and boric acid, cathodic current efficiencies were notably reduced. The introduction of additives resulted in a refinement of grains on the coating surface. This

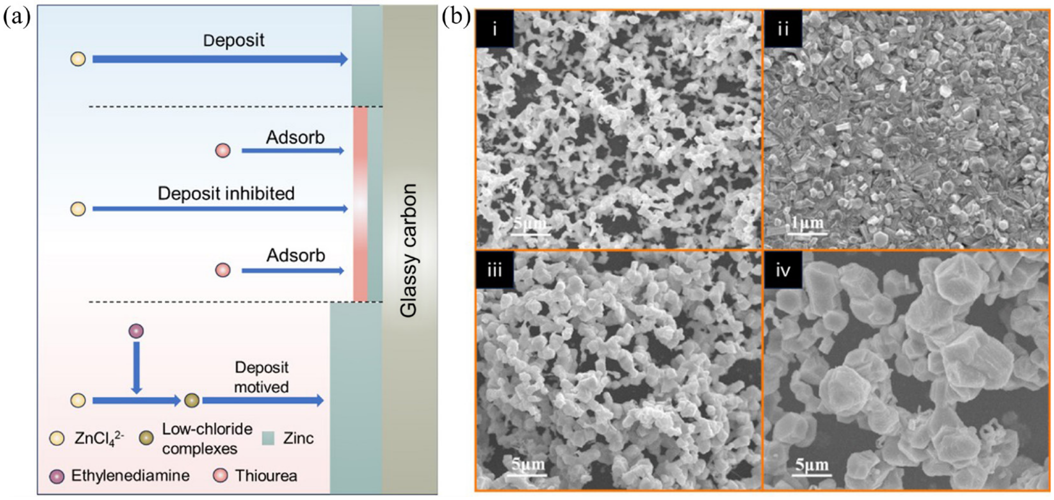

Fig. 5 Influence of additives on morphology of deposits: (a) Effect of different additives on metal electrodeposition; (b) SEM micrographs of zinc deposits with additives: (i) Blank; (ii) Thiourea; (iii) Ammonia; (iv) Ethylenediamine [48]

outcome is considered as the adsorption of additives on the electrode surface, reducing the active sites for zinc deposition, inhibiting zinc growth, and decreasing the formation of large clusters. On the other hand, some additives have different influences and mechanism. A study of the influence of amine additives reported by SONG et al [48] showed that the additions led to an increase in the reduction peak current of zinc, along with an increase in the electrolyte's conductivity. Cyclic voltammetry showed that the addition of ammonia and ethylenediamine accelerated the nucleation rate of zinc, which was also confirmed by chronoamperometry. SONG et al [48] explained that the amine additives altered the active species in the DES, shifting them from more stable $ZnCl_4^{2-}$ to chlorozinc complexes with low coordination number of chloride which are easier to reduce, promoting zinc deposition. As shown in Fig. 5(b), this effect significantly increases the grain size.

In summary, the effects of additives on electrodeposition have two typical ways. Firstly, they may adsorb onto the surface of electrode, inhibiting the deposition of target metals. Secondly, they can change the characteristics of the active species within the DESs, resulting in different outcomes depending on the nature of the used additives.

3.4 Current modes

In the process of electrodeposition in DESs, the common current modes include constant current, constant voltage, pulse voltage, reverse pulse voltage, etc. These current modes, as well as different conditions within the same current mode, have a significant impact on the results of the electrodeposition.

In the early stages of research, the current mode did not receive particular attention. Until 2011, GU and TU [108] studied the influence of current modes, including constant voltage, pulse voltage, and reverse pulse voltage, on the morphology of deposited nickel. They found that, when the current mode was pulse voltage, duty cycle could affect the morphology of the deposits. Specifically, with a lower duty cycle, the products were regularly arranged, and as the duty cycle increased, randomness also increased. As an extreme case of high duty cycle, the constant voltage mode showed a similar random arrangement, which is shown in Fig. 6(a) [108]. The phenomenon can be explained by the fact that, at low duty cycles, atoms were allowed to find the lowest energy sites, while at high duty cycles, atoms did not have enough time for regular arrangement. Different from the two modes mentioned above, under the reverse pulse voltage mode, nickel deposits with a flower-like microstructure were obtained. It can be confirmed that the reverse voltage could remove some of the metal, leading to an overall uniform distribution.

In the study of palladium [113], it was found that pulsed current can make the surface of the deposit finer and denser. However, micro-cracks were produced due to mutually interfere when additives exist. Further parameter adjustments are needed to obtain a palladium film with sufficient adhesion and high surface quality.

As illustrated in many examples, such as constant voltage mode, different parameters can also lead to different deposition results. For example, in a study of Zn−Ni alloy coatings [114], it was mentioned that a high overpotential can refine the grain size because the nucleation rate increases at high overpotentials.

In addition to the fine control of surface morphology, pulse current could achieve high current efficiency and produce fine grain sizes as demonstrated in the copper electrodeposition (Figs. 6(b) and (c)) [115]. Moreover, as the pulse cycle duration shortens, the current efficiency further increases. This is attributed to the replenishment of consumed copper ions during the current-off intervals, which helps overcome the limitations of mass transport efficiency. At the same time, applying current during the on-periods allows higher current density, which is favorable for the instantaneous nucleation of copper. Similar phenomena have been reported in the electrodeposition of other metals, such as zinc [114] and Co−Cr alloy [116].

In conclusion, the effect of current modes on electrodeposition in DESs is significant, mainly because they directly influence the nucleation and growth processes of deposition. By cleverly utilizing different current modes, we can achieve higher current efficiency, faster deposition rates, finer grain sizes, and even choose the desired microstructure.

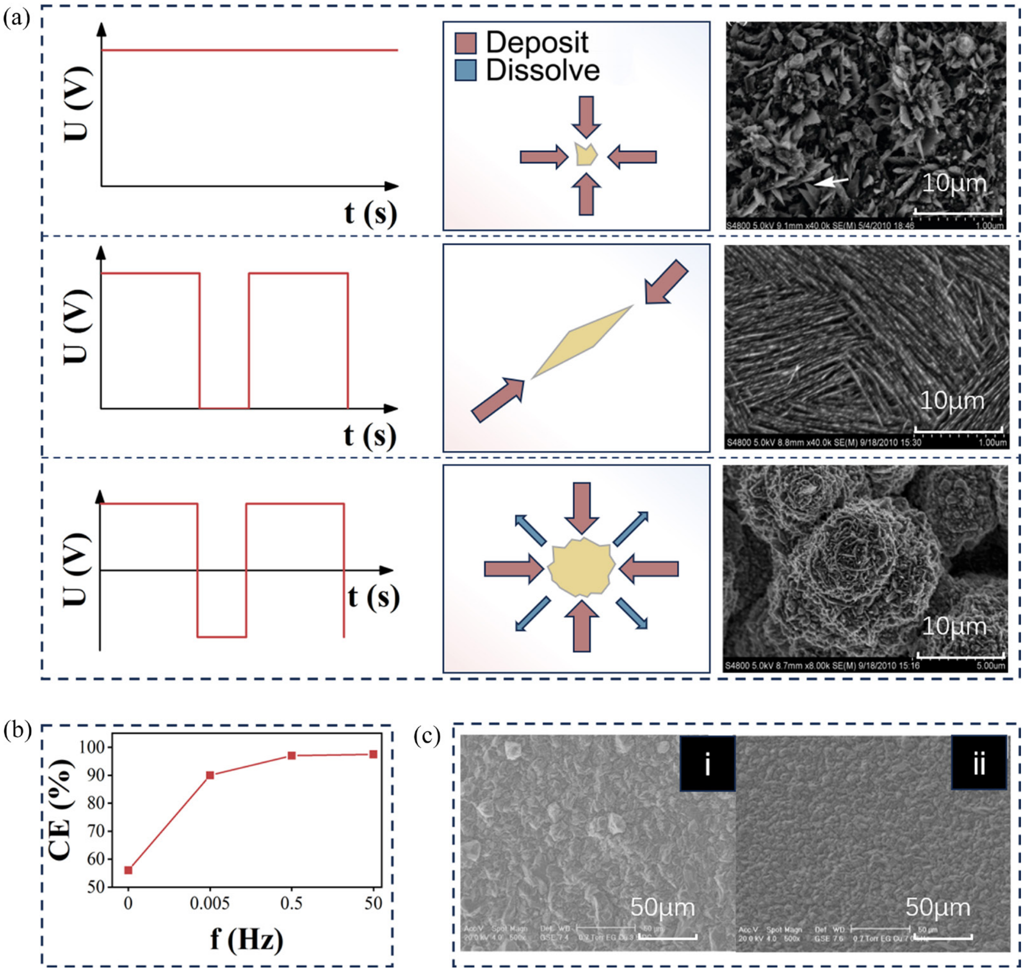

Fig. 6 Influence of current mode on morphology of deposits: (a) Schematic diagram and SEM images of nickel deposition patterns under different current modes [108]; (b) Influence of frequency on current efficiency (CE) of copper electrodeposition at duty cycle of 0.5; (c) Copper grain size with different current modes: (i) direct current and (ii) pulse current [115]

4 Summary and outlook

In this review, we have summarized the research on the electrodeposition of various metal/alloy films and coatings in DESs and discussed the influencing factors in this process. This is a highly valuable topic because metal/alloy electrodeposition in DESs demonstrates its advantages of low pollution and low energy consumption compared with the traditional electrodeposition methods. However, the large-scale industrial application of DESs is still a big challenge. To unlock DESs' full potential, both fundamental research and industrial studies on the metal electrodeposition in DESs should be expanded. This chapter will be structured around Fig. 7.

4.1 Fundamental research on metal electrodeposition in DESs

Although much progress has been made in the research of metal electrodeposition in DESs, many metals and alloys are not suitable for electrodeposition using DESs. For instance, copper can be electroplated in aqueous solutions with relatively high current efficiency, and DESs do not offer a cost advantage compared to aqueous solutions. Nonetheless, DESs still possess unique advantages in certain applications where aqueous solutions are unsuitable. An example is the electroplating on magnesium alloy surfaces. In aqueous systems, magnesium alloys react with the electrolyte, leading to passivation or corrosion. However, this can be avoided in DESs. Similarly, DES electrolytes are better than aqueous systems for metals with more negative deposition potentials and prone to

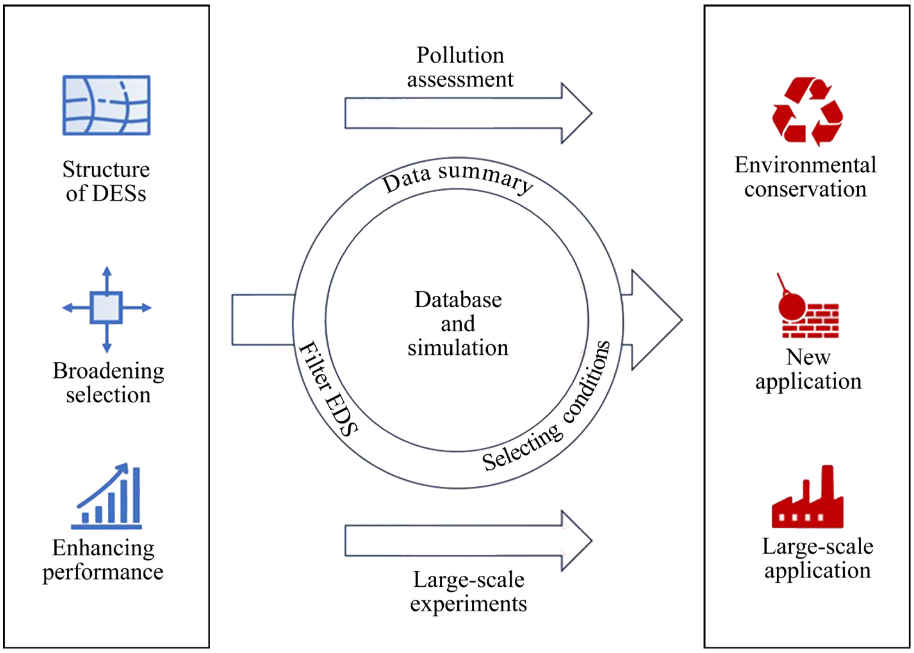

Fig. 7 Challenges and corresponding solutions of metal electrodeposition in DESs

hydrogen evolution reactions during electrodeposition, such as zinc and other metals [117].

To better understand and develop DESs, there are several directions that require effort. Firstly, there is considerable room for improving the understanding of the impact of DESs' structure and dynamics on their physical and electrochemical properties. Secondly, the performances of existing DESs still need to be optimized so as to meet the industrial demands. Thirdly, although the tunability of DESs is apparent, only a few types have been widely studied so far, which limits the application of DESs, such as in the electroplating of rare earth metals and lithium metal batteries. To address these challenges, it is necessary to broaden the research scope on DESs. Lastly, further exploration of metals and alloys' electrodeposition is also needed where DESs offer advantages over traditional aqueous systems in electrodeposition applications.

In response to the issues, we proposed some solutions. Firstly, a deep investigation into the structure and behavior of DESs is very important. One of the research focuses is the hydrogen bond networks. Hydrogen bonds are highly suspected to be crucial intermolecular forces in the formation of DESs, and recent research has suggested that differences in hydrogen bonding may be the cause of performance variations between DESs. The double-layer states, morphologies of active species, and deposition mechanisms in the electrodeposition processes of DESs require thorough investigation. Secondly, understanding the relationship between structure and properties, and potential connections between different properties, can guide the optimization of DESs. For instance, the relationship between conductivity and viscosity highlights the significant impact of viscosity on electrodeposition efficiency. In the efforts to optimize DESs performance, an overall understanding is necessary to identify available means for the entire DESs. Thirdly, after refining our fundamental understanding of DESs, we can establish a database for DESs. This involves organizing information collected from studies on the physical and chemical properties of DESs, synthesis methods, and treatment and preprocessing techniques. With concentrated data from focused research, new relationships and insights can be discovered. Founding more predictive models for DES performance guides researchers in designing DESs based on predicted mixture properties. Data obtained by researchers in experiments can further refine simulation systems. Through strengthening the collaboration between simulation and experiment, the potential of DESs' controllability can be realized. And finally, we can use the new knowledge to select DESs that have more advantages compared to traditional water systems.

4.2 Industrial applications of metal electrodeposition in DESs

Apart from fundamental research, the industrial application of DESs faces several challenges. Firstly, in comparison to conventional electrolytes, DESs have environmental advantages. However, in some studies, the "green" nature of DESs has been questioned [118−120]. Secondly, traditional DESs encounter challenges in many applications, such as high viscosity, absorbing water from the air, a narrow electrochemical window preventing the deposition of alkali metals, and insufficient solubility of oxides limiting the types of extractable metals. Finally, regardless of excellent performance in the laboratory, DESs should be used in large-scale applications.

After discussing these challenges, we need to turn to how to solve these problems. In response to environmental concerns, researchers need to conduct further quantitative assessments of the recyclability, biotoxicity, and environmental impact throughout the lifecycle of DESs. This is necessary to evaluate the validity of the concerns and to identify DESs with minimal environmental harm. To address the challenges encountered in practical applications, methods can be considered, such as raising temperatures to reduce viscosity and developing new DESs with better performance. While the research community conducts more in-depth studies, large-scale experiments on DESs should also be established to find optimal processes for the electroplating, refining, and metal recovery applications of DESs. Research should focus on the performance of DESs under conditions more representative of practical situations, such as high current density and extended operational periods. At the same time, we should also pay attention to the differences between experimental conditions and large-scale production conditions, such as heat transfer, mass transfer, and changes in product performance.

CRediT authorship contribution statement

Man-peng LIN: Writing – Original draft; **Han-dong JIAO:** Conceptualization, Writing – Review & editing; **Rui YUAN**, **Le-yang LI**, **Lin-lin WANG**, **Rui-yang SUN**, and **Dong-hua TIAN:** Writing – Review & editing; **Shu-qiang JIAO:** Conceptualization, Writing – Review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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在深共晶溶剂中电沉积制备金属/合金薄膜或涂层的研究进展

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摘 要：开发能耗低、环境友好的金属/合金薄膜或涂层电沉积制备技术是当前学术界的研究重点之一。为此，深共晶溶剂(DESs)因其操作温度低和电势窗口宽等优势成为电沉积技术实施的重要电解质种类。目前，关于这一新兴领域的文献很多，但尚未出现围绕该主题的综述性论文。在此，本文作者首先简要回顾了 DESs 的概念和历史，随后，系统总结了 DESs 中金属/合金电沉积的最新进展。此外，还讨论了电沉积过程中的关键影响因素，并分析了相应的作用机制。在此基础上，强调了建立预测模型以应对大规模应用所面临挑战的重要性。

关键词：研究进展；电沉积；金属/合金薄膜；深共晶溶剂；电极过程

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