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ELECTROCHEMICAL SYNTHESIS AND FUNCTIONAL PROPERTIES OF METAL AND ALLOY-BASED COMPOSITION COATINGS

This work presents a comprehensive review on the electrochemical synthesis and functional properties of metal and alloy-based composition coatings. Nowadays, electrochemical synthesis is considered a versatile and widely used technique for producing metal and alloy coatings with precisely controlled composition, thickness, and microstructure. These coatings offer a variety of functional properties that can enhance the performance and durability of various substrates. It typically involves immersing a substrate (cathode) in an electrolytic solution containing dissolved metal ions. A current is applied between the cathode and an anode, causing the metal ions to be reduced and deposited onto the cathode surface as a coating. The specific properties of the coating, such as composition, thickness, and morphology, can be controlled by various parameters, including electrolyte composition, current density and potential, temperature and pH as well as substrate material and pretreatment. The utilization of electrochemical methods in coating fabrication has gained significant attention due to their efficiency, precision, and environmental friendliness. As such, the focus of this review is on the synthesis processes, structural characterization, and functional properties of coatings comprising various metals and alloys to help understand electrochemical deposition techniques for tailored coatings with enhanced properties for diverse applications by summarizing previous studies.

Kew words: electrochemical synthesis, composition coatings, electrolyte composition, substrate material, synthesis processes.

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Электрохимический синтез и функциональные свойства композиционных покрытий на основе металлов и сплавов

В данной работе представлен комплексный обзор электрохимического синтеза и функциональных свойств композиционных покрытий на основе металлов и сплавов. В настоящее время электрохимический синтез считается универсальным и широко используемым методом получения покрытий из металлов и сплавов с точно контролируемым составом, толщиной и микроструктурой. Эти покрытия обладают множеством функциональных свойств, которые могут улучшить эксплуатационные характеристики и долговечность различных поверхностей. Обычно это включает погружение подложки (катода) в электролитический раствор, содержащий растворенные ионы металлов. Между катодом и анодом подается ток, в результате чего ионы металла восстанавливаются и осаждаются на поверхности катода в виде покрытия. Конкретные свойства покрытия, такие как состав, толщина и морфология, можно контролировать с помощью различных параметров, включая состав электролита, плотность и потенциал тока, температуру и pH, а также материал подложки и предварительную обработку. Использование электрохимических методов изготовления покрытий привлекло значительное внимание благодаря их эффективности, точности и экологичности. Таким образом, основное внимание в этом обзоре уделяется процессам синтеза, структурным характеристикам и функциональным свойствам покрытий, содержащих различные металлы и сплавы, чтобы помочь понять методы электрохимического осаждения для индивидуальных покрытий с улучшенными свойствами для различных применений путём обобщения предыдущих исследований.

Ключевые слова: электрохимический синтез, композиционные покрытия, состав электролита, материал подложки, процессы синтеза.

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Металдар мен қорытпалар негізінде композиттік жабындардың электрохимиялық синтезі және функционалдық қасиеттері

Бұл жұмыста металл және қорытпалы композициялық жабындардың электрохимиялық синтезі мен функционалдық қасиеттеріне жан-жақты шолу жасалды. Қазіргі уақытта электрохимиялық синтез құрамы, қалыңдығы және микроқұрылымы дәл бақыланатын металдар мен қорытпалардан жабын алудың әмбебап және кеңінен қолданылатын әдісі болып саналады. Бұл жабындар әртүрлі беттердің өнімділігі мен беріктігін жақсартатын көптеген функционалдық қасиеттерге ие. Бұл әдетте субстратты (катодты) еріген металл иондары бар электролиттік ерітіндіге батыруды қамтиды. Катод пен анод арасында ток беріледі және нәтижесінде металл иондары тотықсызданып, катод бетіне жабын түрінде тұнбаға түседі. Композиция, қалыңдық және морфология сияқты жабынның ерекше қасиеттерін электролит құрамы, токтың тығыздығы мен потенциалы, температура мен рН, субстрат материалы және алдын ала өңдеу сияқты әртүрлі параметрлер арқылы басқаруға болады. Жабындарды жасаудың электрохимиялық әдістерін қолдану олардың тиімділігіне, дәлдігіне және тұрақтылығына байланысты айтарлықтай назар аударды. Осылайша, бұл шолуда алдыңғы зерттеулерді жалпылау арқылы әртүрлі қолданбалар үшін жақсартылған қасиеттері бар жеке жабындар үшін электрохимиялық тұндыру әдістерін түсінуге көмектесу үшін әртүрлі металдар мен қорытпалардан тұратын жабындардың синтез процестеріне, құрылымдық сипаттамаларына және функционалдық қасиеттеріне назар аударылады.

Түйін сөздер: электрохимиялық синтез, композициялық жабындар, электролит құрамы, субстрат материалы, синтез процестері.

Introduction

A key component of engineering technology is materials and the surface is where the electrochemical, thermal, and mechanical interactions between the materials start. But material surfaces are always vulnerable to deterioration and corrosion, which causes enormous losses in the industrial sector. Therefore, it is now inevitable to apply surface improvement technology to stop or lessen the loss. Significant advances in metallurgy and materials science have taken place in the previous few decades, leading to the development of novel engineered materials with improved qualities. It is difficult to develop appropriate processing to manufacture desired materials; this involves changing the materials' intrinsic qualities. The broader economic perspective and its effects on the environment are taken into account.

Innovation is fueled by the search for materials with precisely regulated qualities in a variety of industries. An effective tool for creating metal and alloy coatings with specific functional properties in this endeavor is

electrochemical synthesis. Electrochemical synthesis is a technique that uses electric current to deposit metal or alloy layers on a substrate. It is based on the principle that a layer of coating is formed as a result of the electrode - electrolyte electrochemical reactions occurring leading to electrodeposition of ions contained in electrolyte. The composition of the coating can be controlled by varying the electrolyte composition, the applied voltage, the current density, the deposition time, and other parameters. Electrochemical synthesis can produce coatings with different functional properties, such as corrosion resistance, wear resistance, catalytic activity, optical reflectivity, and electrical conductivity. One electrochemical method for altering the surface structure is electrodeposition. According to certain theories, the use of electrodeposition in surface engineering dates back almost 200 years. Its development of electrodeposition can be traced back to the early 19th century, with significant contributions from various scientists and researchers [1]. The invention of the galvanic cell in the early 1800s made it possible to employ electric current as a more economical method of producing coatings. The first large-scale industrial plant for electrodeposition was built in the 19th

century for the deposition of gold, silver and copper for decorative applications. Since then, electrodeposition has rapidly grown and it has been applied for the deposition of metals, alloys and composites with complex shapes (i.e. coatings, micropillars, nanowires) and to produce dense nanostructures and amorphous coatings with increased mechanical, corrosion, electrical and magnetic properties. Thanks to this progress, today, electrodeposition is a widely employed technique for the production of coatings ranging from decorative to technological applications [2]. There are various reasons why the electrodeposition process is superior to other coating procedures [3, 4]:

- Its initial investment is low combined with high production rate.
- It can be used with a wide variety of shapes and sizes of substrates.
- Ease of producing economically viable quantities of nanocomposite materials, with grain sizes as small as 10 nm.
- Products of electrodeposition require no further processing and can be used right away.
- Easy reproducibility in labs and industry with little difficulty owing to technological limitations.
- Electrodeposited coatings have exhibited better physical properties.

A wide range of coatings can be produced by incorporating particles into an electrodeposit and many materials can be combined in composite coatings applied by electrodeposition. Electrodeposition of composite coatings offer the benefits of good control over deposition rate (hence thickness), coating composition and deposit properties [5]. Electrochemical synthesis methods include direct current electrodeposition, pulse current electrodeposition and jet electrodeposition.

Coatings made of metal and alloys have several benefits, in fields such as energy, electronics, biomedicine, and engineering, such as better wear and abrasion resistance, greater corrosion resistance, and special electrical and catalytic qualities. But attaining the intended capabilities frequently calls for close attention to composition, microstructure, and morphology[6]. A new platform for attaining this kind of control is offered by electrochemical synthesis, which makes it possible to precisely deposit metals, alloys, and even composites with the appropriate properties.

The present work focuses on understanding the intricate relationship between electrochemical synthesis parameters, coating composition, and resulting functional properties since electrochemical synthesis and functional properties of metal and alloy-based composition coatings are a significant area of research due to their potential applications in

various fields, including energy storage, corrosion protection, and surface engineering. Through a combination of experimental investigation and theoretical analysis, it aims to shed light on the underlying mechanisms governing the formation and properties of these coatings. In summary, recent advancements in the electrochemical synthesis of metal and alloy-based composition coatings have demonstrated the potential for creating materials with improved corrosion resistance and functional properties, which are crucial for various industrial applications. This review explores the synthesis and functional characteristics of composition coatings made by electrochemical techniques, providing an in-depth look at this fascinating field.

Electrodeposition

Electrodeposition is a widely employed versatile and ecofriendly technique for coating fabrication. The electrochemical analysis deals with chemical reactions of samples or solutions with the applied electrical potential. The rate of redox reactions is analyzed and controlled by a potentiostat, connected to electrodes submerged in an electrolyte [7]. It is an electrochemical process applied for surface structure modification and is considered as an easy and cost-effective technique for preparation of alloy coating. Electrodeposition processes are usually performed with aqueous-based electrolytes, at ambient pressure, and low operating temperatures (i.e. lower than 100 °C) owing to an increased understanding of the electrodeposition fundamentals, with a proper control of the deposition parameters, it is possible to fine-tune the material characteristics (e.g. composition and microstructure) and consequently the material properties. The deposition process involves the reduction of metal ions onto a conductive substrate, leading to the formation of a coherent coating. This deposition technique is mostly used to deposit thin films of material to the surface of an object to change its external properties such as to increase corrosion protection, increase abrasion resistance, improve decorative quality, or simply to deposit a layer which is part of a more complicated device. In this technique three electrodes are used which are working, reference, and counter (sometimes secondary) electrodes, respectively. The electrodes are connected to a potentiostat which is the instrument which controls the deposition process. These electrodes were kept within a container containing a liquid which has ionic species dissolved within it, such as copper ions dissolved in water [8]. Schematic of electrodeposition is shown in Figure 1.

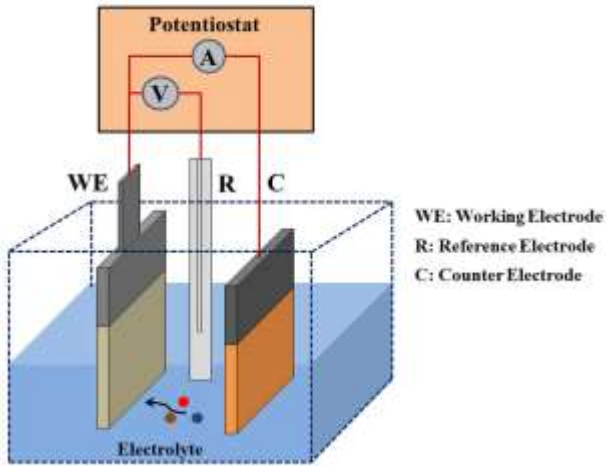


Figure 1 – Schematic of electrodeposition method. Adapted from [8]

Pulse Current Electrodeposition

Over the years, pulse electrodeposition (PED) has been used in metal-alloy composition coatings with a particular interest for the synthesis of anticorrosive coatings with controlled morphology and composition [10]. In PED, The properties of the coatings are affected by three fundamental parameters: peak current (I_{peak}), pulse imposition time (ON-time, T_{ON}) and switch off time (OFF-time, T_{OFF}). Mathematically, these parameters can be related to evaluate pulse frequency (Eq.1), duty cycle (Eq.2) and average current density (Eq.3) [11]

$$f = \frac{1}{T_{OFF} + T_{ON}}, \quad (1)$$

$$\gamma = \frac{T_{ON}}{T_{OFF} + T_{ON}} \times 100, \quad (2)$$

$$I_{ave} = \frac{T_{ON}}{T_{OFF} + T_{ON}} \times I_{peak} = I_{peak}, \quad (3)$$

where γ is the duty cycle, f is the frequency, I_{peak} is the peak current density, and I_{ave} is the average current density.

In pulse electrodeposition, current density has a significant effect on structure, mechanical, tribological, and corrosion properties of composite coatings [12] and it showed several advantages over DC electrodeposition, such as improved wear resistance and hardness, particle distribution, structure, morphological structure and the ability to control the grain sizes of the deposits [13].

Jet Electrodeposition

In this type of electrodeposition, a jet of plating solution is directed at the cathode surface directly. There exists an electrical field between the anode (located in the nozzle) and cathode (substrate). The following Figure 2 demonstrate a setup of schematic representation of jet electrodeposition technique.

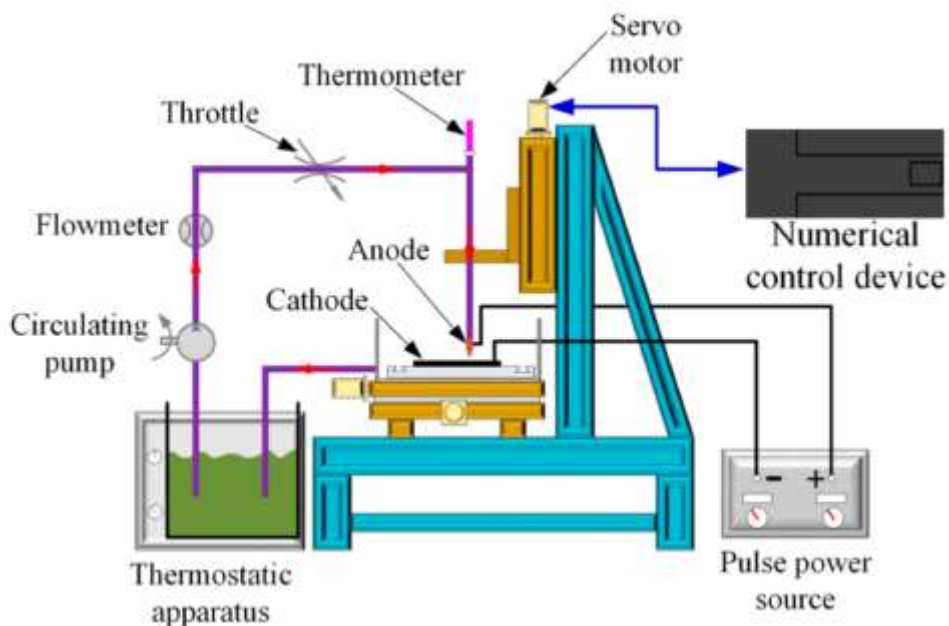


Figure 2 – Schematic of electrodeposition method. Adapted from [14]

As the plating solution flows, electric current is transferred along the stream of fluid to the substrate surface. This enables deposition to occur on the cathode surface where the jet flows over [15]. Jet electrodeposition is a high-speed electroplating technique that provides a wide range of advantages over other coating techniques including: high deposition rate and more efficient grain size refining effect [16].

Electrodeposition parameters for metal and alloy-based composition coatings

The effects of deposition parameters such as current density, bath composition, electrolyte, pH, and temperature on the coating properties can be thoroughly investigated. By adjusting the electrolyte composition and polarization mode (static or pulse, reverse current or potential decrease), electrochemical synthesis technique offers the flexibility to alter component concentration, rate of deposition, and surface condition. In [17], composite coatings of Fe-W-WO₂ and Fe-Mo-MoO₂ were deposited on cast iron substrate by different electrolysis modes from citrate Fe(III)-based bath. The hardening phase of refractory metal oxides is formed directly in the cathode process and is included in the alloy matrix, which helps to increase the uniformity of the distribution of components over the thickness of the coating and its surface. The use of pulse current electrolysis allows obtaining more uniform coatings enriched with alloying components with a smaller amount of adsorbed nonmetallic impurities. Composite Fe-Mo-MoO₂ has a higher corrosion resistance due to the chemical stability of molybdenum and its oxides in environments of various aggressiveness including chloride-containing solutions. The physic-mechanical properties such as microhardness, friction coefficient, and wear resistance of Fe-W-WO₂ composites surpass not only the base material but also the molybdenum-containing coatings. Combination of higher strength characteristics and increased corrosion resistance of composite coatings Fe-W-WO₂ and Fe-Mo-MoO₂ in comparison with cast iron allowed to consider them as promising materials in the technology of surface hardening and restoration of worn surfaces of parts.

As indicated in [18], by adjusting the concentration ratios of iron(III) sulfate and sodium molybdate (tungstate), uniformly brilliant and lustrous deposits of binary Fe-Mo and ternary Fe-Mo-W alloys can be generated from the citrate electrolyte (pH 3–4) in both dc and pulsed mode with current efficiency of 65.0–85.0%. Ternary galvanic alloys of cobalt with molybdenum and zirconium of different composition and morphology are obtained from

polyligand citrate-pyrophosphate electrolyte in a pulsed mode by varying the current density and duration of pulse and pause. It is shown that coatings with micro-globular morphology with low level of stress and cracks are formed at a current density of 4–6 A/dm² and polarization on/off time 2/10 ms [19]. When ternary Fe-Co-W alloys with micro globular surface of different composition were deposited by direct and pulse current from citrate Fe (III) electrolyte, current density and time parameters of pulse electrolysis are shown to be effective tools to control the refractory metals content and electrolysis efficiency, providing increased tungsten content [20]. Electrodeposition of binary and ternary coatings Co-Mo, Co-W and Co-Mo-W from bi-ligand citrate pyrophosphate electrolyte has been investigated in [21] with determination of the effect of both current density amplitude and pulse on/off time on quality, composition and surface morphology of the galvanic alloys resulting in improved physical and mechanical properties of deposits as well as high corrosion resistance.

Functional Properties

Functional properties of metal and alloy-based composition coatings such as corrosion resistance, mechanical properties (coating hardness, adhesion, and wear resistance) as well as electrical and thermal conductivity depend on various factors, including composition, microstructure, and surface morphology. Studies have focused on tailoring these properties to meet specific application requirements.

According to [22], the advantage of coatings with alloys in comparison with individual metals, as well as the realization of synergism during the electrolytic alloy formation, manifest themselves in a change in the microhardness of materials depending on their composition and structure. In a study conducted to analyze influence of the compositions of Fe-Co-Mo, Fe-Co-W, and Co-Mo-W coatings on the corrosion resistance in media with different acidities, it is demonstrated that molybdenum (and tungsten) significantly boosts the coatings' chemical resistance when compared to the substrate material. Simultaneously, an increase in solution pH and the addition of refractory components to the alloy reduce the rate of corrosion [23]. Moreover, in the process of deposition of composite electrolytic coatings, two kinds of adsorption were discovered playing an essential role in the formation of these coatings, namely, the adsorption of dispersed particles on the cathode and the adsorption on the surfaces of these particles in the process of their introduction in the electrolyte. It is also indicated in [24, 25] that when ternary Fe-Co-Mo(W) alloys with micro-globular

surface of different composition were deposited by direct and pulse current from citrate Fe(III) based electrolyte, the current density and time parameters of pulse electrolysis have been shown to be effective tools to control the refractory metals content. Here, the micro-globular surface of the Fe-Co-Mo(W) alloys is caused by refractory metals incorporation. The amorphous-crystalline structure of deposits with coherent-scattering region sizes of the amorphous part of 2–8 nm has been found and the microhardness of the ternary Fe-Co-Mo(W) alloys is about 4–5 times that of the mild steel substrate and has been shown to increase with tungsten incorporation. Moreover, the magnetic characteristics of the amorphous Fe-Co-Mo(W) coatings were measured in dependence of thickness and deposition time supposedly proving that the content of magnetic phase in upper layers of coating is greater than in the bottom ones. In a study where nano-composite electrolytic coatings (nano-CEC) has been applied on steel, corrosion resistance of the substrate was found to be increased substantially [26].

For the creation of mixed oxide systems with varying dopant contents, the composition of citrate-pyrophosphate electrolytes with the inclusion of iron triad metal sulfates was put fort [27, 28]. The formation and properties of oxide films on titanium using plasma electrolytic oxidation with the addition of iron triad metals such as iron, cobalt, and nickel is discussed and explored that composition of citrate-pyrophosphate electrolytes with dopants to create mixed oxide systems focusing on voltage formation of the oxide systems, the corrosion resistance of the coatings, surface development, and catalytic activity of the mixed oxide coatings. The obtained mixed oxide coatings can potentially be used in catalytic purification systems.

Important consumer properties like corrosion resistance, wear resistance, hardness, and other functional properties of coatings of iron (cobalt)-tungsten alloys in environments of different acidity depends on the content of the refractory component, and the increase in corrosion resistance in an acidic

environment is due to the formation of acidic tungsten oxide on the surface [29, 30]. According to the deep corrosion index coatings with Co(Fe)-W alloys with a tungsten content of 20–40 mas. % belong to the group of very resistant, which allows them to be used as protective coatings in corrosive environments, especially weakly acidic. Here, the microhardness of tungsten electrolytic alloys with iron (cobalt) depends on its content and exceeds the characteristics of coatings with individual metals, which allows us to recommend such materials as an alternative to hard chromium coatings. One of the reasons for the increased corrosion resistance and microhardness of coatings with alloys was found to be the formation of a fine-crystalline, and sometimes, X-ray amorphous structure of the surface layers when using non-stationary modes of electrodeposition.

Conclusion

This review has considered electrochemical synthesis and functional properties of metal and alloy-based composition coatings. The concept of electrodeposition as a versatile, cost-effective and widely used environmentally friendly technique for producing metal and alloy coatings is discussed. It is reviewed how this technique can be used to enhance desired properties of substrates such as corrosion resistance, surface hardness and morphology as well as other technical properties. Types of electrodeposition techniques along with their comparative advantages and drawbacks is discussed. Besides, parameters for metal and alloy-based composition coatings such as bath concentration, pH, temperature and electrolyte are reviewed. Finally, the kind of functional properties most widely considered to perform electrochemical synthesis techniques on various substrates are reviewed. Researches indicated that electrodeposition techniques have a lot to offer when it comes to enhancing desired physical and chemical properties of various substrates under controlled parameters.

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