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Review

Solvent-Driven Electroless Nickel Coatings on Polymers: Interface Engineering, Microstructure, and Applications

Chenyao Wang [1], Heng Zhai [2,*], David Lewis [2], Hugh Gong [1], Xuqing Liu [1,*] and Anura Fernando [1,*]

[1] Department of Materials, The University of Manchester, Manchester M13 9PL, UK; chenyao.wang-3@postgrad.manchester.ac.uk (C.W.); hugh.gong@manchester.ac.uk (H.G.)
[2] Department of Chemical Engineering, The University of Manchester, Manchester M13 9PL, UK; david.lewis-4@manchester.ac.uk
* Correspondence: heng.zhai@manchester.ac.uk (H.Z.); xqliu@nwpu.edu.cn (X.L.); anura.fernando-2@manchester.ac.uk (A.F.)

Abstract

Electroless nickel deposition (ELD) is an autocatalytic technique extensively used to impart conductive, protective, and mechanical functionalities to inherently non-conductive synthetic substrates. This review systematically explores the fundamental mechanisms of electroless nickel deposition, emphasising recent advancements in surface activation methods, solvent systems, and microstructural control. Critical analysis reveals that bio-inspired activation methods, such as polydopamine (PDA) and tannic acid (TA), significantly enhance coating adhesion and durability compared to traditional chemical etching and plasma treatments. Additionally, solvent engineering, particularly using polar aprotic solvents like dimethyl sulfoxide (DMSO) and ethanol-based systems, emerges as a key strategy for achieving uniform, dense, and flexible coatings, overcoming limitations associated with traditional aqueous baths. The review also highlights that microstructural tailoring, specifically the development of amorphous-nanocrystalline hybrid nickel coatings, effectively balances mechanical robustness (hardness exceeding 800 HV), flexibility, and corrosion resistance, making these coatings particularly suitable for wearable electronic textiles and smart materials. Furthermore, commercial examples demonstrate the real-world applicability and market readiness of nickel-coated synthetic fibres. Despite significant progress, persistent challenges remain, including reliable long-term adhesion, internal stress management, and environmental sustainability. Future research should prioritise environmentally benign plating baths, standardised surface activation protocols, and scalable deposition processes to fully realise the industrial potential of electroless nickel coatings.

Keywords: electroless nickel deposition; synthetic yarns; surface activation; coating adhesion; microstructure; solvent systems; wearable electronic textile

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1. Introduction

Electroless deposition (ELD), an autocatalytic process, facilitates the reduction in metal ions onto a substrate without the application of an external electrical current [1,2]. This method offers distinct advantages over traditional electrolytic deposition, including uniformity of the deposited layer, especially on complex geometries, the ability to deposit on non-conductive materials and precise control over the thickness of the deposited layer [3]. Its application spans a wide range of industries, including electronics, aerospace, and corrosion-resistant coatings, where these characteristics are particularly valued [4].

The development of electroless deposition can be traced back to the mid-20th century, with the seminal work of Brenner and Riddell in 1946, who were credited with discovering the first reliable electroless nickel-phosphorus plating process [5]. Prior to their work, early experiments struggled with bath instability, which led to unpredictable results and inconsistent coatings. The breakthrough achieved by Brenner and Riddell allowed for creating a stable bath capable of depositing high-quality nickel-phosphorous (Ni-P) coatings, which revolutionised the surface finishing industry [6]. Over time, the evolution of this technology has been categorised into five key stages: the discovery phase (1916–1940s), early development (mid-1940s to 1959), a period of slow growth (1960–1979), rapid growth (1980–1999), and finally, an era of fundamental research and nanoelectroless plating (1999–continued) [7].

Electroless deposition offers numerous advantages, including uniform coating thickness, the ability to coat non-conductive materials, and enhanced mechanical properties such as hardness, wear resistance, and corrosion resistance. These qualities make electroless nickel coatings ideal for applications in industries such as automotive, aerospace, and electronics. Furthermore, the process is highly adaptable, allowing for the deposition of coatings on substrates with complex geometries without requiring external electrical power [8]. Despite its many benefits, electroless deposition is not without challenges. Maintaining bath stability is critical to achieving high-quality coatings. Variations in bath composition, temperature, and pH can lead to inconsistent deposition, reduced coating quality, or bath decomposition [9]. In addition, while heat treatment can improve the hardness of the coating, it may also reduce corrosion resistance by promoting the formation of crystalline phases, particularly in Ni-B coatings [8]. Careful control of deposition parameters and heat treatment conditions is essential to balance these trade-offs and optimise the coating's performance.

Several mechanisms have been proposed to explain the electroless deposition process. Among the earliest and most influential was the "atomic hydrogen mechanism" [10] put forth by Brenner and Riddell [5]. This theory suggested that atomic hydrogen, generated during the reduction process, was responsible for reducing the metal ions onto the substrate. However, over the decades, other mechanisms, such as the hydride transfer and electrochemical mechanisms, have emerged to account for observations that the atomic hydrogen mechanism could not explain. For instance, the hydride transfer mechanism posits that the reduction occurs via the transfer of hydride ions. In contrast, the electrochemical mechanism suggests that metal deposition is driven by local electrochemical reactions on the substrate's surface [7,11]. Figure 1 demonstrates an example of a Ni-P ELD mechanism. Despite these advancements, no single theory has been universally accepted, as the complexity of the reactions involved often necessitates a multi-mechanistic explanation. In addition to the mechanism, Figure 1b,c illustrate standard electroless deposition process flows, including sequential steps such as surface cleaning, sensitisation, activation, and metallization. Figure 1d further presents an advanced approach using laser-assisted surface modification followed by selective electroless plating, showcasing the versatility of activation techniques used in modern ELD processes [12–14].

Several earlier studies have reviewed electroless nickel plating on textile fabrics and yarns, focusing primarily on surface treatment techniques and the resulting functional properties [15,16]. While these works offered foundational insights, they largely centred on polyester substrates and did not address recent advances in solvent engineering, metal–polymer interface control, and structure–property relationships critical to emerging applications. This review aims to bridge that gap by discussing recent developments in nickel coating mechanisms, alternative polymer substrates, and the influence of solvent systems on the microstructure and functional performance of electroless coatings. In doing

so, it offers a broader perspective on the chemistry-processing-property linkage relevant for both textiles and advanced functional polymer materials.

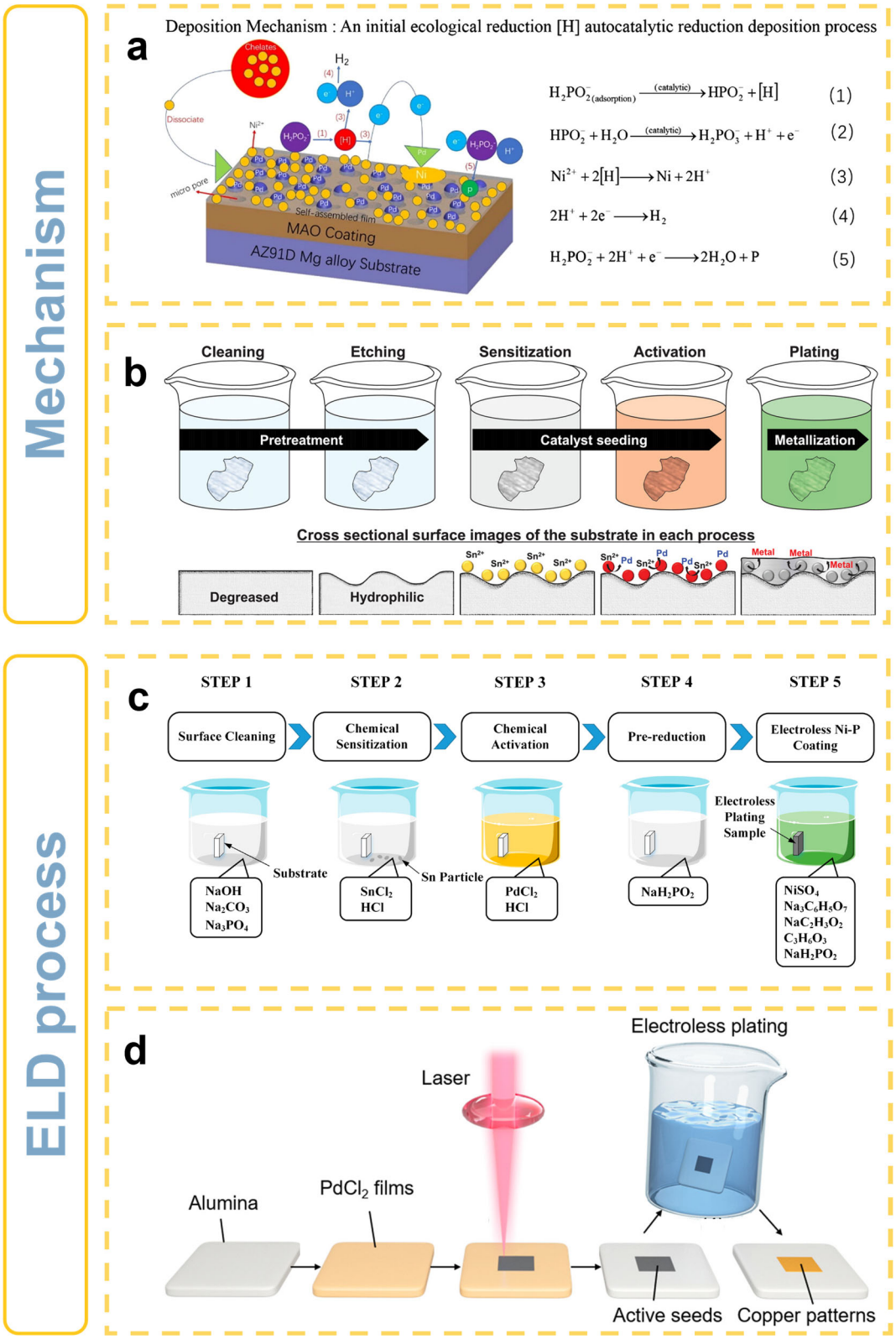


Figure 1. Representative demonstrations of electroless deposition mechanisms and processing strategies. (**a**) Mechanism of Ni-P ELD [17]. Copyright © 2019 Elsevier. (**b**) Schematic of typical ELD process [18]. Copyright © 2025 Wiley-Advance. (**c**) Process of Ni-P ELD [19]. Copyright © 2023 MDPI. (**d**) Process of using laser surface modification for ELD [20]. Copyright © 2022 Elsevier.

Electroless nickel plating has found widespread industrial use due to its superior properties, such as excellent corrosion resistance, enhanced hardness, and uniform coating distribution [21]. Figure 2 shows general types of electroless nickel coatings. In particular, the development of alloy coatings, including ternary and multicomponent systems like Ni-P-W and Ni-P-B [22,23], has significantly improved wear resistance, making these coatings highly suitable for demanding environments [11]. Further advancements include electroless ternary and quaternary alloys, such as Ni-Mo-P, Ni-Mo-P-B, and Co-W-P/Ni-B, which have demonstrated improved diffusion barrier performance, thermal stability, and tailored electronic properties, especially in microelectronic and barrier-layer applications [24,25]. While these alloy systems are less frequently applied to textile substrates, they underscore the versatility and tunability of electroless deposition chemistry. In recent years, the focus has also shifted toward developing nanoalloys created through nanoelectroless deposition processes [26]. These nanoalloys exhibit exceptional mechanical and chemical properties superior to conventional electroless coatings, positioning them as a critical area of future research [27,28].

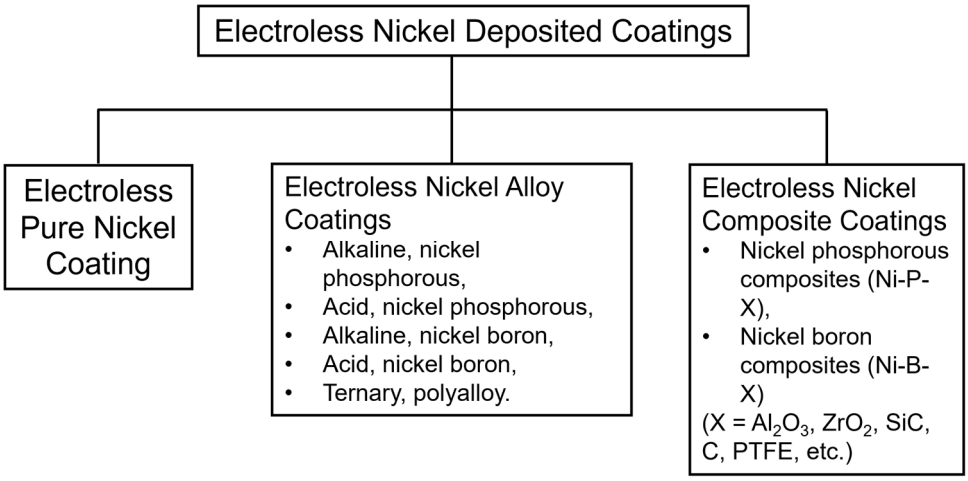


Figure 2. General classification of electroless nickel coatings, summarising major types reported in the literature.

The role of alloying elements in electroless nickel coatings extends beyond deposition kinetics to critically influence the resulting microstructure and functional properties. For instance, phosphorus promotes amorphous structure formation and enhances corrosion resistance but reduces electrical conductivity due to increased electron scattering. Boron tends to yield more crystalline Ni-B coatings, which are harder and more conductive, making them attractive for electromagnetic shielding. Tungsten and molybdenum improve high-temperature oxidation resistance and structural stability, particularly in ternary systems such as Ni-W-P or Ni-Mo-P. The addition of rhenium has been shown to further enhance creep resistance and stability in high-temperature environments. These compositional modifications allow tailoring of coatings to meet specific performance criteria, highlighting the importance of elemental selection in electroless nickel systems [24].

One of the primary challenges in electroless deposition has been ensuring adequate adhesion between the deposited metal and non-metallic substrates, such as glass [29]. For example, in the case of electroless copper deposition on glass substrates for microsystems applications, adhesion has traditionally been poor [30,31]. To address this, various surface modification techniques, such as surface roughening via abrasive machining or high-temperature electrochemical discharge machining, have been developed [32]. These methods increase the surface energy of the glass, thus improving the adhesion of the copper

layer. Additionally, post-deposition annealing has been shown to release residual stresses in the coating, further enhancing adhesion strength [33].

Looking toward the future, electroless deposition continues to expand its industrial applications, particularly in electronics and microsystems, where miniaturisation and precision are paramount. The ability to deposit nanoalloys, which offer superior corrosion resistance and mechanical properties, is likely to see further development. Moreover, the drive to improve bath stability, enhance deposition rates, and develop more environmentally friendly processes will remain key research directions in the ongoing advancement of this versatile technology. This review aims to highlight critical challenges in the electroless nickel coating of synthetic yarns, compare activation strategies, and suggest emerging research directions in wearable electronics.

2. Mechanism of Electroless Nickel Deposition

Electroless nickel deposition is a widely used technique for producing uniform metal coatings, particularly nickel-based alloys, on various substrates. This autocatalytic process allows for continuous deposition once the reaction is initiated. The most deposited nickel alloys are nickel-phosphorus (Ni-P) and nickel-boron (Ni-B), with each system providing distinct properties depending on the composition of the bath and the deposition conditions. Table 1 provides a comparison of mechanisms of electroless nickel deposition.

The fundamental mechanism of electroless nickel deposition involves the reduction of nickel ions (Ni^{2+}) to metallic nickel (Ni^0) through the oxidation of a reducing agent. In the case of Ni-P coatings, sodium hypophosphite (NaH_2PO_2) is the typical reducing agent, whereas nickel-boron coatings utilise sodium borohydride ($NaBH_4$) or dimethylamine borane (DMAB) as the reducing agents. The general redox reactions governing the deposition process can be described by the following half-reactions [9]:

For **Ni-P** coating:

$Ni^{2+} + 2e^- \rightarrow Ni^0$ (Nickel Reduction Reaction)

$H_2PO_2^- + H_2O \rightarrow HPO_3^{2-} + 2H^+ + 2e^-$ (Hypophosphite Oxidation Reaction)

For **Ni-B** coating:

$Ni^{2+} + 2e^- \rightarrow Ni^0$ (Nickel Reduction Reaction)

$(CH_3)_2NH \cdot BH_3 + 3OH^- \rightarrow (CH_3)_2NH + B(OH)_3 + 3H_2 + 3e^-$ (DMAB Oxidation Reaction)

$NaBH_4 + 2H_2O \rightarrow NaBO_2 + 4H_2\uparrow$ ($NaBH_4$ Oxidation Reaction)

In these reactions, the nickel ions are reduced to metallic nickel, which deposits onto the substrate. The oxidation of the reducing agent provides the electrons required for this reduction, and the process generates hydrogen gas as a byproduct [34,35]. This autocatalytic nature of the reaction allows the deposited nickel to act as a catalyst for further deposition, enabling the process to continue without external electrical energy input [36].

Table 1. Comparison of proposed mechanisms for electroless nickel deposition.

Mechanism	Key Concept	Advantages	Limitations	Reaction Pathway
Atomic Hydrogen Mechanism [13]	Reduction via atomic hydrogen generated by the reducing agent.	Simple explanation of reaction initiation.	Does not fully explain all observed reaction phenomena.	$H_2PO_2^- + H_2O \rightarrow HPO_3^{2-} + 2H^+ + 2e^-$ $Ni^{2+} + 2e^- \rightarrow Ni^0$
Hydride Transfer Mechanism [37]	Direct transfer of hydride ions from reducing agents to metal ions.	Better explanation of phosphorus incorporation.	Lacks experimental confirmation for some systems.	$(CH_3)_2NH{\cdot}BH_3 + 3OH^- \rightarrow (CH_3)_2NH + B(OH)_3 + 3H2 + 3e^-$ $Ni^{2+} + 2e^- \rightarrow Ni^0$

Table 1. *Cont.*

Mechanism	Key Concept	Advantages	Limitations	Reaction Pathway
Electrochemical Mechanism [13]	Formation of local galvanic cells driving nickel deposition.	Explains surface reactions in more detail.	Complexity in modelling local electrochemical interactions.	$Ni^{2+} + 2e^- \rightarrow Ni^0$ $H_2PO_2^- \rightarrow HPO_3^{2-} + H^+ + e^-$

The bath composition is critical to the successful deposition and quality of the final coating [38,39]. A typical electroless bath contains several key components, including nickel salts such as nickel chloride ($NiCl_2$) or nickel sulphate ($NiSO_4$), a reducing agent (sodium hypophosphite for Ni-P and sodium borohydride for Ni-B), complexing agents, and stabilisers. The complexing agents, such as ethylenediamine, play an important role in controlling the availability of nickel ions, ensuring a steady and uniform deposition. Stabilisers and surfactants are often added to prevent unwanted side reactions and maintain the bath's stability, thus ensuring consistent deposition over time [8,38].

Nucleation

The nucleation and growth behaviour in ELD is critically influenced by the activation of the substrate surface [40]. During the initial stages, fine nuclei form uniformly across the activated surface, initiating a columnar growth structure [41]. As deposition progresses, a diffusion layer develops around each growing crystallite, where the concentration of metal ions is locally reduced. Overlapping diffusion layers between neighbouring nuclei restrict lateral ion transport, promoting continued vertical crystallite growth. This transition leads to the evolution of a nodular or cauliflower-like morphology, characteristic of electroless coatings [42]. Such microstructures enable uniform coating formation even on complex or non-conductive substrates, enhancing the versatility and industrial relevance of electroless processes. The nucleation density and subsequent diffusion-controlled growth play pivotal roles in determining the microstructure of ELD coatings. As illustrated in Figure 3a, the density of initial nucleation sites profoundly affects the resulting coating morphology. When nucleation sites are uniformly and densely distributed, the growing nickel crystallites rapidly impinge upon one another, forming a fine-grained, continuous, and smooth coating. In contrast, sparser nucleation allows individual crystallites to grow larger before coalescence, resulting in a rougher, nodular surface texture with larger grain sizes. This initial distribution of nuclei sets the foundation for the coating's mechanical and electrochemical properties [43]. While the general mechanisms of nickel nucleation are well understood, additional factors such as grain boundary segregation, bath agitation, and impurity incorporation can significantly influence final film structure and conductivity.

Beyond nucleation density, the mass transport of ions within the diffusion layers surrounding each growing nucleus further influences the growth direction, as depicted in Figure 3b. The research assumed that a thicker diffusion layer forms between two nuclei (position 1) compared to that at the top (position 2). This facilitates faster growth along the vertical direction. In the early stages of deposition, isolated nuclei are surrounded by localised depletion zones where ion concentration drops. As deposition progresses and diffusion layers begin to overlap, lateral growth becomes restricted, favouring vertical columnar growth. This transition promotes the development of dense, vertically aligned columnar structures, characteristic of high-performance electroless nickel-boron and nickel-boron-tungsten coatings. The interplay between nucleation density and diffusion-limited crystallite growth ultimately governs the uniformity, roughness, hardness, and corrosion resistance of the final electroless coating [44].

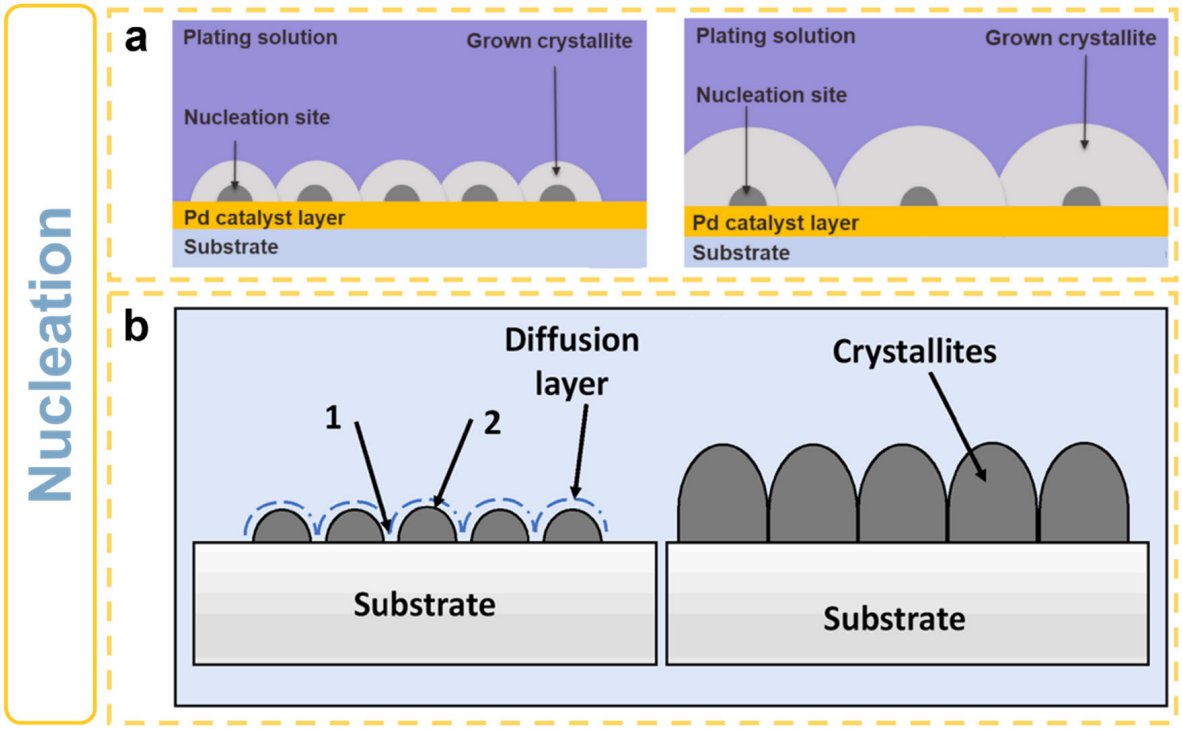


Figure 3. Schematic illustrations of the nucleation and growth behaviour during electroless deposition. (**a**) Effect of nucleation site density: densely distributed nucleation sites lead to fine-grained, smooth coatings, whereas sparse nucleation results in rougher, nodular coatings [43]. Copyright © 2021 MDPI. (**b**) Influence of diffusion layers during growth: overlapping diffusion layers between adjacent nuclei promote vertical columnar growth, leading to the formation of dense crystallite structures [44]. Copyright © 2022 ASME.

3. Electroless Nickel Deposition on Non-Conductive Surfaces

While electroless nickel deposition has proven effective on conductive surfaces, its application on non-conductive substrates, such as plastics, glass, and fabrics, presents significant challenges due to the inherent lack of conductive pathways necessary for the autocatalytic reaction to take place [45]. Nickel is often the metal of choice for electroless deposition due to its excellent combination of properties that make it highly suitable for various industrial applications [46]. One of the primary advantages of nickel is its ability to provide uniform coatings on complex geometries, including sharp edges and blind holes, without the need for external electrical current, which is a significant limitation in traditional electroplating techniques [47]. Nickel coatings are known for their excellent corrosion resistance, making them ideal for use in harsh environments, including marine and industrial applications [48]. Additionally, the hardness and wear resistance of Ni-P and Ni-B alloys are superior, mainly when heat-treated, providing durability and long-term performance in mechanical and abrasive settings [49].

Another significant benefit of nickel as a coating material is its versatility in modifying the bath chemistry to tailor the coating properties [48]. By adjusting the bath composition, pH, and temperature, the physical and mechanical characteristics of the nickel coating can be fine-tuned, resulting in coatings that range from soft and ductile to hard and wear-resistant [50,51]. This flexibility makes nickel an ideal candidate for applications requiring specific properties, such as in electronics, automotive, aerospace, and chemical processing industries [52]. Furthermore, nickel deposits tend to be more ductile than other hard coatings, such as chromium, allowing for its use in applications requiring toughness and protective qualities [53].

3.1. Challenges in Coating on Non-Conductive Surfaces

The main challenge in electroless nickel deposition on non-conductive substrates is that these materials lack the ability to catalyse the reduction in metal ions naturally. Unlike metals, which facilitate the deposition process, non-conductive surfaces must be treated before supporting autocatalytic plating. This surface modification is essential to trigger the reduction of nickel ions to metallic nickel, which is necessary to initiate the electroless deposition process [45,46].

Beyond initiating the plating process, poor adhesion is another significant obstacle when working with non-conductive materials [54]. These surfaces do not bond well with the nickel layer, often leading to weak adhesion, delamination, and reduced coating durability. Moreover, ensuring a uniform nickel layer is particularly difficult on non-conductive substrates with complex geometries, as the deposition process can become inconsistent without precise control of the process parameters [11]. Therefore, surface activation techniques and optimised plating conditions are crucial to overcoming these challenges. Several solutions have been developed to address these challenges, particularly surface activation and adhesion improvement. Table 2 provides the key components for electroless nickel coating on general non-conductive surfaces. The most employed technique is surface activation through catalytic particles, such as palladium, deposited on the surface before the electroless plating process [50]. This catalytic layer facilitates the reduction of nickel ions, thus enabling the deposition to begin [55]. Additionally, mechanical or chemical roughening of the surface is often employed to increase adhesion by providing a larger surface area and physical interlocking sites for the nickel layer [56,57]. This method enhances the bonding strength between the coating and the non-conductive substrate, thereby reducing the risk of delamination [58].

Another approach to improving the deposition process involves using polymer-assisted deposition techniques, where polymer layers, such as polyamines, are applied to the substrate before plating [56,59]. These polymers enhance the adhesion of metal ions to non-conductive surfaces and serve as a medium for catalyst immobilisation, thereby enhancing the efficiency of the deposition process [56]. Moreover, post-deposition annealing treatments have been shown to significantly improve adhesion by relieving internal stresses in the coating and promoting interfacial diffusion between the substrate and the nickel layer [53,59]. This treatment leads to stronger bonding and improved durability of the coatings.

Table 2. Roles of key components in the electroless nickel deposition process (table adapted [21,50]).

Component/ Parameter	Stage in ELD Process	Function	Examples
Surface Pretreatment Agents	Surface Preparation	Clean and roughen substrate to improve adhesion.	Acid etchants, plasma treatment, mechanical abrasion
Catalysts	Catalyst Immobilisation	Provide active sites to initiate the deposition reaction.	Palladium (Pd), Silver (Ag), Copper (Cu)
Metal Ions	Deposition Process	Source of nickel for coating formation.	Nickel sulphate ($NiSO_4$), Nickel chloride ($NiCl_2$)

Table 2. *Cont.*

Component/ Parameter	Stage in ELD Process	Function	Examples
Reducing Agents	Deposition Process	Supply electrons to reduce metal ions to metallic nickel.	Sodium hypophosphite (NaH_2PO_2), DMAB, $NaBH_4$
Complexing Agents	Bath Stabilisation	Prevent metal ions from precipitating and ensure controlled release.	Sodium citrate, EDTA, Lactic acid
Accelerators	Deposition Process	Enhance reaction rate and improve coating efficiency.	Fluoride ions, organic acids
Stabilisers	Bath Stabilisation	Prevent spontaneous decomposition of the plating bath.	Lead acetate, thiourea, organic surfactants
Buffers	pH Control	Maintain pH stability throughout the deposition process.	Ammonium salts, borate buffers
pH Regulators	pH Adjustment	Adjust and control bath pH to optimise deposition conditions.	Ammonia (NH_3), Sodium hydroxide (NaOH)
Temperature Control	Deposition Process	Provides the required activation energy for the reaction.	Heating systems, thermostats

3.2. Electroless Nickel Deposition on Non-Conductive Polymer Yarns

Synthetic yarns, such as nylon, polyester, and polypropylene, are widely used in various industries, including textiles, electronics, and medical applications, due to their high durability, flexibility, and resistance to environmental degradation [57,60]. These materials offer a range of mechanical properties that can be tailored for specific uses, including high tensile strength and elasticity, making them suitable for both conventional fabrics and advanced applications, such as wearable electronics and smart textiles [56,57]. However, synthetic yarns are inherently non-conductive [61], presenting a significant challenge when attempting to integrate functionalities, such as electrical conductivity, for applications in flexible electronics and sensors [62]. Electroless nickel deposition on synthetic yarns presents unique challenges primarily related to the non-conductive nature of the substrate [63]. Without the ability to conduct electricity, synthetic yarns do not naturally support the autocatalytic reactions necessary for electroless deposition [64].

Synthetic polyamide, is a widely used textile material known for its strength, flexibility, and chemical resistance [65]. However, its hydrophobic nature [66] and lack of reactive surface sites make it challenging to directly coat with metals using electroless deposition [67]. To overcome this limitation, the surface of the yarn must undergo a series of treatments to enhance its adhesion and reactivity. Common approaches include using surface activation techniques [59,68], such as chemical roughening [32] and applying a catalytic layer (often palladium-based) to initiate the nickel deposition process [50].

The functional performance of metallised synthetic yarns is highly dependent on both the ELD process conditions and the characteristics of the underlying polymer substrate. For instance, coating thickness and deposition uniformity directly influence electrical conductivity and flexibility, with thicker coatings often improving conductivity but increasing stiffness and reducing drape. However, variability in yarn composition (e.g., nylon 6 vs. 6,6), prior surface treatments, and even storage conditions (e.g., humidity) can

lead to inconsistent coating adhesion and performance. Moreover, environmental ageing effects such as UV exposure, thermal cycling, and mechanical abrasion are seldom addressed in the literature. This lack of standardisation and real-world performance data limits the comparability and industrial translation of metallised yarn systems. As such, future research should prioritise long-term reliability studies, develop test protocols for textile-based electronics, and establish clear correlations between deposition process parameters and functional yarn properties.

3.3. Surface Activation Techniques

3.3.1. Wet Chemical Layer Deposition

The acid surface modification [69], as illustrated in Figure 4a, involves a multi-step process where the substrate (e.g., Nylon 6 fabric) is first cleaned with acetone, immersed in an acid solution to introduce surface roughness and functional groups, and subsequently treated with a catalyst ink before undergoing electroless metal plating. Acid treatment is one of the most commonly used surface activation methods for electroless nickel deposition on non-conductive substrates. This process involves the application of strong acids such as sulfuric acid, nitric acid, or chromic acid to modify the surface characteristics of polymer fibres, enhancing adhesion and promoting catalyst immobilisation. Acid etching increases surface roughness by chemically dissolving the outermost polymer layers, exposing functional groups that facilitate metal ion bonding during ELD. The effectiveness of acid treatment lies in its ability to introduce reactive functional groups, such as hydroxyl and carboxyl groups, onto the polymer surface, significantly improving wettability and adhesion strength. This method is particularly advantageous for synthetic fibres like nylon and polyester, where surface hydrophobicity poses challenges for uniform coating formation. By enhancing surface roughness and porosity, acid treatment creates a mechanical interlocking effect that further enhances adhesion.

However, acid treatment has several limitations that must be addressed for optimal application. Prolonged exposure to strong acids can lead to excessive degradation of the polymer substrate, resulting in reduced mechanical strength and flexibility. Additionally, achieving uniform etching across the entire fibre surface can be challenging, leading to inconsistencies in the final coating quality. Another concern is the environmental impact of acid waste disposal, as the process generates hazardous by-products that require proper treatment and disposal methods. Despite these challenges, acid treatment remains a cost-effective and efficient method for large-scale surface activation in industrial applications. Researchers are exploring alternative acid formulations and hybrid approaches that combine acid treatment with other techniques, such as plasma or polymer coatings, to mitigate some drawbacks while retaining its adhesion-enhancing benefits. Future studies should focus on optimising treatment parameters to balance surface modification effectiveness with preserving fibre integrity.

The mussel-inspired surface modification method [70], shown in Figure 4b, leverages the adhesive properties of polydopamine (PDA) inspired by natural mussel adhesion mechanisms. In this process, dopamine undergoes self-polymerisation on fibre surfaces, forming a thin, adherent PDA layer that provides reactive sites for catalyst immobilisation and subsequent metal deposition. PDA chelates palladium ions, facilitating their uniform distribution and enhancing catalytic activity for ELD. The paper highlights how this technique results in durable and flexible conductive fibres with excellent mechanical and electrical properties, making them suitable for wearable electronic applications. Tannic acid (TA) serves as an effective wet chemical deposition agent in ELD due to its strong adhesion, metal ion complexation, and mild reducing properties [71]. Rich in catechol and galloyl groups, TA forms a stable interfacial layer on synthetic fibres, enhancing surface reactivity

for metal ion coordination [56]. Its ability to chelate metal ions, such as palladium and silver, improves dispersion and promotes uniform deposition while reducing agglomeration. Compared to traditional stannous chloride sensitisation, TA-based methods are more environmentally friendly and enhance adhesion strength, ensuring durable and conductive coatings [72]. This scalable approach has been successfully applied to metallising synthetic fibres for flexible electronics and conductive textile applications.

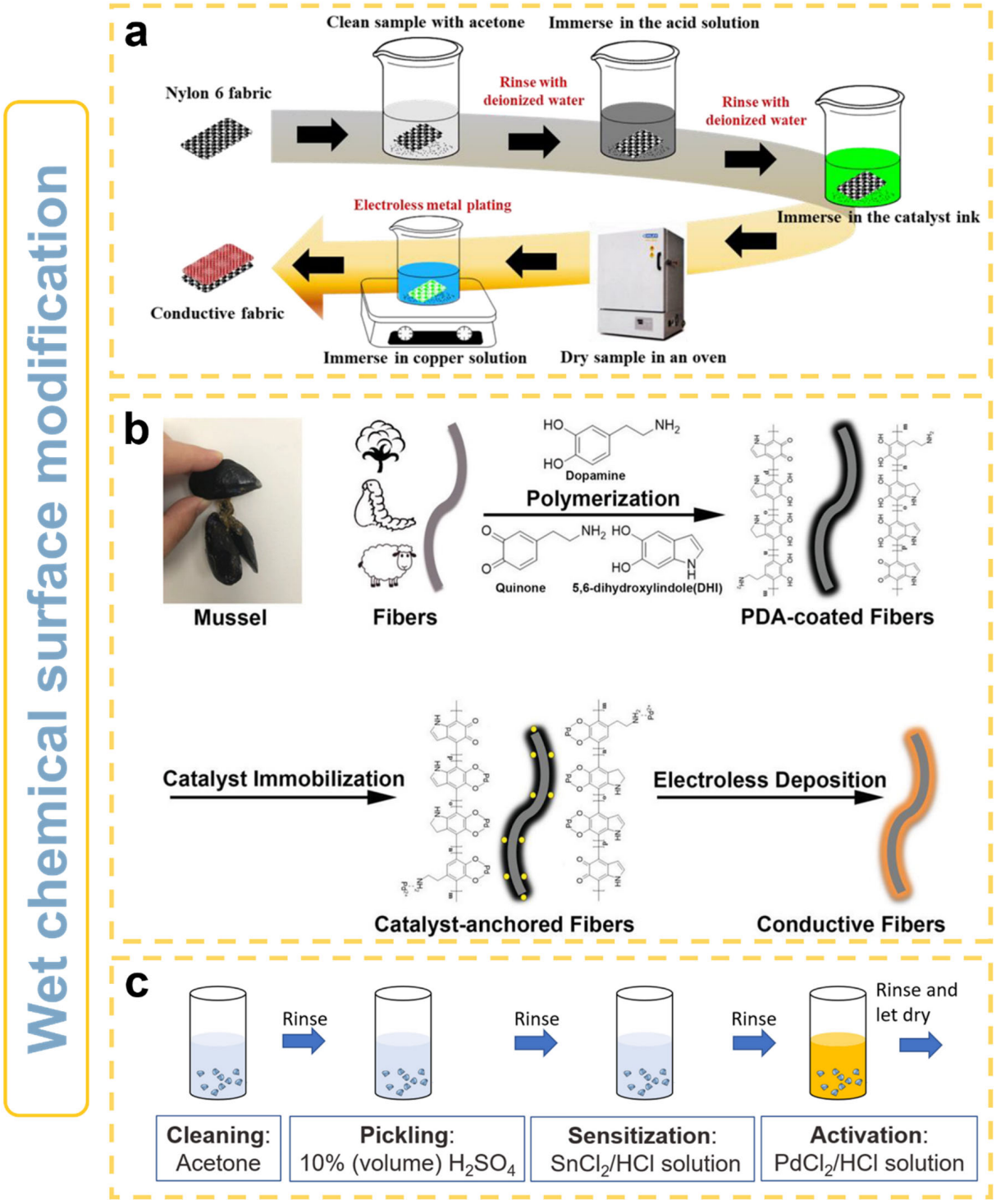


Figure 4. Surface modification strategies, (**a**) acid treatment [69]. Copyright © 2019 Elsevier. (**b**) PDA treatment [70]. Copyright © 2018 Wiley-Advance. (**c**) $SnCl_2$/HCl treatment [43]. Copyright © 2021 MDPI.

The pretreatment method employed by Ma et al. in Figure 4c involved a multi-step wet chemical process to activate diamond abrasive surfaces prior to electroless Ni-P deposition [43]. Initially, diamond particles were cleaned using acetone to remove organic contaminants. Subsequently, the particles underwent pickling in a 10% (volume) sulfuric acid (H_2SO_4) solution to eliminate surface oxides and further cleanse the surface. After thorough rinsing, sensitisation was performed using a 0.1 M $SnCl_2 \cdot 2H_2O$ and 0.1 M HCl solution at room temperature, which deposited a layer of Sn^{2+} ions onto the diamond surfaces. This sensitised surface was then activated by immersing in a $PdCl_2$/HCl solution, facilitating the replacement of tin ions with catalytically active Pd clusters. Finally, the activated particles were rinsed again and air-dried, resulting in a uniformly catalysed surface ready for effective and consistent electroless nickel-phosphorus deposition.

The wet chemical surface activation method in a study employs a layer-by-layer (LbL) approach using silane-based monomers to enhance metal adhesion on ABS polymers before the electroless deposition was reported by Chen et al. [73]. The surface is first modified with P-TES, which is UV-activated to form covalent bonds, followed by N-TES, introducing thiol groups for improved metal nucleation. Unlike $SnCl_2$-$PdCl_2$ sensitisation, this method provides a chemically bonded interface, significantly enhancing adhesion. Peel tests confirm strong metal retention on treated surfaces, demonstrating this scalable, eco-friendly alternative for metallisation in conductive coatings and industrial applications.

3.3.2. Plasma Treatment

Plasma treatment has emerged as an effective and environmentally friendly method for surface activation of non-conductive materials, such as synthetic yarns and polymer fibres, in preparation for ELD. This technique significantly enhances surface wettability, increases surface energy, and introduces reactive functional groups that promote adhesion and catalyst immobilisation without the use of harmful chemicals. Plasma treatment methods primarily include three categories: low-pressure plasma, atmospheric pressure plasma, and dielectric barrier discharge (DBD) plasma, each offering distinct advantages regarding scalability, processing conditions, and effectiveness.

Low-pressure plasma treatment operates under vacuum conditions, providing precise control over the surface modification process through reactive gases such as oxygen, nitrogen, or argon [74]. This method is highly effective for laboratory-scale applications requiring uniform surface modifications, though it involves higher operational costs and limited scalability for industrial processes. In contrast, atmospheric pressure plasma (APP) treatment (Figure 5a) eliminates the need for vacuum conditions, thus making it more suitable for industrial-scale applications. APP employs gases such as air, nitrogen, or argon, introducing oxygen-containing functional groups that significantly enhance surface adhesion without compromising the mechanical integrity of the substrate [75]. Although APP is cost-effective and scalable, careful management of discharge parameters is essential to prevent surface damage and achieve uniform modifications.

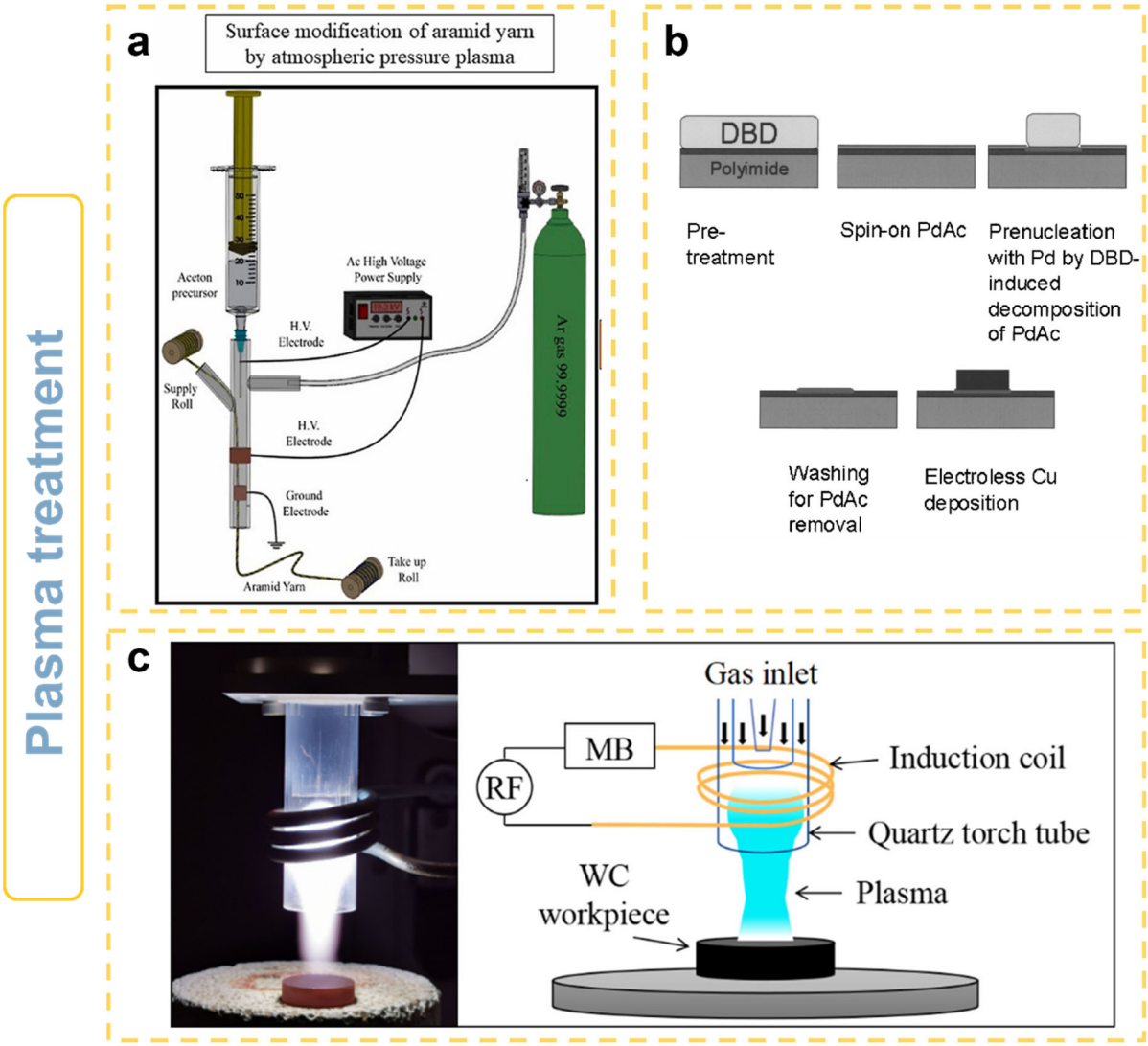


Figure 5. (**a**) A schematic diagram of the APP experimental setup [76]. Copyright © 2022 Elsevier. (**b**) Schematic diagram of the deposition process [77]. Copyright © 2000 Elsevier. (**c**) Schematic diagram of AP plasma experimental setup [78]. Copyright © 2023 Elsevier.

DBD plasma (Figure 5b) is another atmospheric pressure plasma technique that generates plasma through electrical discharges across a dielectric barrier [79]. DBD is extensively used in textile applications due to its capability to uniformly treat fibrous substrates while preserving their mechanical flexibility. It notably increases surface roughness and introduces functional groups essential for strong catalyst adhesion during ELD [77]. Nevertheless, excessive or improper treatment may lead to fibre degradation, and achieving uniform penetration in densely structured textiles remains challenging. Laser-assisted plasma treatments have further expanded the precision of plasma-based surface modifications, enabling selective surface texturing that significantly enhances mechanical interlocking and adhesion through tailored surface topographies. Such treatments combine laser ablation with plasma-induced chemical modifications, thus offering increased adhesion strengths and improved durability, which is particularly beneficial for ceramic and high-performance polymer substrates.

Guo et al. (2023) investigated inductively coupled atmospheric-pressure (ICP AP) plasma (Figure 5c) to enhance electroless Ni-P plating quality on binderless tungsten carbide (WC) [78]. Their ICP AP plasma system effectively activated WC surfaces through oxygen radical formation, increasing surface roughness and significantly enhancing surface

energy and wettability. This pretreatment facilitated the uniform growth of crystalline Ni-P coatings, reduced processing complexity and time, and improved plating adhesion, overcoming the limitations of traditional chemical treatments. However, extended treatment times increased surface roughness and caused isotropic oxidation, indicating a need for precise optimisation of process parameters.

Overall, plasma treatment substantially alters the surface properties of substrates, enhancing ELD outcomes by increasing surface energy, improving wettability, facilitating mechanical interlocking through surface roughening, and creating reactive sites for robust catalyst interactions [80]. While APP and DBD are highly suited for polymer and textile substrates, ICP AP plasma has shown particular promise for harder substrates like WC, demonstrating significant improvements in surface activation, coating uniformity, and adhesion strength. Nonetheless, carefully optimised plasma parameters, including gas composition, treatment duration, and discharge power, are essential to maximise benefits and prevent unintended substrate degradation. These advanced plasma-based activation techniques represent promising avenues for industrial applications, offering efficient, sustainable, and scalable alternatives to conventional surface activation processes.

In contrast, chemical methods targeting amide functionalities aim at selectively introducing reactive groups onto nylon surfaces without degrading the polymer backbone [81]. Such approaches precisely introduce functional groups like amines or hydroxyl groups, enhancing metal adhesion and coating uniformity. However, achieving durable and uniform coatings on synthetic yarns continues to pose significant challenges due to the inherent smoothness of fibre surfaces and mechanical flexibility, which often induce stress-related coating defects such as cracking or delamination during practical use [67]. Consequently, ongoing research is essential to explore novel surface activation methods, combined treatment strategies, and optimised deposition conditions to enhance the performance and stability of electroless nickel coatings on synthetic yarn substrates.

3.4. Roles of Surface Chemistry in Coating Adhesion

Surface chemistry is fundamental in determining the adhesion strength and durability of electroless nickel coatings on non-conductive substrates, particularly synthetic yarns. Table 3 summarises a comparison of surface modification methods for ELD on non-active substrates. Effective adhesion depends on the nature of surface functional groups, the interactions between the substrate and the deposited metal layer, and the chemical modifications employed to enhance metal-substrate bonding. By tailoring the surface chemistry, it is possible to improve coating uniformity, mechanical stability, and long-term performance in applications requiring robust conductive coatings.

One of the primary challenges in electroless nickel deposition on synthetic yarns, such as nylon, is their inherent chemical inertness and low surface energy. To address this, surface functionalisation methods introduce reactive groups that facilitate metal ion coordination. Various chemical treatments, including aminosilanes [82,83], thiols [84], PDA [70,85], tannic acid [56,71], and polyethylenimine (PEI) [86], have been employed to improve adhesion. Aminosilanes, such as (3-aminopropyl)triethoxysilane (APTES), introduce amine groups that form strong covalent bonds with the substrate and enhance nickel adhesion. Thiol-based modifications, including mercaptosilanes, provide high affinity for metal ions and are widely used in metal–polymer interface engineering [87]. PEI, a branched polyamine, enhances adhesion by providing a dense network of primary, secondary, and tertiary amines that can coordinate with metal ions [88]. These modifications create a chemically active surface that enhances nucleation and stability of the deposited metal layer. Surface energy directly influences the wetting behaviour of the electroless plating solution, impacting the uniformity and adhesion of the deposited coating. A higher surface energy facilitates

better solution spreading, leading to more uniform nickel deposition [89–91]. Chemical modifications that introduce polar functional groups, such as hydroxyl (-OH) and amine (-NH_2) groups, increase surface energy and improve wetting [92].

Various chemical interactions, including covalent bonding, electrostatic attraction, hydrogen bonding, and van der Waals forces, govern the adhesion of nickel coatings to modified polymer surfaces [93–95]. Covalent bonding arises when functional groups such as amines, thiols, or silanes chemically react with the polymer surface, forming stable linkages that enhance adhesion. Electrostatic attraction occurs when charged functional groups on the substrate interact with metal ions, aiding in the initial nucleation phase of electroless deposition [1,17]. Hydrogen bonding between surface-modified groups and nickel precursors further stabilises the deposited layer, promoting uniformity and reducing delamination risks [94]. Additionally, van der Waals forces contribute to adhesion in cases where specific chemical bonding is weak, but surface roughness facilitates mechanical interlocking [96]. Reducing agents in the electroless plating bath, such as sodium hypophosphite or dimethylamine borane (DMAB), drive redox reactions that enable strong metal-substrate interactions by facilitating the reduction of Ni(II) to metallic Ni(0) [97]. This complex interplay of chemical and physical forces ensures robust nickel adhesion, enhancing the mechanical stability and durability of the coating. Different surface modification strategies yield varying levels of coating adhesion and mechanical stability. Plasma treatment introduces oxygen-containing functional groups, enhancing adhesion through increased surface roughness and chemical activity. Acid etching modifies surface morphology while introducing polar functional groups, though excessive etching can degrade fibre integrity. Bio-inspired coatings balance adhesion enhancement and substrate preservation, making them ideal for flexible and mechanically resilient applications. Silane coupling agents and thiol-based surface treatments are also widely used due to their ability to form robust covalent bonds with organic and inorganic materials, further improving adhesion stability.

Table 3. Comparison of surface modification methods for ELD on synthetic yarns.

Surface Modification Method	Advantages	Limitations
Aminosilanes (e.g., APTES) [83]	Forms strong covalent bonds with the substrate, improves adhesion and stability, enhances metal ion coordination	Requires precise control of reaction conditions, and potential hydrolysis in humid environments.
Thiol-based Modifications (e.g., Mercaptosilanes) [98]	High affinity for metal ions, strong sulphur-metal interaction, improves the uniformity of nickel deposition	Can degrade over time due to oxidation, sensitive to environmental conditions.
PDA Coating [70]	Bio-inspired adhesion, improves surface hydrophilicity, provides functional groups for catalyst immobilisation	Requires alkaline conditions for polymerisation, may lead to excessive surface roughness.
TA Treatment [71]	Introduces phenolic hydroxyl groups for metal ion binding, enhances adhesion without aggressive etching	Selective adhesion properties, less effective on highly hydrophobic surfaces.
PEI Functionalisation [86]	Provides dense network of amines, enhances catalyst immobilisation, flexible and adaptable to various substrates	Can lead to excessive surface charge, may require cross-linking for stability.

Table 3. *Cont.*

Surface Modification Method	Advantages	Limitations
Plasma Treatment [78]	Increases surface energy and wettability, creates functional groups for better coating adhesion	Requires specialised equipment, may cause fibre degradation with prolonged exposure.
Acid Etching [99]	Improves mechanical interlocking, introduces reactive functional groups, cost-effective	Can degrade fibre integrity, produces hazardous waste requiring proper disposal.
Silane Coupling Agents [100]	Strong bonding with both organic and inorganic surfaces, enhances long-term stability	Sensitivity to moisture, requires careful selection of silane type for optimal results.

In addition to conventional surface treatments such as plasma, acid etching, or bio-inspired coatings, alternative activation strategies have been explored to improve catalyst adhesion and coating uniformity. One notable approach involves the use of self-assembled monolayers (SAMs), such as 3-aminopropyltrimethoxysilane (APTMS), which enable the immobilisation of Pd(II) ions through coordination bonding to amino groups, offering enhanced adhesion and Sn-free activation routes. For example, Guo et al. demonstrated electroless nickel deposition on polyester fabric via APTMS-mediated Pd activation, resulting in improved coating continuity and EMI shielding performance [101]. Similarly, nanoparticle-based catalytic systems have been investigated to improve dispersion and reduce Pd usage, though their applicability to synthetic yarns remains relatively underexplored.

Despite advancements in surface modification, challenges remain in achieving optimal adhesion while maintaining the mechanical properties of the substrate. Issues such as delamination, coating brittleness, and environmental degradation need to be addressed through further research. Future developments may focus on hybrid approaches that combine multiple surface treatments to maximise adhesion efficiency without compromising flexibility. Additionally, integrating sustainable and non-toxic surface modification methods will be crucial for widely adopting electroless nickel coatings in environmentally sensitive applications.

4. Exploring Solvent Systems in Electroless Nickel Deposition

The choice of solvent in electroless nickel deposition significantly affects the deposition rate, coating quality, and microstructural properties of the resulting nickel layer [102]. Solvents can be broadly categorised into polar protic solvents, polar aprotic solvents, and non-polar solvents [103]. Each type interacts differently with the reducing agent, nickel ions, and substrate, leading to variations in coating characteristics. In electroless deposition, solvents can be used as the primary medium or as co-solvents in an aqueous-based plating bath to fine-tune the deposition process [102,104].

4.1. Polar Protic Solvents

Polar protic solvents, such as water and alcohols, are characterised by their ability to donate hydrogen bonds [105], which makes them highly effective for dissolving ionic compounds like nickel salts [106]. These solvents can stabilise charged species in the solution and facilitate the reduction of nickel ions by the reducing agent [107]. Water is the most used polar protic solvent in electroless nickel deposition [104]. Still, other alcohol-based solvents, such as ethanol and methanol, have also been explored for their unique effects on the deposition process [108,109].

4.1.1. Water

Water is the most used solvent in electroless nickel deposition, forming the foundation for nearly all commercial applications. Its high dielectric constant allows it to efficiently dissolve nickel salts (Ni^{2+}) and reducing agents like sodium hypophosphite or dimethylamine borane (DMAB) [1,11,110], enabling effective deposition. The mechanism of nickel deposition in aqueous systems relies on the autocatalytic reduction of nickel ions in the presence of hydroxide ions (OH^-), stabilising the reaction and promoting continuous deposition on the substrate [111]. This reaction leads to uniform coatings on conductive and non-conductive substrates, such as polymers and textiles, making aqueous electroless nickel deposition widely used in various industries [1,97].

A typical aqueous bath consists of nickel salts, reducing agents, stabilisers, and complexing agents that regulate the deposition rate and ensure bath stability [38,112,113]. This method is valued for its cost-effectiveness and scalability, particularly in industrial settings where large-scale applications require efficient processes. The high solubility of nickel salts and reducing agents in water ensures consistent deposition rates, while the resulting coatings are well-regarded for their excellent corrosion resistance and mechanical strength. This makes aqueous-based nickel deposition particularly suitable for environments exposed to harsh conditions, such as marine, aerospace, and automotive industries [104].

However, aqueous systems present several challenges. One of the most significant is incorporating hydrogen during the plating process. Hydrogen, produced as a by-product of the reduction reaction, can become trapped in the Ni-P alloy, leading to internal stresses and micro-cracks formation [97,104]. These cracks can compromise the mechanical integrity of the coating, especially in applications where durability is critical. Another challenge is the bath instability caused by the buildup of reaction by-products, which can reduce the efficiency and quality of the deposition process over time. As a result, careful monitoring and periodic replenishment of the bath components are necessary to maintain optimal conditions [104]. Aqueous-based electroless nickel deposition also faces difficulties when applied to non-conductive substrates like textiles [67]. Nylon yarns, for instance, which are hydrophobic and chemically inert [47,114], prevent the nickel ions from properly adhering to the surface [115]. The coating may be uneven and have poor adhesion without sufficient surface preparation, such as chemical etching or plasma treatment [116,117]. To address this, surface modification techniques are often used to enhance the wettability and reactivity of the substrate, enabling better interaction with the nickel ions in the aqueous solution [20,92,118]. Despite these efforts, achieving strong adhesion and uniform coating on flexible materials like textiles remains challenging [119].

Research by Schlesinger and Paunovic (2010) demonstrated that aqueous systems produce highly uniform and adhesive coatings with excellent corrosion resistance on various substrates but also highlighted the rigidity of the crystalline nickel layer as a drawback for flexible applications [2]. Similarly, Mallory and Hajdu (1990) found that while aqueous electroless nickel deposition excels in producing hard, protective coatings, the resulting coatings lack the flexibility for dynamic substrates like textiles [1]. These findings have led to ongoing research on modifying aqueous-based deposition systems to achieve more compliant coatings without sacrificing durability [119]. Despite these challenges, aqueous-based electroless nickel deposition remains one of the most effective and widely used methods in many industries. Further research is needed to optimise this method for non-conductive, flexible substrates by exploring additives, co-solvents, or alternative deposition conditions that enhance flexibility while maintaining the high-quality coating properties associated with nickel.

4.1.2. Ethanol

In recent years, ethanol has gained attention as an effective solvent in electroless nickel deposition, particularly as an alternative to traditional aqueous systems. Ethanol-based deposition improves the quality and uniformity of nickel coatings, especially in applications involving porous or small-pore substrates [119]. Its lower surface tension than water allows the plating solution to infiltrate fine pore structures, enabling more uniform deposition throughout the substrate [118]. This has proven highly beneficial in fabricating porous materials such as nickel foams, where ethanol-based systems have successfully produced foams with pore sizes in the micrometre range and porosities exceeding 90% [109]. These properties are difficult to achieve with water-based systems, where nickel tends to deposit only on the outer surfaces due to insufficient infiltration.

In a recent study by Wang et al. [120], ethanol-based electroless nickel deposition was applied to PA6.6 yarns using dimethylamine borane as the reducing agent. The resulting coatings exhibited amorphous–nanocrystalline structures with finer crystallite size (3.2–4.6 nm by XRD), improved thermal stability (~425 °C) compared to aqueous-deposited coatings. Moreover, elevated temperatures in the ethanol bath further refined the grain structure, enhancing flexibility and reducing internal stress. This work highlights ethanol's unique capability in achieving conformal and functional Ni coatings on polymeric substrates, something rarely attainable in water-based systems.

Although less polar than water, alcohols like ethanol and methanol are also classified as polar protic solvents [121,122]. When used as co-solvents with water or as the primary solvent in electroless deposition, they influence the solubility of metal ions and reducing agents [104]. Ethanol alters the interaction between nickel ions [123] and the reducing agent due to its lower polarity, resulting in thinner and more flexible coatings than those produced in water-based systems [122]. This characteristic makes ethanol-based systems advantageous in applications requiring a balance between flexibility and durability. While methanol shares similar benefits, it is less commonly used due to its higher volatility and associated safety risks in industrial settings [124]. Nonetheless, both ethanol and methanol have been shown to produce softer and more flexible coatings, making them ideal for specific applications that require such properties.

One of the key benefits of ethanol-based electroless deposition is its ability to reduce hydrogen inclusion during the plating process. This leads to smoother, less brittle coatings with fewer internal stresses [121,125]. Such coatings are particularly advantageous in high-performance applications, such as energy storage devices, where nickel foams with small pore sizes and high surface area are critical [109]. Additionally, ethanol improves the co-deposition of hydrophobic particles like SiO_2, which are challenging to incorporate in aqueous solutions [119]. Furthermore, ethanol offers a wider electrochemical window and enhanced thermal stability, making it suitable for filtration systems, noise and vibration absorption, and 3D graphene substrates [109,126]. Despite these advantages, ethanol-based electroless nickel deposition comes with certain limitations. The use of organic solvents, such as ethanol, increases the overall cost of the process compared to aqueous systems. Ethanol's flammability also raises safety concerns, requiring careful handling and storage protocols [127]. Nevertheless, ethanol-based systems remain a viable alternative for producing high-quality nickel coatings, particularly in specialised applications where traditional aqueous solutions fall short.

4.2. Polar Aprotic Solvents

Polar aprotic solvents are a class of solvents that, while polar, do not have hydrogen atoms directly bonded to electronegative atoms like oxygen or nitrogen, meaning they cannot donate hydrogen bonds [128]. These solvents are particularly useful in reactions

involving nucleophiles and electrophiles because they dissolve ionic compounds well without engaging in hydrogen bonding, thus enhancing the reactivity of certain ions [129,130]. In electroless deposition, polar aprotic solvents such as dimethyl sulfoxide (DMSO) and ethylene glycol play critical roles in enhancing the dissolution of metal salts and improving the overall deposition process [131,132]. By facilitating better solubility of the metal ions and reducing agents, these solvents help produce more uniform and high-quality metal coatings on conductive and non-conductive surfaces [133].

DMSO, as a polar aprotic solvent used in electroless deposition, is favoured for its high dielectric constant and ability to dissolve a wide range of organic and inorganic substances [134,135]. This allows for the efficient deposition of metals such as silver, copper, and nickel, with improved adhesion and mechanical properties on challenging substrates. On the other hand, ethylene glycol, another essential polar aprotic solvent, offers the advantage of acting both as a solvent and a reducing agent, streamlining the deposition process by reducing the need for additional toxic chemicals [136,137]. The dual functionality of ethylene glycol and its ability to produce smoother coatings and better control particle size make it a versatile solvent in environmentally friendly electroless deposition processes [138].

4.2.1. DMSO

DMSO has been effectively employed in electroless plating processes, primarily to enhance the adhesion and uniformity of metal coatings on polymeric substrates. Xue et al. demonstrated the beneficial role of DMSO when grafting 1,2-ethylenediamine (EDA) onto poly(ethylene terephthalate) (PET) surfaces prior to electroless copper plating [139]. DMSO as a solvent during surface treatment significantly increased the surface roughness of PET substrates, improving the anchoring and adsorption of catalytic gold particles. Consequently, this enhanced the adhesion strength of copper layers compared to traditional aqueous-based grafting methods, resulting in superior mechanical properties and reliable metal-polymer bonding. Similarly, Cordonier et al. described the incorporation of DMSO in the formation of photoreactive titanium-copper complex solutions for electroless deposition [140]. DMSO was utilised as a co-solvent to improve the solubility and stability of the metal complexes, facilitating uniform deposition and enhancing the formation of catalytic oxide layers upon pyrolysis. This method resulted in increased plating adhesion and enabled the precise formation of sub-micrometre metallic patterns, illustrating the versatility and utility of DMSO in micro-patterning applications. Vasconcelos et al. used DMSO to create a physical interpenetrating polymer network on thermoplastic polyurethane (TPU) by dissolving polyvinylpyrrolidone (PVP) in DMSO and subsequently immersing the polymer substrate [141]. This approach significantly improved the surface compatibility and chemical affinity for silver ions, providing strong adhesion for subsequent electroless silver plating. The resultant silver coatings demonstrated excellent mechanical robustness, high conductivity, and stable electrochemical performance, highlighting DMSO's critical role in polymer functionalisation processes aimed at metallisation.

Pang et al. introduced a distinctive approach to electroless nickel deposition by utilising DMSO not merely as a solvent but as a swelling agent to facilitate catalyst incorporation into the near-surface region of Kevlar fibres [142]. Unlike previous studies that employed DMSO to enhance surface roughness or solubility, such as Xue et al., who used it to increase anchoring of amine groups on PET, or Cordonier et al., who used it to stabilise photoreactive metal complexes, Pang et al. leveraged DMSO's ability to disrupt the dense, crystalline Kevlar structure. This allowed silver nitrate to penetrate and uniformly embed beneath the surface, where it was reduced to catalytic silver nanoparticles, enabling highly adherent nickel plating without damaging the fibre. Compared to the polymer-surface-level modi-

fications achieved in prior work, Pang et al.'s method uniquely enhances catalyst depth and distribution through a physical swelling mechanism, making it especially valuable for modifying high-performance, chemically inert fibres like Kevlar that resist conventional surface treatments.

Another benefit of DMSO is its enhanced solubility of metal salts and complexing agents, which leads to more uniform and controlled metal deposition. This is particularly advantageous when plating on complex geometries or porosity substrates, where achieving consistent deposition with aqueous systems can be challenging. DMSO-based solutions allow for better penetration of metal ions into intricate surfaces, producing smoother and more homogeneous coatings [141]. Additionally, DMSO facilitates improved adhesion of metal coatings to non-metallic substrates, such as polymers. The solvent swells the polymer, enabling metal ions to penetrate the surface more effectively, resulting in enhanced mechanical interlocking and improved adhesion strength. DMSO's ability to reduce the deposition temperature is another significant advantage. Electroless plating in DMSO can be carried out at lower temperatures than in aqueous systems, which is particularly beneficial for thermally sensitive materials like polymers. Using DMSO, the researchers improve the adhesion strength and uniformity of the nickel coating on the fibre surface, resulting in a dense, smooth nickel layer. Recently, direct incorporation of DMSO as a co-solvent in aqueous electroless nickel baths has emerged as a promising strategy for tuning interfacial properties between polymer substrates and metal coatings. For example, Wang et al. [143] demonstrated that introducing 1 wt.% DMSO into a standard alkaline Ni-ELD bath significantly improved coating uniformity, reduced grain size, and enhanced thermal, mechanical, and electrochemical stability of nickel-coated nylon-6,6 yarns. These effects were attributed to DMSO's ability to plasticise the nylon-6,6 surface, facilitate catalyst infiltration, and regulate nickel nucleation and growth kinetics. This study establishes a clear link between solvent environment and coating performance, supporting the potential of solvent-assisted metallisation strategies for wearable electronics and functional textiles. Furthermore, DMSO is considered relatively environmentally benign compared to other organic solvents, with lower toxicity and minimal environmental impact. This characteristic positions DMSO as a safer and more sustainable option for industrial electroless deposition processes.

Despite its advantages, DMSO also presents several limitations. The most notable drawback is the higher cost associated with DMSO compared to water-based systems. The added expense can make large-scale industrial adoption less feasible, although it may still be justified for specialised applications where the benefits outweigh the costs [141]. In addition, while DMSO is generally safe, its strong solvating power can result in solvent retention within polymer substrates. This can affect the long-term mechanical properties of the substrate, as residual solvent may alter the material's flexibility and durability over time [144]. Handling and formulating DMSO-based baths also introduce some complexities. The solvent can interact with other bath components, such as reducing agents and stabilisers, which may affect deposition kinetics. As a result, the formulation of DMSO-based baths requires careful optimisation to avoid inconsistencies in the final coating. This increased complexity may add time and effort to the process, limiting its practical use in certain settings [131,145].

4.2.2. Ethylene Glycol

Ethylene glycol has emerged as a promising solvent in electroless deposition processes, offering several advantages in the fabrication of metal coatings, particularly for copper and nickel. It serves as a solvent and a reducing agent, allowing for metal ion reduction without additional toxic reducers such as formaldehyde [146,147]. This dual function makes it

attractive for environmentally friendly electroless deposition, as it reduces the chemical load in the process while still enabling the effective deposition of metals on both conductive and non-conductive substrates.

One of the key advantages of ethylene glycol-based electroless deposition is its ability to provide greater control over the morphology and particle size of the deposited metal [148]. Studies have shown that using ethylene glycol can result in smaller and more uniform nanoparticles, particularly when depositing metals like nickel and copper [132,138]. For instance, nickel-cobalt nanoparticles synthesised using ethylene glycol exhibited a high degree of uniformity and small particle sizes, which is beneficial in applications requiring fine control over the deposited material's microstructure [132]. Additionally, ethylene glycol allows for the formation of coatings with superior smoothness and reduced internal stresses compared to water-based systems. This makes it an excellent choice for depositing metals on complex geometries and porous substrates. Moreover, ethylene glycol's low vapour pressure and relatively high boiling point provide stability during the deposition process, particularly at elevated temperatures. This thermal stability allows for more consistent deposition, even at higher process temperatures, without significant solvent evaporation [149]. This characteristic is especially advantageous when depositing metals that require higher temperatures for reduction, such as nickel and cobalt.

Using ethylene glycol in electroless deposition also presents certain limitations. One key challenge is the relatively slow deposition rate compared to aqueous systems [150]. While ethylene glycol can provide superior control over particle size and morphology, the deposition kinetics are slower, which can extend the overall process time. This limitation may reduce its efficiency in industrial applications requiring high throughput. Additionally, although ethylene glycol is less toxic than some traditional solvents, it still requires careful handling due to its potential health risks upon exposure and environmental impact if not properly managed. Ethylene glycol facilitates uniform deposition, and improper control of process parameters can lead to byproducts or large metal clusters; however, it may reduce the purity and quality of the resulting coating [148,151].

4.3. Non-Polar Solvents

Non-polar solvents like hexane have garnered interest in electroless deposition processes due to their specific interactions with certain substrates, particularly non-conductive polymers like polyimide. These solvents, characterised by their low dielectric constants and inability to form hydrogen bonds, are crucial in facilitating the initial catalysation step in electroless plating [152]. In this step, the solvent aids in depositing catalytic particles, such as palladium (Pd), onto the surface of the substrate, which is essential for initiating metal plating. While non-polar solvents are not commonly used in electroless deposition baths themselves, they have been explored in pre-treatment or catalyst application steps due to their unique interfacial properties [153].

One of the primary advantages of using non-polar solvents, such as hexane, in electroless deposition is their chemical affinity with hydrophobic substrates like polyimides. This affinity enables the solvent to interact favourably with the substrate surface, allowing for the deposition of catalytic particles required for metal plating. Additionally, non-polar solvents are simpler to handle than more complex systems like supercritical carbon dioxide (Sc-CO_2). Hexane, for instance, can be used at standard atmospheric pressure, eliminating the need for specialised high-pressure equipment. This makes non-polar solvents more accessible for small-scale laboratory processes or applications where advanced infrastructure may not be available [153].

However, the use of non-polar solvents in electroless deposition is not without limitations. A significant drawback is their low diffusion coefficient, which hinders the deep

penetration of catalytic particles into the substrate. As a result, the adhesion of the deposited metal layer is weaker compared to when high-diffusion solvents like Sc-CO_2 are used. This insufficient penetration of the catalyst results in thin, unstable films that are prone to surface defects such as cracks and peeling. Moreover, non-polar solvents tend to produce films with lower mechanical strength [153]. This weakness is particularly pronounced when the metal films are subjected to thermal or mechanical stress, as the mismatch in thermal expansion between the metal film and the polymer substrate often leads to film failure.

Despite these limitations, non-polar solvents offer distinct benefits in specific contexts. Their chemical stability and low reactivity with other components in the deposition bath reduce the likelihood of unwanted side reactions during the plating process. Additionally, the ease of use and simple handling of non-polar solvents like hexane make them an attractive option for certain experimental setups or applications where process simplicity is a priority. Nonetheless, the weaker adhesion and compromised mechanical properties of the resulting metal films limit the broader applicability of non-polar solvents in high-performance or industrial settings [154].

Non-polar solvents provide several advantages in electroless deposition. They exhibit good chemical affinity with hydrophobic substrates, facilitating catalyst deposition on materials like polyimides. The ease of handling non-polar solvents, such as hexane, makes them convenient for smaller setups that do not require complex equipment like high-pressure systems. Additionally, their low reactivity with other bath components helps reduce unwanted chemical interactions, simplifying the deposition process [154,155]. However, non-polar solvents also have notable limitations. Their low diffusion coefficient results in insufficient catalyst penetration, leading to weaker adhesion and poorer film quality. Films produced using non-polar solvents often suffer from surface defects, such as cracks and peeling, particularly when subjected to mechanical or thermal stress. Furthermore, the lower mechanical strength of these films restricts their use in applications requiring robust and durable coatings [153,154].

5. Microstructure of Electroless Nickel Coatings

The microstructure of electroless nickel coatings plays a critical role in determining their mechanical, electromechanical and electrochemical properties. The structure of the nickel deposit, whether crystalline, amorphous, or amorphous-nanocrystalline, highly depends on the deposition conditions, including the type of solvent used, the temperature and pH of the plating bath, and the reducing agent—for example, Ni-P coatings with high phosphorus content (greater than 8%) tend to form amorphous structures, while lower phosphorus levels result in crystalline deposits [3,156]. In Ni-P systems, high phosphorus content results in amorphous coatings, which provide excellent corrosion resistance due to the absence of grain boundaries. Conversely, low phosphorus content leads to crystalline structures with moderate hardness and wear resistance. On the other hand, nickel-boron coatings typically exhibit higher hardness and wear resistance than Ni-P coatings, especially when subjected to heat treatment [9]. The heat treatment process induces the formation of hard nickel boride (Ni_3B) phases, further enhancing the mechanical properties of the coating [157]. Heat treatment can significantly alter the microstructure and corrosion behaviour of electroless nickel coatings. For example, annealing of amorphous Ni-B coatings leads to crystallisation and the formation of Ni_3B and Ni phases, which can either improve or reduce corrosion resistance depending on the thermal parameters and alloy composition [158]. These microstructural characteristics directly influence the coating's ability to withstand mechanical stresses, such as bending, stretching, or abrasion, which are particularly relevant for applications on flexible substrates like textiles [159]. While the general properties of electroless nickel coatings have been widely reviewed in the context

of aqueous baths, there is limited discussion on how the choice of solvent system influences the resulting coating's crystallinity, morphology, and performance. This section highlights recent studies on solvent-engineered electroless plating baths, examining how variations in solvent polarity and coordination chemistry affect the structure-property relationships of the coatings, especially in the context of flexible or polymeric substrates.

5.1. Crystalline Nickel

Nickel, in its crystalline form, is predominantly characterised by a face-centred cubic (FCC) crystal structure (Figure 6) [160]. It is widely valued for its excellent mechanical properties, thermal stability, and corrosion resistance, making it highly versatile in industrial metallurgy [161]. Its crystalline structure provides the basis for its strength, ductility, and formability. Crystalline nickel can be categorised into single-crystalline, polycrystalline, and nanocrystalline forms, each exhibiting distinct microstructural characteristics influencing their mechanical properties. These variations in atomic arrangement and grain structure allow nickel to be tailored for specific applications, ranging from high-performance components to coatings and films, where different balances of strength, ductility, and hardness are required.

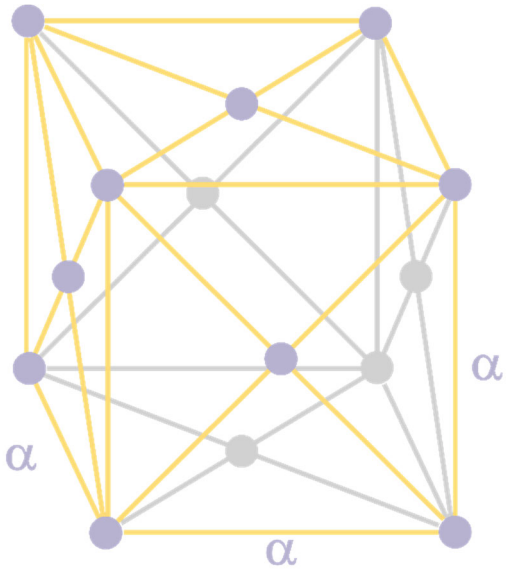


Figure 6. Nickel FCC crystal structure.

5.1.1. Single-Crystalline

Single-crystalline nickel is widely used in high-performance applications such as turbine blades and aerospace components due to its superior creep resistance, corrosion resistance, and high-temperature strength, properties derived from the absence of grain boundaries. These alloys are typically engineered with γ' precipitates in a γ matrix, which impede dislocation movement and enhance mechanical stability under thermal stress [162–164]. However, it is important to note that single-crystalline nickel cannot be produced via electroless deposition, as the process involves simultaneous nucleation at multiple sites, inherently resulting in polycrystalline or amorphous structures. This section is included briefly to distinguish electroless coatings from other nickel microstructures used in advanced engineering contexts [165].

5.1.2. Polycrystalline

Nickel polycrystalline materials, formed from multiple grains of nickel, exhibit distinct mechanical and physical properties compared to single crystalline materials due to the

presence of grain boundaries [166]. These grain boundaries play a key role in defining the material's strength, hardness, and resistance to wear and corrosion. Polycrystalline nickel, especially when deposited through electroless deposition methods, has gained widespread use across industries for producing protective coatings on various substrates, including metals and non-metals [166,167].

Electroless deposition is a popular technique for producing polycrystalline nickel coatings because it allows uniform coatings on complex geometries without requiring an external electrical current. In electroless nickel deposition, the reduction of nickel ions occurs in an aqueous solution using a reducing agent like sodium hypophosphite. This leads to forming a nickel-phosphorus alloy, which typically exhibits a polycrystalline structure with fine grain sizes. Several factors, including the pH of the bath, temperature, and the concentration of nickel ions and phosphorus, heavily influence the structure of the deposited film [3,166].

The microstructure of polycrystalline nickel coatings produced via electroless deposition can be manipulated through heat treatment. Post-deposition heat treatment can lead to grain growth and the precipitation of secondary phases like Ni_3P, which affects the hardness and wear resistance of the material [167]. Heat treatment above 300 °C promotes the growth of nickel grains and the formation of Ni_3P, while temperatures exceeding 600 °C can cause the coatings to lose hardness due to grain coarsening. Researchers have shown that controlling the phosphorus content and deposition conditions allows fine-tuning grain size and resulting mechanical properties. For example, deposits with lower phosphorus content tend to exhibit more crystalline structures, while higher phosphorus content leads to amorphous or mixed amorphous-crystalline phases, which have different mechanical behaviours [166].

Nickel polycrystalline coatings via electroless deposition offer an efficient and versatile method for producing durable and corrosion-resistant coatings. The process parameters and post-treatment conditions, such as bath composition and heat treatment, allow for precise control over the grain structure and mechanical properties, making polycrystalline nickel coatings suitable for various industrial applications. Further research continues to explore how the microstructure can be optimised to enhance specific properties like wear resistance and corrosion protection in challenging environments [168].

5.1.3. Nanocrystalline

Nanocrystalline nickel (NC-Ni) coatings have garnered significant attention due to their exceptional mechanical properties, wear resistance, and corrosion protection [169]. These coatings are produced through electroless deposition, a chemical reduction method that allows for the formation of nanocrystalline nickel coatings without the need for an external electrical current. This makes the process highly suitable for coating complex geometries and non-conductive surfaces. The ultra-fine grain structure of nanocrystalline nickel, often in the range of 10–100 nm [170,171], imparts unique properties that make these coatings desirable for various high-performance applications, including electronics, automotive, and aerospace industries [9]. Ni-P is one of the most common alloys through electroless deposition, with the phosphorus content playing a crucial role in determining the microstructure and properties of the coating [169]. Low phosphorus content, generally below 3%, results in a more crystalline nickel structure, while higher phosphorus levels lead to a mixed amorphous-nanocrystalline structure [172]. These nanocrystalline structures balance hardness, toughness, and corrosion resistance. One of the key advantages of NC-Ni coatings is their high hardness, achieved through fine grain sizes and a compact microstructure. This high hardness and low porosity contribute to excellent wear resistance and enhanced barrier properties against corrosive environments [173].

An important enhancement to nanocrystalline coatings is the incorporation of alloying elements or hard particles, such as tungsten (W), molybdenum (Mo), or boron (B), which further improves the mechanical and thermal properties of the coatings. For example, alloys like Ni-W-P and Ni-Mo-P provide better thermal stability and hardness, making them suitable for high-temperature applications. Quaternary coatings, such as Ni-W-Mo-P, are more crystalline due to the lower phosphorus content, improving mechanical properties. In the case of Ni-B coatings, the addition of boron carbide (B_4C) particles significantly enhances hardness, wear resistance, and adhesion, making these coatings particularly suitable for applications requiring high durability [172,174]. However, despite the superior properties of nanocrystalline nickel coatings, the electroless deposition process presents certain challenges. Achieving consistent grain sizes and preventing the coalescence of nanocrystals requires precise control of the bath composition and deposition parameters [175]. Heat treatments often improve the hardness and mechanical properties by promoting grain growth and phase transformations [176]. However, this can sometimes reduce the toughness of the coating and lead to brittleness [9]. Another significant challenge is managing the internal stresses that develop during deposition, which can lead to cracking if not properly controlled [9,176,177].

Nanocrystalline nickel coatings produced via electroless deposition offer a combination of high hardness, wear resistance, and corrosion protection, particularly when alloying elements or reinforcement particles are incorporated. However, the success of these coatings depends on carefully optimising the deposition process and post-deposition treatments to ensure uniformity, minimise internal stresses, and prevent defects such as cracking and grain growth. These coatings are highly desirable for various industrial applications requiring superior mechanical properties and durability.

5.2. Amorphous-Nanocrystalline Nickel

Amorphous-nanocrystalline nickel coatings, produced through electroless deposition, are highly valued due to their unique combination of amorphous and crystalline structures. This hybrid material consists of nanocrystalline nickel grains dispersed within an amorphous matrix [178]. The nanocrystalline regions retain the FCC crystal structure of nickel, providing localised areas of atomic order, while the amorphous phase lacks long-range atomic arrangement [179,180]. This combination of ordered and disordered phases imparts exceptional mechanical properties, as the crystalline grains contribute to strength and ductility, while the amorphous matrix enhances hardness, corrosion resistance, and wear resistance [178,181].

The formation of amorphous-nanocrystalline nickel typically occurs through electroless deposition, electrodeposition, or rapid solidification processes, where the deposition conditions or cooling rates are carefully controlled to prevent full crystallisation. In electroless deposition, the Ni-P or Ni-B alloys are widely used, with the phosphorus or boron content playing a critical role in determining the microstructure. Phosphorous contents between 5% and 9% are typically associated with a hybrid structure [182,183], balancing the high hardness of nanocrystalline materials and the corrosion resistance of amorphous phases [184,185]. Nanocrystals embedded within the amorphous matrix provide multiple advantages, such as reducing internal stresses, enhancing hardness, and improving wear resistance [181]. Nanocrystalline grains act as barriers to dislocation motion, strengthening the material, while the amorphous phase restricts grain growth, leading to improved thermal stability [186]. This hybrid microstructure provides the material with excellent mechanical properties, combining the high strength and hardness of nanocrystalline structures with the toughness and resistance to brittle fracture associated with amorphous phases [179,183,187].

Incorporating alloying elements such as W, Mo, or silicon dioxide (SiO_2) nanoparticles further enhances the performance of amorphous-nanocrystalline coatings [188]. These elements improve hardness, wear resistance, and thermal stability, making the coatings suitable for high-temperature applications and protective engineering components. For example, Ni-W-P coatings with SiO_2 particles exhibit a hybrid microstructure that combines amorphous and nanocrystalline phases, improving the wear resistance and mechanical stability of the coating [179]. Despite their advantages, achieving consistent grain sizes and uniform distribution of nanocrystals remains a challenge [189]. Controlling deposition parameters such as bath composition, temperature, and pH is critical to ensuring the proper balance between amorphous and nanocrystalline phases. Heat treatments are often used to control the crystallisation of the amorphous phase into nanocrystals. Still, this process can introduce internal stresses, which may cause cracking if not properly managed [181].

Amorphous-nanocrystalline nickel coatings produced via electroless deposition offer a highly versatile solution for applications requiring a balance of hardness, toughness, wear resistance, and corrosion protection. Their hybrid nature, combining the best attributes of both amorphous and nanocrystalline structures, makes them ideal for advanced engineering components and protective coatings. However, the success of these coatings depends on optimising deposition parameters to ensure uniformity and minimise challenges such as nanoparticle agglomeration and internal stresses.

5.3. Amorphous Nickel

Amorphous nickel coatings, produced via electroless deposition, are widely recognised for their superior corrosion resistance, hardness, and wear performance, making them suitable for various industrial applications such as electronics, automotive, aerospace, and chemical processing [7]. These coatings owe their unique properties to their amorphous structure, formed when the deposition process prevents the long-range atomic ordering typically found in crystalline materials [190]. The key factor influencing the formation of amorphous nickel coatings through electroless deposition is the presence of phosphorus or boron in the alloy. The phosphorus content is pivotal in determining the final microstructure of the deposited Ni-P alloy [7]. When phosphorus content exceeds approximately 8%, the deposited alloy adopts an amorphous structure due to the disruption of the nickel crystal lattice. This amorphous structure, characterised by the absence of grain boundaries, contributes to the exceptional corrosion resistance of these coatings, as grain boundaries typically serve as initiation sites for corrosion in crystalline materials [191].

Amorphous Ni-P coatings are typically deposited from an aqueous bath containing nickel salts, sodium hypophosphite as the reducing agent, and stabilisers to control the deposition rate [99]. These coatings are highly uniform and exhibit a dense, smooth surface. The high phosphorus content, often ranging from 8% to 12%, enhances the amorphous nature of the coating and contributes to its mechanical properties [189]. Electroless Ni-B coatings, which rely on boron instead of phosphorus, produce amorphous structures with excellent hardness and wear resistance, particularly when used in high-friction environments. The absence of grain boundaries in amorphous nickel coatings significantly improves their corrosion resistance, making them ideal for use in harsh environments, such as chemical processing and marine applications. The smooth surface of these coatings further enhances their tribological properties, reducing friction and wear during operation. Amorphous Ni-P coatings typically exhibit hardness values around 600 HV (Vickers Hardness) in the as-deposited state, which heat treatment can further enhance [190].

Heat treatment is a common method for improving the mechanical performance of amorphous Ni-P coatings. The amorphous phase begins to crystallise upon heating, forming fine nickel and Ni_3P grains. This transformation significantly increases the coatings'

hardness, with values reaching up to 1100 HV after optimal heat treatment. However, excessive heat treatment can lead to grain coarsening, which reduces hardness and mechanical integrity, underscoring the importance of precise control over the thermal treatment process [190].

The microstructure of electroless Ni-B coatings is significantly influenced by boron content and subsequent heat treatment, which directly impact their crystallinity, grain size, and preferred orientation. As the boron content increases, the XRD patterns (Figure 7A) exhibit peak broadening and intensity reduction, indicating a transition from a crystalline to a more amorphous structure. At lower boron concentrations, the coatings predominantly display an FCC nickel phase with a (200) preferred orientation. However, as boron incorporation increases beyond ~2.5 wt.%, a shift toward the (111) orientation is observed, attributed to boron-induced lattice strain and nanocrystallisation effects.

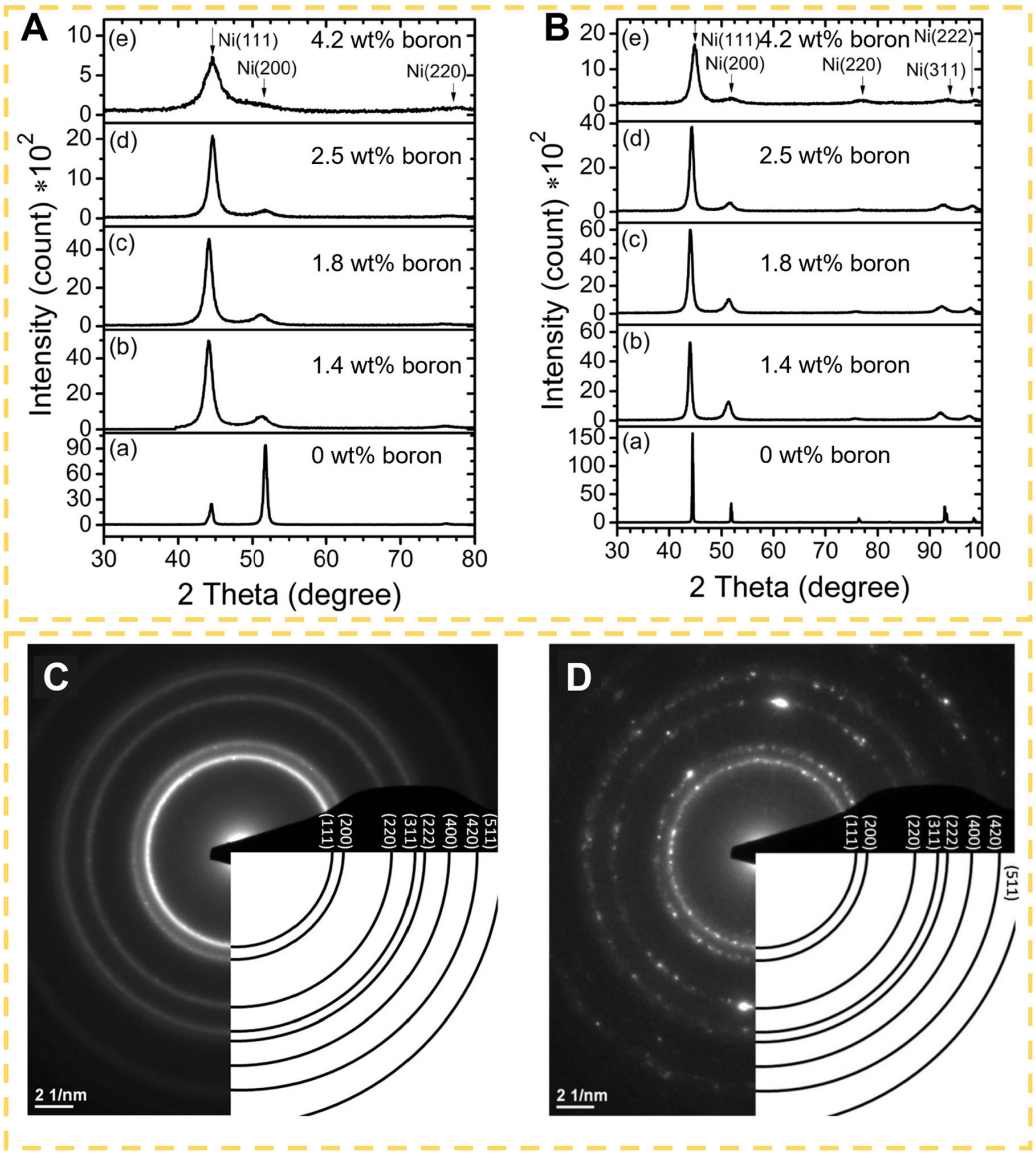


Figure 7. (**A**) XRD patterns of the as-deposited coatings with different volumes of boron. (**B**) XRD patterns of heat-treated coatings with different volumes of boron, (**C**) SAED patterns of the as-deposited Ni-2.5 wt.% B alloy, (**D**) SAED patterns Ni-2.5 wt.% B alloy coating heat-treated at 300 °C for 1 h [192]. Copyright © 2018 Elsevier.

High-resolution TEM analysis (Figure 7C) reveals that Ni-B coatings with moderate boron content (e.g., ~2.5 wt.%) consist of a dual-phase nanostructure, where equiaxed

nanocrystals (~4.3 nm) are embedded within an amorphous matrix. This structural evolution is attributed to boron segregation at grain boundaries, restricting grain growth and promoting the formation of amorphous intergranular regions. Upon annealing at 300 °C, enhanced crystallisation occurs, leading to grain growth in pure nickel coatings, while Ni-B coatings retain their nanocrystalline structure due to the pinning effect of the amorphous phase [193–196]. Further heating to ~400 °C (Figure 7B,D) results in the precipitation of Ni_3B and Ni_2B phases, contributing to increased hardness and wear resistance [192]. The transition from an amorphous-nanocrystalline composite to a more ordered crystalline structure with heat treatment plays a crucial role in determining the mechanical and electrochemical properties of Ni-B coatings.

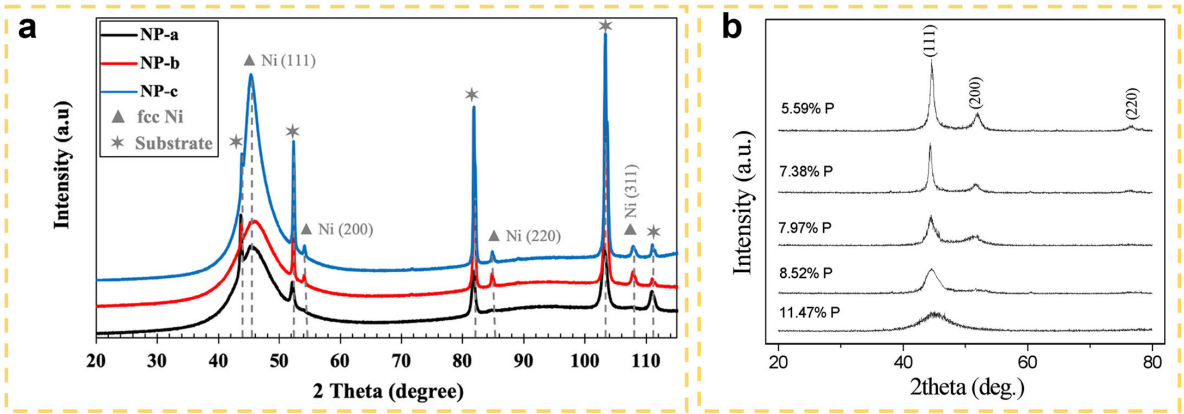
Amorphous nickel coatings also exhibit excellent wear resistance. Their dense, non-porous structure allows them to withstand high levels of friction and mechanical stress, making them suitable for protective coatings in moving parts and machinery [7,190]. Additionally, the coatings show superior performance in aggressive chemical environments due to the absence of grain boundaries, further emphasising their value in high-corrosion applications [191,197]. Despite their advantages, amorphous nickel coatings face some challenges. One major limitation is the brittleness at high phosphorus levels, reducing toughness under certain loading conditions [191]. Researchers have explored overcoming this limitation by introducing alloying elements like tungsten or molybdenum or incorporating nanoparticles to create hybrid amorphous-nanocrystalline structures. These hybrid coatings combine the toughness of crystalline phases with the corrosion resistance of amorphous phases, providing an optimal balance of mechanical properties. Another challenge is controlling deposition parameters to ensure consistent amorphous structures. Bath composition, temperature, and pH must be carefully optimised to achieve the desired amorphous microstructure [197,198]. Any parameter deviation can lead to partial crystallisation, reducing the coating's overall performance.

In conclusion, amorphous nickel coatings produced via electroless deposition offer a versatile and effective solution for industries requiring high-performance protective coatings. Their ability to resist corrosion and wear, combined with the flexibility to tailor their mechanical properties through heat treatment and alloying, makes them ideal for various applications. However, further research is needed to optimise deposition parameters and explore hybrid structures like amorphous-nanocrystalline coatings to exploit their potential fully in even more challenging environments.

6. Advanced Applications of Electroless Nickel Coatings

The influence of reducing agent content on the microstructure and properties of electroless nickel coatings has been explored extensively in the literature, with studies focusing on both corrosion resistance and mechanical performance. Two notable investigations provide insights into these aspects: one emphasises corrosion behaviour and microstructural evolution [199], while the other focuses on hardness and wear resistance [200]. Figure 8 summarises the crystallinity of Ni-P influenced by the content of phosphorus. The first study examines how phosphorus content affects the crystallinity of Ni-P coatings and their localised corrosion resistance. It identifies that low phosphorus (3–5 wt.%) coatings are typically crystalline, with prominent Ni(111) and Ni(200) peaks observed in XRD, corresponding to larger grains (~18 nm) and moderate hardness. Medium phosphorus (6–10 wt.%) coatings exhibit a mixed amorphous-crystalline structure (β and γ phases), often with the finest grains (~4 nm) and the highest hardness (~910 HV). High phosphorus (11–18 wt.%) coatings are mostly amorphous, showing improved corrosion resistance due to the absence of grain boundaries, though with reduced hardness.

The methodologies employed in these studies differ according to their respective objectives. The corrosion-focused study utilises electrochemical techniques such as scanning Kelvin probe force microscopy (SKPFM), electrochemical impedance spectroscopy (EIS), and potentiodynamic polarisation (PDP) to analyse localised charge distribution and corrosion susceptibility. It concludes that high-phosphorus coatings form stable passive layers that hinder corrosion. In contrast, low-phosphorus coatings are prone to microgalvanic corrosion due to the presence of nodular boundaries and structural defects. On the other hand, the study on mechanical performance employs microhardness testing, wear resistance measurements, X-ray diffraction (XRD), and transmission electron microscopy (TEM) to investigate the structural evolution and mechanical properties of Ni-P coatings. The findings suggest that a balance between hardness and wear resistance can be achieved by optimising phosphorus content without needing post-deposition heat treatment.



c

Phosphorus Content (wt.%)	Microstructure and Corrosion Behaviour	Hardness and Wear Resistance
3–5 wt.% (Low P)	Predominantly crystalline (Ni (111), Ni (200) peaks).	Crystalline phase with large grains (~18 nm), moderate hardness.
6–10 wt.% (Medium P)	Mixed amorphous-crystalline structure (β and γ phases coexist).	Nanocrystalline structure with fine grains (~4 nm), highest hardness (910 HV).
11–18 wt.% (High P)	Fully amorphous structure, improved corrosion resistance, absence of grain boundaries.	Fully amorphous phase, reduced hardness and wear resistance.

Figure 8. Comparison of phosphorus content influence on the crystalline structure of electroless Ni-P coatings. (**a**) XRD spectra of three Ni-P coatings [199], Copyright © 2024 Elsevier. (**b**) XRD spectra of five Ni-P coatings [200], and Copyright © 2008 Elsevier. (**c**) A summarised table to compare two methods of Ni-P coatings.

From an application perspective, selecting phosphorus content in Ni-P coatings depends on the desired performance characteristics. The corrosion study suggests that high-phosphorus coatings are best suited for applications exposed to aggressive environments, such as marine or electronic components, where corrosion protection is critical. In contrast, the mechanical study highlights that coatings with moderate phosphorus content are ideal for wear-resistant applications, such as tooling and machinery components, where hardness and durability are paramount. Despite these insights, both studies have limitations. The corrosion study does not provide detailed mechanical performance data, whereas the mechanical study lacks an in-depth assessment of the coatings' corrosion resistance in harsh environments. A more comprehensive study that integrates both aspects would provide a holistic understanding of the material's performance under real-world conditions.

6.1. Corrosion Protection and Wear Resistance

Nickel coatings are widely recognised for their excellent corrosion resistance, which is largely influenced by their microstructure and alloy composition [169,201]. Bonin et al.

explored electroless Ni-B coatings prepared without stabilisers, emphasising their environmentally friendly nature and effective corrosion protection [202]. Studies indicate that electroless Ni-B coatings completely encapsulate the substrate, acting as effective barrier layers [202,203]. These coatings exhibited smooth, featureless morphologies and high boron content, both of which contributed to their barrier function and reduced anodic dissolution. The comparative findings from these studies underline that incorporating ternary elements such as Cu or B and controlling microstructural features like crystallinity, phase distribution, and surface morphology are crucial strategies to improve the corrosion performance of electroless nickel coatings. The observed positive shift in corrosion potential suggests improved resistance, as nickel is more noble than iron. The amorphous nature of the as-deposited Ni-B structure contributes to high corrosion resistance in various corrosive media. Figure 9a demonstrates the Ni coating after 168 h of exposure, the Ni-B coating remains largely intact, with only minimal surface oxidation, while the Ni-B-Pb coating exhibits significant corrosion initiation. At 240 h, the Ni-B coating continues to provide a barrier against environmental degradation, showing only minor rust spots, whereas the Ni-B-Pb coating suffers from extensive corrosion, with visible rust propagation and coating delamination. This highlights that the absence of heavy metal stabilisers in the optimised Ni-B bath results in a denser, more uniform structure that effectively mitigates electrolyte penetration and enhances corrosion resistance [202].

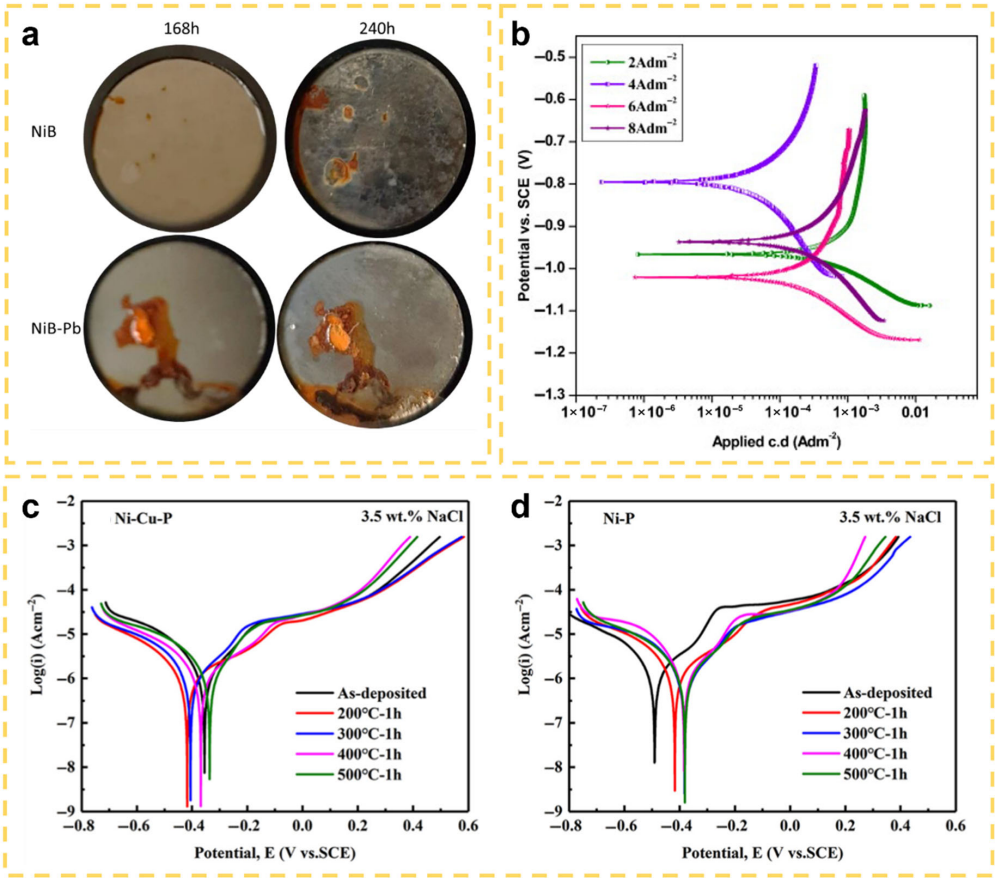


Figure 9. The corrosion resistance of Ni coatings. (**a**) The surface aspect of electroless Ni-B and Ni-B-Pb 15 ± 1 µm coatings after 168 h and 240 h salt spray testing [202]. Copyright © 2018 Elsevier. (**b**) Potentiodynamic polarisation behaviour of Fe-Ni coatings deposited from the optimal bath at different c.d.'s [161]. Copyright © 2012 Elsevier. (**c**,**d**) Electrochemical polarisation plots of as-deposited and annealed alloys in 3.5% NaCl solution [204]: (**c**) Ni-Cu-P and (**d**) Ni-P. Copyright © 2019 Elsevier.

Binary Ni-P alloys, particularly in their amorphous state, exhibit superior corrosion protection due to the absence of grain boundaries that typically act as initiation sites for corrosion [205,206]. As shown by Chen et al. in Figure 9c,d, the corrosion resistance of Ni-P coatings can be further enhanced by alloying with copper to form Ni-Cu-P coatings, which not only improve thermal stability but also result in the formation of passive films with p-n bipolar semiconductor characteristics [204]. This dual-type passivation layer offers improved barrier properties compared to the single p-type behaviour of standard Ni-P coatings, enabling enhanced ion-blocking efficiency and corrosion resistance in chloride environments. In contrast, nanocrystalline Fe-Ni coatings (Figure 9b) studied by Pavithra and Hegde demonstrate high corrosion resistance due to the dense structure and controlled phase composition achieved through optimised electroplating parameters [161].

Electroless Ni-B coatings generally exhibit lower corrosion resistance compared to their electroless Ni-P counterparts [207]. The superior resistance of electroless Ni-P coatings is attributed to a hypophosphite-derived protective layer, which forms due to the oxidation of phosphorus at the surface. In contrast, Ni-B coatings lack the formation of such a stable passivating film. Furthermore, the columnar microstructure of some electroless Ni-B coatings can create pathways for electrolyte penetration, increasing susceptibility to corrosion. Research has shown that modifying the bath formulation can enhance the corrosion resistance of electroless Ni-B coatings. For instance, coatings deposited from baths without stabilisers exhibit smoother surfaces, as the absence of stabilisers prevents the typical cauliflower-like morphology [202]. This reduction in intercolumnar zones results in fewer corrosion initiation sites, improving resistance. Additionally, coatings produced using tin-stabilised baths have demonstrated better corrosion performance due to tin oxides on the surface, further enhancing protection against corrosive attack. Deposition conditions also significantly influence corrosion resistance [208]. Studies have shown that coatings deposited at lower temperatures, such as 60 °C, exhibit finer grain structures, contributing to better corrosion performance [209]. Additionally, nickel-boron coatings have been observed to reduce susceptibility to chloride-induced corrosion by forming a protective surface layer that minimises attack by chloride ions [210].

The corrosion resistance of electroless Ni-B coatings is highly dependent on the surrounding environment. In alkaline conditions, these coatings effectively passivate, exhibiting excellent resistance. However, in neutral and acidic environments, including mild solutions such as sodium sulphate (Na_2SO_4), their passivation behaviour is less pronounced, making them more vulnerable to corrosion. Understanding these factors is crucial for optimising electroless nickel-boron coatings for industrial applications requiring robust corrosion resistance. In addition to corrosion resistance, electroless nickel coatings offer excellent wear resistance and hardness, making them suitable for applications where mechanical parts are subjected to continuous friction and stress. For instance, electroless Ni-B coatings are widely used in automotive, aerospace, and heavy manufacturing industries due to their high hardness, which can reach values comparable to or exceeding that of hard chrome. Electroless nickel-boron coatings have demonstrated hardness values up to $872HV_{100}$, making them ideal for applications such as saw blades, firearms, and valves. The fine-grained structure of electroless Ni-B coatings also contributes to their superior durability and resistance to wear under heavy load conditions [21,50,211,212].

6.2. Electronics and Microelectronics

Electroless nickel coatings are extensively used in the electronics and microelectronics industries due to their excellent solderability, uniform thickness, and oxidation resistance. In printed circuit boards (PCBs), electroless nickel serves as a conductive layer, providing reliable electrical connections and protecting against environmental degradation [213,214].

Nickel-boron coatings are particularly useful in microelectronics, where they are applied to form conductive surfaces and interconnections [34,215]. The uniformity and low resistance of these coatings make them essential for ensuring the long-term performance and reliability of electronic devices. In addition to their use in circuit boards, electroless nickel coatings have applications in manufacturing supercapacitors and catalytic surfaces. Ni-B alloys are known for their catalytic properties, especially in hydrogenation and dehydrogenation reactions, making them valuable in the chemical industry. The coatings' durability and resistance to chemical attack are also crucial for protecting sensitive components in electronic devices and energy storage systems. As energy storage systems become more advanced, the need for materials that can withstand the harsh conditions inside batteries and supercapacitors becomes more critical, and electroless nickel coatings are increasingly used to extend the life of these devices [21,216].

6.3. Nanotechnology and Energy Storage Applications

Electroless nickel deposition has found growing applications in nanotechnology and energy storage systems, particularly in producing nanostructures such as nanowires, nanotubes, and thin films. These nanostructures, including batteries and supercapacitors, are essential for advanced energy conversion and storage applications [8,217]. The ability of electroless nickel to create uniform, thin coatings on intricate nanostructures enhances the performance of electrodes and conductive substrates, making it a valuable tool in these cutting-edge fields. In energy storage devices, the durability of electroless nickel coatings ensures that electrodes remain stable over time, improving the overall efficiency and lifespan of the devices [8,218]. In addition to its mechanical and catalytic benefits, electroless nickel deposition has become more environmentally friendly with recent developments in plating bath formulations. Lead-free and thallium-free electroless nickel baths have been introduced, reducing the environmental impact of the process without sacrificing performance [21]. These environmentally conscious advances, along with incorporating surfactants and composite coatings, have expanded the range of applications for electroless nickel in industries where sustainability is a growing concern [219].

6.4. Wearable Electronic Textile Applications

Recent advances in flexible electronics and smart textiles have created a strong demand for lightweight, conformable, and electrically conductive fibres. EN deposition on synthetic yarns can address these needs by converting conventional polymer-based textiles into functional materials capable of sensing, energy storage, or signal transmission. Due to nickel's high electrical conductivity, corrosion resistance, and decent mechanical properties, Ni-coated synthetic fibres are emerging in applications ranging from wearable biosensors to heated garments.

Recent advances in flexible electronics and smart textiles have created a strong demand for lightweight, conformable, and electrically conductive fibres. EN deposition on synthetic yarns can address these needs by converting conventional polymer-based textiles into functional materials capable of sensing, energy storage, or signal transmission [56,67]. Due to nickel's high electrical conductivity, corrosion resistance, and decent mechanical properties, Ni-coated synthetic fibres are emerging in applications ranging from wearable biosensors to heated garments [220,221]. Figure 10 illustrates several recent advances in nickel-based and nickel-alloy coatings on textile substrates.

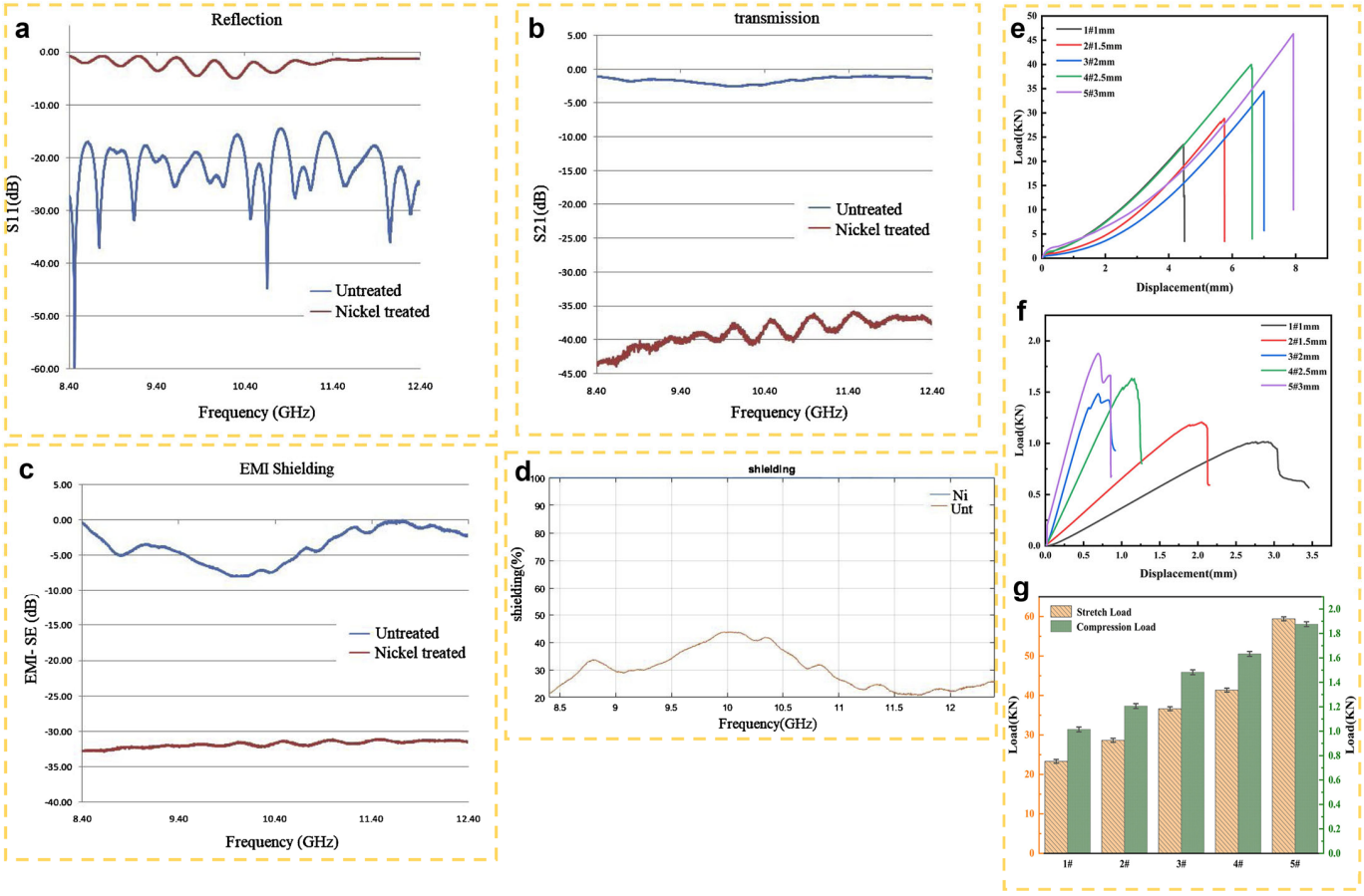


Figure 10. (**a**–**d**) EMI shielding (dB) of untreated and nickel-treated polyester fabric [220], Copyright © 2018 Elsevier. (**e**–**g**) Mechanical properties of GFs/nickel plated fabric/UDCFs/EP composites with different thicknesses: (**e**) Tensile load–displacement graph, (**f**) bending load–displacement graph, and (**g**) maximum load bar graph [222]. Copyright © 2024 Springer Nature.

Figure 10a–d highlight how electroless Ni deposition yields closely packed nickel nanoparticles on polyester fibres, thereby conferring moderate-to-high electrical conductivity and full electromagnetic shielding in the Ku-band frequency range. Several commercial efforts have demonstrated the viability of metallised yarns in real-world applications. For example, Shieldex® produces nickel-coated polyamide yarns used in EMI shielding fabrics and conductive threads for smart textiles. Similarly, companies like TexTrace and Myant Inc. have explored integrating conductive yarns into RFID-tagged clothing and biosignal-monitoring garments, respectively. These developments highlight the growing market for durable, conductive, and washable textiles, reinforcing the industrial relevance of electroless metallization approaches in the wearable electronics sector.

One of the key challenges in wearable textiles is preserving conductivity and mechanical integrity during repeated deformation, washing, and friction. Because electroless nickel coatings produce uniform metal layers with controllable thickness—even on complex or flexible substrates—they are especially attractive for e-textiles. For instance, treating nylon or polyester fibres with acid etching or mussel-inspired PDA layers [70] provides an activated, adhesive surface that can anchor subsequent nickel films. Such coatings often remain stable under bending cycles, highlighting their robustness for use in soft electronics, health monitoring devices, and flexible display interconnects. Furthermore, incorporating nickel-coated fibres into layered composites can improve mechanical strength and wave absorption, a strategy evidenced by recent work on Ni-coated woven fabrics for microwave-absorbing composites [222]. Figure 10e–g demonstrate that Ni-plated woven

fabrics not only achieve strong microwave absorption—due to the formation of uniform, dense Ni layers—but also exhibit enhanced mechanical properties when incorporated into layered, load-bearing composites.

Moreover, the ability to fine-tune coating characteristics (e.g., thickness, nickel-alloy composition) allows for balancing conductivity with flexibility. Low-phosphorus Ni deposits provide higher conductivity, whereas Ni-B coatings impart greater hardness and wear resistance—key considerations for garments subject to abrasion or high-cycle use [21,50]. Composite coatings that embed nanoparticles (e.g., SiO_2) can enhance durability and reduce friction, preserving conductivity over multiple wash cycles. However, integrating these coatings into large-scale textile production requires optimising bath stability and addressing environmental factors—for instance, replacing toxic stabilisers and reducing rinse-water waste. Ongoing work on novel chemistries and eco-friendly plating baths will be crucial for making Ni-coated yarns truly viable in commercial wearables.

Looking ahead, electroless nickel-coated synthetic fibres promise to bridge the gap between traditional textiles and cutting-edge electronics. The uniform coverage, proven corrosion resistance, and controllable microstructure of EN coatings lend themselves to many wearable applications—such as biometric sensing (e.g., pulse or sweat analysis), EM shielding, and flexible energy storage. Yousif et al. show that dual-metal (Ni-Cu) coatings effectively balance high initial conductivity (from copper) and long-term air stability (from nickel), underscoring nickel's valuable role as a robust barrier against corrosion or oxidation [221]. Continued research on catalyst immobilisation, polymer-compatible bath formulations, and multi-scale mechanical characterisation will accelerate the transition from lab demonstrations to everyday smart garments. By tailoring coating chemistry and process parameters, researchers can develop resilient, lightweight, and highly conductive yarns poised to redefine the next generation of wearable technologies.

7. Challenges and Perspectives

Electroless nickel deposition is a versatile and widely utilised process in various industries due to its ability to provide uniform coatings, superior corrosion resistance, and excellent mechanical properties. However, despite its advantages, the process faces several challenges that limit its broader adoption. Moreover, continuous advancements are being made to address these limitations and expand their applicability in emerging technologies. This section will discuss the primary challenges associated with electroless nickel deposition and explore potential future directions for further development.

7.1. Challenges

One of the most significant challenges in electroless nickel deposition is achieving uniform coatings on complex geometries. While the autocatalytic nature of the process allows for uniform deposition, controlling the thickness of the coating across intricate surfaces can be difficult, especially in applications requiring precise tolerances. Industries such as aerospace, automotive, and electronics demand uniformity in coatings to ensure performance and durability, making this a critical challenge to address. Another key issue is bath stability and composition control. Over time, by-products accumulate in the electroless plating bath, decreasing efficiency and instability. The accumulation of these by-products can cause spontaneous metal precipitation or "plate-out", where uncontrolled metal deposition occurs on the walls or equipment inside the plating bath. This phenomenon not only leads to material waste but also shortens bath lifespan and compromises coating uniformity, which diminishes the bath's performance. This necessitates constant monitoring and replenishment of bath components, such as the reducing agent and stabilisers, to maintain the desired chemical balance. Ensuring bath stability over extended periods remains a

significant challenge for researchers and industry professionals. Environmental and safety concerns are also prevalent in electroless nickel deposition. Traditional baths often contain toxic heavy metals such as lead, used as stabilisers. Disposing of these substances is highly regulated, contributing to increased environmental and financial costs. Additionally, using hazardous chemicals poses risks to operators and the surrounding environment. The push for greener and more sustainable processes has led to ongoing research into environmentally friendly alternatives, but finding effective substitutes that maintain the performance of traditional baths remains difficult. Lastly, internal stress and brittleness present a challenge for electroless nickel coatings, particularly in high-phosphorus and nanocrystalline coatings. Internal stresses can accumulate during the deposition process, leading to coating cracking or delamination, compromising its mechanical properties and longevity. This issue is especially problematic in applications requiring high wear resistance and mechanical durability, such as hydraulic systems and heavy machinery.

7.2. Future Directions

In response to these challenges, several avenues of research have emerged that aim to improve the electroless nickel deposition process and expand its range of applications. One important area of research is the development of nanocrystalline and amorphous-nanocrystalline coatings. Compared to traditional crystalline coatings, these coatings offer enhanced mechanical properties, such as increased hardness and wear resistance. Nanocrystalline coatings provide superior performance due to their fine grain size, which serves as a barrier to dislocation motion and enhances strength. Amorphous-nanocrystalline structures balance hardness and toughness, making them suitable for applications requiring both wear resistance and mechanical flexibility. Future research is expected to focus on refining the deposition conditions to achieve uniform nanocrystalline structures and improve control over grain size. Hybrid coatings represent another promising future direction for electroless nickel deposition. Researchers have developed quaternary alloys that exhibit enhanced properties by incorporating elements such as tungsten, molybdenum, or silicon carbide into the nickel matrix. For example, Ni-W-P and Ni-Mo-P coatings have demonstrated improved thermal stability, wear resistance, and corrosion resistance, making them ideal for high-temperature environments and aggressive chemical conditions. Developing such hybrid coatings is likely to play a crucial role in expanding the use of electroless nickel in more demanding applications.

Finally, applications in advanced technologies offer significant opportunities for the future of electroless nickel deposition. As industries such as electronics, renewable energy, and medical devices continue to evolve, the demand for coatings that provide both durability and precision will increase. Electroless nickel coatings are well-suited for these applications due to their uniformity, corrosion resistance, and mechanical robustness. For instance, electroless nickel coatings are used to enhance the performance and longevity of electrodes in energy storage devices such as batteries and supercapacitors. The ability to coat intricate and non-conductive surfaces in flexible electronics opens new possibilities for innovative designs. While electroless nickel deposition faces several challenges, including bath stability, environmental concerns, and internal stress in coatings, ongoing research addresses these issues and unlocks new possibilities for its use. Future research will focus on developing environmentally friendly stabilisers, refining nanocrystalline and hybrid coatings, and exploring new applications in advanced technologies. By overcoming these challenges, electroless nickel deposition will continue to play a pivotal role in various industries, offering a versatile and reliable solution for protective and functional coatings.

To support the continued advancement and scalability of electroless nickel coatings on polymer substrates, several key challenges should be prioritised in future studies:

- Coating adhesion and delamination: Improving the mechanical robustness of metal-polymer interfaces remains the most pressing challenge, especially under bending, washing, or thermal cycling.
- Surface activation standardisation: Developing low-cost, effective, and environmentally friendly activation methods that work across a variety of polymers and geometries is crucial.
- Bath stability and reproducibility: Achieving consistent deposition rates and coating quality across long plating durations or in large-scale processes requires better control over bath ageing and composition.
- Environmental sustainability: Reducing or replacing toxic stabilisers, improving waste treatment, and enabling bath recyclability are essential for greener implementation.
- Scalability and integration: Translating lab-scale processes to roll-to-roll or industrial-scale systems without compromising coating performance remains a significant hurdle.

8. Conclusions

The literature review comprehensively examines the electroless nickel deposition process, delving into its underlying mechanisms, diverse applications, and the ongoing challenges in optimising its use across various industries. Electroless nickel deposition is an autocatalytic chemical process that facilitates the uniform deposition of nickel onto a substrate without needing external electrical current, distinguishing it from traditional electroplating methods. This advantage, coupled with the process's ability to uniformly coat complex geometries, has made it an integral technique in aerospace, electronics, and automotive manufacturing, where precision and durability are critical.

At the core of electroless nickel deposition is the reduction of nickel ions by a chemical reducing agent, such as sodium hypophosphite or sodium borohydride. These reducing agents form Ni-P or Ni-B coatings, each with unique properties. Ni-P coatings are widely recognised for their superior corrosion resistance, making them highly suitable for applications in harsh environments, such as marine or chemical processing industries. On the other hand, Ni-B coatings are favoured for their exceptional hardness and wear resistance, lending themselves to applications where high durability under mechanical stress is required, such as in cutting tools or heavy-duty machinery. The surface preparation of substrates is crucial for the success of the deposition process, particularly on non-conductive surfaces like plastics or ceramics, which often require surface activation through chemical or mechanical means to ensure proper adhesion of the nickel coating.

This review underscores that selecting the optimal method for electroless nickel deposition is highly dependent on the intended application and required coating properties. Solvent-engineered baths (particularly DMSO or ethanol-based systems) are strongly recommended when uniformity and flexibility are priorities, especially for textile or wearable electronics applications. Bio-inspired activation strategies (e.g., polydopamine or tannic acid) provide superior adhesion suitable for applications where mechanical robustness is crucial. To achieve optimal overall coating performance, hybrid amorphous-nanocrystalline microstructures represent the best compromise, delivering both hardness and corrosion resistance without sacrificing flexibility. The recommended strategy for future studies involves careful balancing of these techniques, integrating standardised surface activation, solvent system optimisation, and targeted microstructure tailoring to meet specific industrial requirements. Ultimately, combining these strategies will accelerate the development and widespread industrial implementation of electroless nickel coatings on polymer substrates.

The microstructure of the electroless nickel coatings plays a vital role in determining their mechanical properties. Crystalline coatings are known for their high strength and

thermal stability, making them ideal for applications requiring resistance to wear and mechanical stresses. In contrast, amorphous and nanocrystalline structures offer enhanced corrosion resistance due to their lack of grain boundaries, which typically act as pathways for corrosive agents. These structures also exhibit improved wear resistance, making them suitable for applications in highly abrasive environments. A key area of ongoing research is the development of amorphous-nanocrystalline coatings, which combine the hardness of nanocrystalline coatings with the toughness of amorphous ones, creating a balance beneficial for applications requiring both strength and flexibility.

In terms of applications, electroless nickel coatings are employed in various sectors. These coatings are particularly effective in providing corrosion protection for components exposed to aggressive environments, such as in marine, oil and gas, or chemical processing industries. Additionally, they enhance the durability of mechanical components, such as gears, pumps, and valves, where wear resistance and long-term reliability are essential. In electronics, electroless nickel is applied to manufacture PCBs and semiconductor devices, where the coatings provide conductive layers and protect against oxidation and environmental degradation. The process has also seen growing applications in energy storage systems, such as batteries and supercapacitors, where electroless nickel coatings enhance the performance and longevity of electrodes and conductive substrates.

Despite these numerous applications, several challenges persist in optimising electroless nickel deposition. Bath stability remains a concern, as the chemical composition of the deposition bath can degrade over time, leading to inconsistencies in the deposition process. Additionally, environmental concerns surrounding using hazardous chemicals in traditional electroless nickel baths, such as lead-based stabilisers, have prompted the search for more sustainable alternatives. The development of lead-free and thallium-free formulations is a growing area of research aimed at reducing the environmental footprint of the process without sacrificing performance. Furthermore, internal stresses that develop during the deposition process, particularly in nanocrystalline coatings, can lead to cracking and reduced durability, necessitating post-deposition treatments like annealing to relieve these stresses.

Looking to the future, research is focused on several key areas. One promising direction is the advancement of nanostructured coatings, which offer improved mechanical properties and corrosion resistance. These coatings, which leverage nanocrystalline or hybrid structures, are being developed for high-performance applications such as flexible electronics and wearable technologies. Additionally, there is growing interest in expanding the use of electroless nickel deposition in energy storage and conversion technologies, where the process can enhance the durability and efficiency of electrodes in batteries and fuel cells. Finally, the continued development of environmentally friendly formulations will be critical for ensuring the long-term sustainability of the process, aligning it with modern environmental regulations and industrial demands.

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