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Development of Multifunctional Hierarchically Restructured Antibacterial Neural Interfacing Electrodes via Plasma-Enhanced Atomic Layer Deposition of Zinc Oxide Thin Films

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ABSTRACT

Next-generation implantable neural-interfacing devices are increasingly constrained by the coupled demands of electrode miniaturization, electrochemical performance, and implantation infection risk. Femtosecond laser-based hierarchical surface restructuring (HSR) technology has emerged as a scalable, manufacturing-compatible platform for overcoming electrochemical performance limitations of conventional electrodes and (micro)electrode arrays by substantially increasing the electrochemically active surface area of electrode contacts. However, imparting antibacterial functionality to HSR electrodes without compromising electrochemical performance remains a critical challenge. We report a surface engineering strategy that integrates the HSR technology with plasma-enhanced atomic layer deposition (PEALD) to controllably introduce antibacterial functionality. ZnO-based ultrathin films were deposited using either oxygen PEALD (O-PEALD), yielding stoichiometric ZnO, or oxygen-hydrogen PEALD (O-H-PEALD), producing a ZnO-Zn nanocomposite containing metallic Zn domains. While O-PEALD films impart antibacterial activity under dark, aerobic conditions, they introduce a resistive barrier that degrades electrochemical performance. In contrast, O-H-PEALD films preserve and enhance electrochemical performance, while maintaining robust antibacterial efficacy against both *Escherichia coli* and *Staphylococcus aureus*. Collectively, this work establishes a scalable and commercially viable strategy for decoupling and optimizing electrochemical performance and antibacterial functionality in implantable neural interfacing electrodes, providing an antibiotic-free pathway toward infection-resistant, high-performance neural interfaces suitable for long-term clinical deployment.

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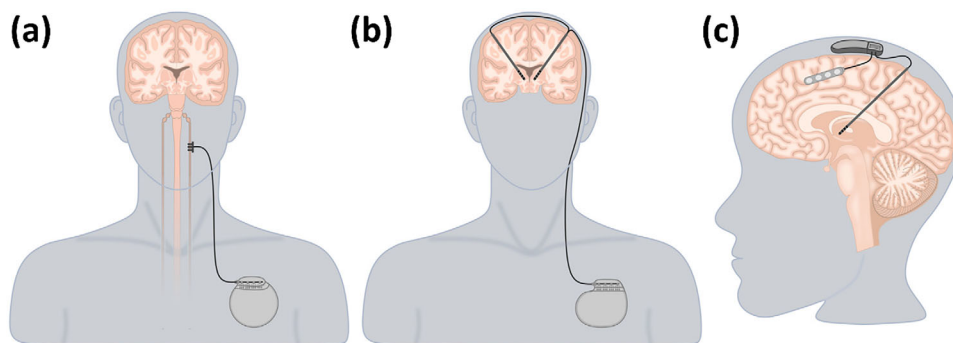


FIGURE 1 | Schematic overview of major neurostimulation therapies used in the treatment of drug-resistant epilepsy, illustrating representative implantation architectures and electrode configurations. (a) Vagus nerve stimulation (VNS) system comprises an electrode with three helical contacts connected to an implantable pulse generator. (b) Deep brain stimulation (DBS) system utilizing depth electrodes connected to an implantable pulse generator. (c) Responsive neurostimulation (RNS) system incorporating depth electrodes and cortical strip electrodes connected to a cranially implanted neurostimulator. Reprinted with permission [6]. Copyright 2024, Elsevier.

1 | Introduction

Neural interfacing devices [1]—such as neurostimulation and cardiac rhythm management devices—function by inhibiting, stimulating, modifying, or otherwise altering neural tissue through the targeted delivery of artificial electrical signals to specific neurological sites within the body [2]. Over the past several decades, millions of patients worldwide have relied on implantable neural interfacing devices to treat a wide range of cardiac and neurological disorders. In these devices, electrical signals are transmitted from a neurostimulator or implantable pulse generator (IPG) to an implanted electrode or electrode array [3–5], where they modulate neural activity at the electrode–tissue interface. Figure 1 provides a schematic overview of representative neurostimulation therapies and implantation architectures, including vagus nerve stimulation (VNS), deep brain stimulation (DBS), and responsive neurostimulation systems (RNS) [6].

To meet the biological and functional demands of their intended applications, electrodes and electrode arrays are engineered with carefully selected sizes, geometries, material compositions, surface characteristics, as well as electrical, electrochemical, and mechanical properties [7, 8]. These stringent requirements have driven continuous innovation in neural interfacing technologies, with a pronounced trend toward electrode miniaturization to improve specificity and spatial selectivity, enable higher electrode channel densities, and enhance patient comfort and compatibility with daily activities [9–14]. Figure 2 illustrates a clinically deployed example, highlighting the implantation architecture of a responsive neurostimulation device and the role of platinum–10% iridium (Pt–10Ir) cortical and depth electrode contacts interfacing directly with neural tissue during chronic neuromodulation.

However, electrode miniaturization introduces fundamental challenges at the electrode–electrolyte interface. As electrode dimensions decrease, maintaining sufficient charge storage capacity, charge injection capability, and low interfacial impedance becomes increasingly difficult, placing greater demands on electrode materials and surface engineering

strategies [16, 17]. Consequently, the performance, stability, and safety of neural interfacing devices are strongly governed by the choice of electrode material and the ability to engineer its surface to maximize electrochemical efficiency without compromising long-term reliability.

Among the materials used in implantable neural interfacing electrodes, platinum–iridium alloys—particularly Pt–10Ir (Pt with 10% Ir by weight)—have emerged as a gold standard due to their electrochemical stability, corrosion resistance, mechanical robustness, and long clinical history. Nevertheless, conventional smooth as-fabricated Pt–10Ir electrodes face inherent limitations in electrochemical performance as device geometries continue to shrink.

Pt–10Ir electrode surfaces can be hierarchically restructured using femtosecond laser processing [17], dramatically increasing the electrochemically active surface area (ESA) [16, 17]. This laser-based hierarchical surface restructuring (HSR) approach substantially enhances electrochemical performance by increasing charge storage and charge injection capacity, improving specific capacitance, and reducing interfacial impedance [9]. Importantly, HSR enables these gains while allowing a reduction in the geometric surface area (GSA) of the electrodes [9, 17], supporting continued device miniaturization without sacrificing electrochemical functionality.

The use of implantable electrodes or leadless pacemakers typically necessitates surgical implantation. Unfortunately, surgical site infections (SSIs) [9, 18, 19] remain a significant clinical concern, occurring in approximately 2–5% of patients undergoing surgical procedures, with an estimated annual incidence of 0.16–0.3 million cases in the United States [2]. However, these figures may underestimate the true prevalence of infection, as post-discharge surveillance remains challenging. In addition, implantable devices carry an ongoing risk of infection at the implantation site, which may present either as surgical site infections within the first year following implantation or as late-onset complications such as lead-associated endocarditis [5, 20–25].

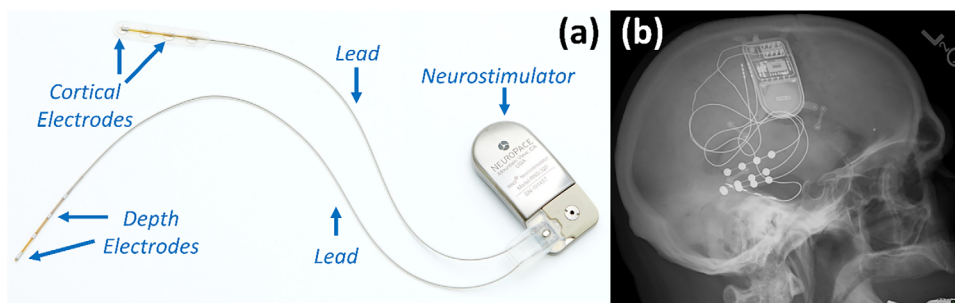


FIGURE 2 | (a) Representative example of a responsive neurostimulation device (RNS System, NeuroPace, Inc.; photo used with permission and courtesy of NeuroPace, Inc.); The neurostimulator is implanted within the skull, replacing a similarly shaped section of bone [6]. The cortical strip or depth electrodes (Pt-10Ir alloy) are implanted in or on the epileptic seizure focus [6]. (b) A skull X-ray image [15] from a patient implanted with the NeuroPace responsive neurostimulation device. The cortical and depth Pt-10Ir electrode contacts appear as bright features. The patient presented with temporal lobe neocortical onset seizures. Reprinted with permission [15]. Copyright 2024, Elsevier.

As global implantation rates continue to rise, the population of patients living with permanent implantable devices is also increasing, raising concerns about a corresponding growth in infection burden. In the context of long-term implantable neurostimulation and cardiac rhythm management devices, bacterial contamination of implanted components remains a significant clinical concern and can progress to severe infections and, in some cases, fatal outcomes [26]. However, the true incidence of surgical site infections (SSIs) remains difficult to quantify due to the limited availability of large-scale, long-term epidemiological studies. Most existing reports are derived from relatively small patient cohorts and are primarily focused on identifying specific risk factors, resulting in inconsistent findings across the literature [2, 20, 26–32]. Collectively, these limitations underscore the need for more comprehensive and systematic investigation of infection risk associated with implantable neurostimulation and cardiac rhythm management devices. Figure 3 provides a broad contextual overview of implantable cardiovascular devices that are susceptible to bacterial colonization and biofilm-related infection. In addition to highlighting representative classes of implantable cardiovascular devices, including pacemakers and defibrillators (Figure 3a), the figure illustrates the implantation architectures and electrode–tissue interfaces of both conventional transvenous pacemakers (Figure 3b) and leadless intracardiac pacing systems (Figure 3c), underscoring the shared materials and surface-exposure challenges across neural and cardiac rhythm management technologies.

In the context of long-term implantable devices [31, 32], bacterial contamination of implanted leads and electrode surfaces is well documented and can progress to severe device-related infections, and in some cases fatal complications [26] if not addressed promptly. Additionally, for implantable neuromodulation devices (e.g., deep brain and spinal cord stimulators), post-operative infection remains a major clinical concern, particularly because these implants remain in the body long term and access to the device site is limited over extended periods. For example, infection rates for spinal cord stimulation (SCS) implants have been reported at approximately 3.1% within 12 months post-implantation [37]. Similarly, reported infection rates for deep brain stimulation (DBS) systems vary widely, ranging from approximately 1.5% to as high as 22.2%, depending on the clinical

center, duration of follow-up, and patient-specific risk factors [38].

These infections can have serious clinical consequences. In DBS, hardware-related infections may necessitate complete device explantation, surgical revision, and prolonged antibiotic therapy, and are associated with increased patient morbidity [39]. More broadly, the management of neuromodulation-associated infections is often complex and resource intensive. While complete device removal remains the most definitive treatment, device salvage strategies—such as partial removal or device retention—have been attempted in select cases, although with variable success and often requiring prolonged follow-up [40].

Overall, outcomes for patients undergoing revision surgery are significantly worse than for those who do not require secondary intervention, including an increase in 10-year mortality rates from approximately 29% to as high as 45% [41]. These infections are often difficult to treat due to the persistence of bacteria on implanted surfaces and their potential resistance to antibiotic therapy [42, 43]. Consequently, the development of new strategies to prevent bacterial infection and inhibit biofilm formation is essential.

One promising approach involves imparting antibacterial functionality directly to implantable electrode surfaces through the use of antibacterial coatings [44–47]. However, most antibacterial materials are electrically insulating, and conventional coating methods can obscure the electrochemically active surface area gained through HSR, thereby potentially diminishing electrochemical efficiency [46, 48, 49]. It is therefore critical that antibacterial coatings are both thin and conformal in order to minimize adverse impacts on electrode electrochemical performance.

Ultrathin nano-coatings enable preservation of the underlying hierarchical nanostructure, thereby maintaining key electrochemical properties [50]. Conformality is essential to ensure complete coverage of the electrode surface by the antimicrobial coating, while precise control over coating thickness is critical to inhibit biofilm formation during the healing period and to enable subsequent dissolution, thereby minimizing long-term exposure to antimicrobial ions and potential health impacts.

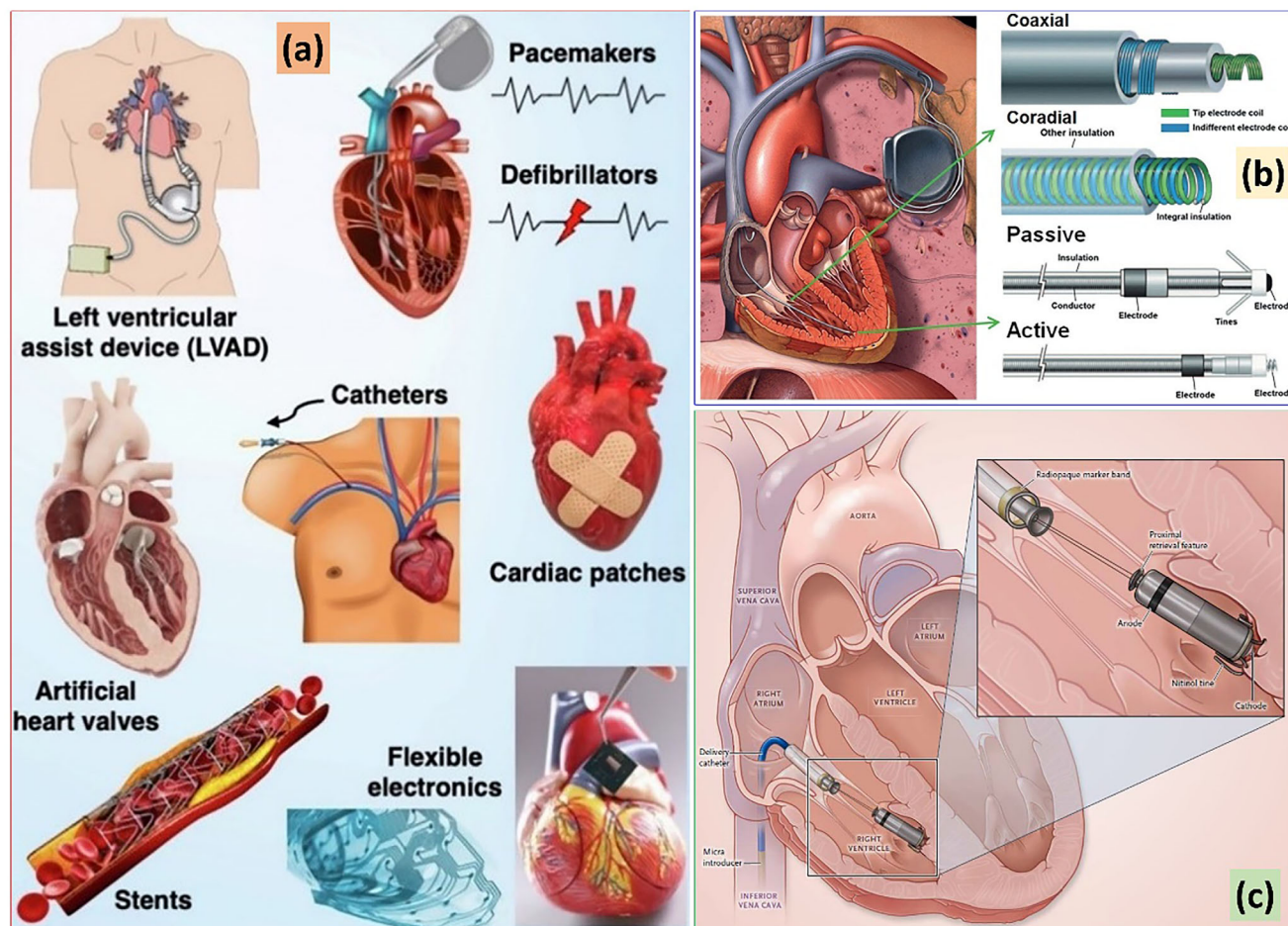


FIGURE 3 | (a) Examples of implantable cardiovascular devices prone to bacterial- and biofilm-related infections, including stents, pacemakers, defibrillators, heart valves, cardiac patches, flexible electronics [33], and leadless pacemakers [22, 23, 34, 35] (not shown in Figure 3a). Reprinted with permission [21]. Copyright 2024, Elsevier. “Antimicrobial surfaces for implantable cardiovascular devices,” p. 100406, 2022 Elsevier, with permission. (b) Schematic overview of a cardiac pacemaker system, consisting of a hermetically sealed implantable pulse generator connected to myocardial tissue via an intracardiac pacing lead. Electrical coupling to the myocardium is achieved through either an actively fixed penetrating platinum–10% iridium (Pt–10Ir) helix electrode (active fixation) or a passively fixed configuration employing tines that anchor within the myocardial trabeculations, with the Pt–10Ir tip electrode positioned adjacent to the tines (passive fixation). Reprinted with permission [36]. Copyright 2024, Elsevier. (c) Schematic overview of a leadless intracardiac pacing system (Micra Transcatheter Pacing System) positioned within the right ventricle. The device integrates the implantable pulse generator and pacing electrodes within a single, self-contained titanium capsule. Electrical stimulation is delivered via a circumferential anode pacing ring located along the proximal region of the device can, enabling direct myocardial pacing without the use of transvenous leads. Reprinted with permission [34]. Copyright 2024, Elsevier.

Our previous work [9] demonstrated that conformal, ultrathin copper oxide (CuO) antibacterial coatings can be deposited using atomic layer deposition (ALD) on hierarchically restructured Pt–10Ir electrodes. The hierarchical nature of laser-restructured Pt–10Ir electrodes, spanning micro- to nanoscale surface features, presents both an opportunity and a challenge for surface functionalization. Figure 4 illustrates the multi-scale surface architecture generated via femtosecond laser hierarchical surface restructuring (HSR), along with a schematic representation of how ultrathin atomic layer deposition (ALD) coatings can conformally envelop these complex features without obscuring the underlying electrochemically active surface. This result highlights the potential of ALD [40, 51–53] as a versatile and commercially viable technique for applying ultrathin, conformal antimicrobial nano-coatings onto complex, hierarchically restructured electrode surfaces used in neurostimulation and

cardiac rhythm management devices, while preserving their electrochemical performance.

It is therefore important to evaluate both the antibacterial efficacy and biocompatible safety of ALD-compatible antibacterial materials. Among these, zinc oxide (ZnO)—an n-type semiconductor—has been extensively investigated for antibacterial and biomedical device applications due to its favorable physicochemical properties [54]. These attributes make ZnO highly versatile for applications spanning medicine, environmental remediation, and electronics. In addition, ZnO is inexpensive, classified as generally recognized as safe (GRAS) by the U.S. Food and Drug Administration [55], biocompatible, and exhibits strong photocatalytic antibacterial activity [56]. ZnO also exhibits excellent chemical stability, strong mechanical durability, and inherent surface hydrophobicity [54, 57–59]. Importantly, ZnO

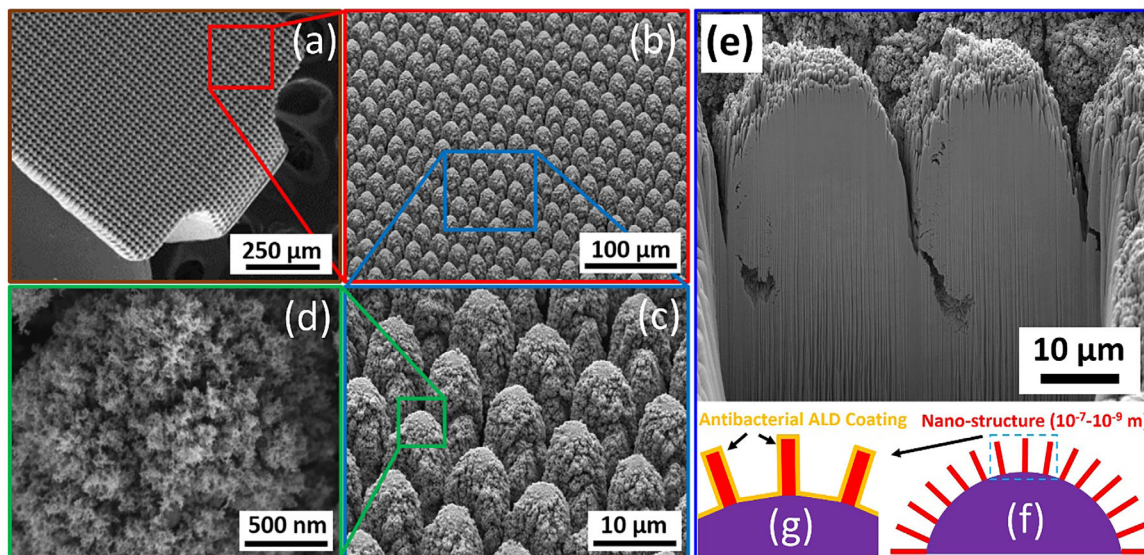


FIGURE 4 | (a–d) SEM micrographs of the hierarchical surface structure induced on the surface of a Pt–10Ir alloy electrode used for a paddle-lead spinal cord stimulation electrode array [9, 17]. (e) Focused ion beam (FIB) cross section of the hierarchically restructured Pt10Ir alloy electrode shown in (a) [17] illustrating the coarse-scale structure of peaks and valleys and the finer nano-structure subset on top of the coarse structures; (f) schematic of a hierarchical surface structure showing the nano-structures (red) on top of the microstructures (purple); and, (g) schematic of an ultra-thin ALD coating forming a conformal antibacterial layer (orange) [9].

has been shown to be effective in preventing or eliminating bacterial growth [58–60], however, its inherently poor electrical conductivity remains a significant limitation for electrochemical applications [61, 62] such as neural interfaces.

The antibacterial efficacy of ZnO, in both nanoparticle form and when deposited using various coating techniques [63], has been extensively studied. Several antibacterial mechanisms have been identified, including the induction of oxidative stress via the generation of reactive oxygen species (ROS) under UV illumination [64], the release of Zn^{2+} ions through dissolution, and the internalization of ZnO nanoparticles by bacterial cells, all of which contribute to cell mortality [65]. Among these mechanisms, ROS generation is generally considered the predominant contributor to ZnO's bactericidal activity, primarily driven by oxidative stress and often accompanied by the release of Zn^{2+} ions [60, 66, 67].

However, because implantable devices operate within the body without exposure to light, it is critical to understand ZnO's antibacterial activity under dark conditions [65]. Furthermore, the release of zinc ions (Zn^{2+}) can damage bacterial cell membranes and potentially interfere with metabolic processes, offering another mechanism for its antibacterial action [68].

Previous studies investigating ZnO's antibacterial properties have predominantly focused on ZnO in nanoparticle (NP) form [69–72]. ZnO nanoparticles have been shown to be biocompatible with human cells, making them attractive antibacterial agents capable of effectively targeting microorganisms while maintaining high compatibility with host tissue [58]. ZnO nanoparticles exhibit strong antibacterial activity, largely attributed to their high surface-area-to-volume ratio, which enhances surface reactivity and interaction with bacterial cells [58].

Therefore, the application of ZnO thin films onto implantable hierarchically restructured (HSR) Pt–10Ir electrodes, as demonstrated in this study, may provide an effective surface-based barrier against bacterial colonization under dark conditions and could be broadly extended to both electrode contacts and implantable leads.

1.1 | Objectives

The objective of this study is to establish a scalable and manufacturing-compatible surface engineering strategy that decouples and independently optimizes electrochemical performance and antibacterial functionality in implantable platinum–iridium (Pt–10Ir) neural interfacing electrodes. Specifically, this work seeks to integrate femtosecond laser-based hierarchical surface restructuring (HSR) technology, which enhances electrochemical efficiency through increased electrochemically active surface area, with plasma-enhanced atomic layer deposition (PEALD) as a controllable route for introducing antibacterial zinc oxide (ZnO) thin films without compromising electrode geometry, mechanical integrity, or manufacturability.

A central objective is to elucidate how ZnO film chemistry and phase structure, controlled through oxygen plasma (O-PEALD) and oxygen–hydrogen plasma (O–H-PEALD) processes, govern the coupled electrochemical and antibacterial behavior of HSR Pt–10Ir electrodes. By directly comparing stoichiometric ZnO films (via O-PEALD) with ZnO–Zn nanocomposite (via O–H-PEALD) coatings, this study aims to identify the mechanisms by which antibacterial efficacy can be achieved under dark, aerobic conditions while preserving—or enhancing—charge storage capacity, specific capacitance, and interfacial impedance characteristics.

Finally, this work aims to define a broadly applicable materials integration framework for incorporating functional thin-film overlays onto hierarchically restructured neural interfacing electrodes. By demonstrating antibacterial activity driven by controlled Zn^{2+} ion release and evaluating electrochemical stability under accelerated testing conditions, this study establishes foundational design principles for infection-resistant, antibiotic-free implantable neural interfacing electrodes, providing a basis for future investigations under physiologically relevant long-term stimulation conditions.

2 | Materials and Methods

2.1 | Femtosecond Laser Hierarchical Surface Restructuring

Pt-10Ir electrodes (0.3 mm thick) were laser-cut and hierarchically surface restructured using a femtosecond laser system (FemtoChisel, Tescan, Storrs, CT) under ambient conditions [73, 74]. The laser system was equipped with a Monaco 1035 femtosecond laser source (Coherent, Santa Clara, CA, USA), which produces 257 fs pulses at a central wavelength of 1035 nm. Beam deflection and targeting were controlled via an IntelliSCAN galvo scan head (SCANLAB, Puchheim, Germany), ensuring precise patterning of the electrode surface for hierarchical surface restructuring.

Electrodes were securely mounted on a porous ceramic vacuum chuck equipped with 3D stage controls (Zaber, Vancouver, BC, Canada) to ensure precise positioning. To maintain accurate focus, the electrodes were leveled with the focal plane of the scan head using a Keyence Confocal Displacement Sensor (Keyence, Osaka, Japan). Targeting and alignment of the laser pattern were verified using a digital microscope (IDS, Massachusetts, USA).

The electrodes included 1 cm squares for microscopy and spectroscopy as well as 6 mm and 10 mm circular discs for electrochemical, ion release and antibacterial studies, respectively. Electrodes were fabricated using a laser fluence of 3.6 J/cm^2 since fluence is a key parameter that directly influences surface morphology and electrochemical properties, and is defined as the energy delivered per unit area [16].

2.2 | Atomic Layer Deposition (ALD) of ZnO Thin Films

Atomic layer deposition (ALD) is a self-limiting, sequential chemical vapor deposition (CVD) process [75] in which the substrate is exposed to alternating precursor and co-reactant pulses, with intermediate purging steps using argon carrier gas. The self-limiting surface reactions inherent to ALD enable the deposition of films that are highly conformal, uniform, and precisely thickness-controlled, even on substrates with complex micro- and nano-morphologies.

Numerous studies have reported the deposition of ZnO thin films via thermal ALD using ozone as the oxidizing co-reactant across a wide range of substrate types [76]. ZnO_x thin films were synthesized using two distinct deposition approaches in a

Veeco Fiji G2 plasma-enhanced atomic layer deposition (PEALD) system (Veeco-CNT, Waltham, MA, USA), operated under continuous argon flow. Diethyl zinc (DEZ) (Strem Chemicals, Inc., Newburyport, MA, USA) was used as the zinc precursor, while oxygen plasma served as the oxidizing reactant. Argon was employed as both the carrier and purge gas, with flow rates of 30 and 100 sccm, respectively.

In the first deposition approach, each ALD cycle consisted of a 1 s DEZ pulse carried by argon, followed by a 10 s argon purge to remove residual precursor and reaction by-products. In the subsequent reactant step, O_2 was introduced at a flow rate of 50 sccm for 10 s through an ignited plasma source operating at 300 W, enabling complete oxidation of the surface-bound DEZ species. The reactor chamber and precursor delivery lines were maintained at 150°C to ensure adequate precursor vaporization and stable film growth. All depositions were performed under high-vacuum conditions, with consistent argon purging between precursor and reactant exposures to preserve the self-limiting surface reactions characteristic of oxygen-plasma-enhanced ALD (O-PEALD). This O-PEALD process yields stoichiometric ZnO films, as demonstrated in the Results section.

In the second deposition approach, an additional hydrogen plasma step was introduced following the previously described PEALD sequence (1 s DEZ pulse followed by 10 s oxygen plasma exposure) to investigate the effects of combined oxygen and hydrogen plasma treatments on the structural, compositional, and surface properties of the resulting ZnO_x films. Following each PEALD cycle, H_2 gas was introduced at a flow rate of 50 sccm for 10 s through an ignited plasma source operating at 100 W, resulting in partial reduction of the ZnO layer and the formation of metallic Zn, as demonstrated in the Results section. This process is referred to as oxygen-hydrogen-plasma-enhanced ALD (O-H-PEALD).

Four substrate types were used for ALD deposition: (i) single-crystal silicon (Si) wafers, (ii) mirror-finish 304 stainless steel substrates, (iii) flat, un-restructured Pt-10Ir electrodes, and (iv) hierarchically surface-restructured (HSR) Pt-10Ir electrodes. To distinguish coated from uncoated regions for subsequent characterization, Kapton tape was applied as a masking layer prior to deposition—following a procedure similar to that used in our previous study [9]—such that ZnO_x coatings were deposited only on the exposed areas.

2.3 | Spectroscopic Ellipsometry and Atomic Force Microscopy

The thickness and optical properties of the ALD thin films were characterized using spectroscopic ellipsometry (M-2000, J.A. Woollam, Lincoln, NE, USA). Ellipsometry data acquired from ALD-coated silicon (Si) substrates were analyzed using CompleteEASE software. Surface morphology and film thickness were further examined on masked Si wafers using an atomic force microscope (AFM) (Park NX10, Park Systems, Santa Clara, CA, USA) operated in non-contact (tapping) mode. Measurements were performed using a pre-mounted Si cantilever tip (OMCL-AC160TS, Santa Clara, CA, USA).

2.4 | Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), and Focused Ion Beam (FIB) Milling

Surface morphology was examined using a field-emission scanning electron microscope (SEM) (Hitachi S-4800, Hitachi, Tokyo, Japan). Elemental composition and spatial distribution were analyzed by energy-dispersive X-ray spectroscopy (EDS) using a Thermo Fisher Scientific Apreo S SEM system (Brno, Czech Republic) equipped with a thermally assisted field emitter source and an Oxford Instruments X-MAX 50 EDS detector (High Wycombe, United Kingdom). Data acquisition and analysis were performed using AZtec software (v3.3), enabling both elemental mapping and selected-area compositional analysis.

EDS spectra were acquired at an accelerating voltage of 10 kV, which exceeds the excitation energies of the relevant characteristic X-ray lines: 2.05 keV for the Pt $M\alpha$ line, 2.15 keV for the Ir $M\alpha$ line, 1.01 keV for the Zn $L\alpha$ line, 0.53 keV for the O $K\alpha$ line, and 1.74 keV for the Si $K\alpha$ line. This operating voltage ensured efficient excitation and reliable elemental analysis from the near-surface region. A detection threshold of 0.5 at.% was selected for the elemental maps to suppress background noise and ensure that only meaningful compositional variations were represented.

Kapton tape was applied to mask the samples and define a sharp boundary between ZnO-coated and uncoated regions. To examine ZnO infiltration into the nanostructured features of HSR Pt–10Ir electrodes, cross-sections were milled at the coated–uncoated boundary using a Tescan S8252X plasma focused ion beam (FIB) system (Brno-Kohoutovice, Czech Republic) equipped with a Xe plasma source. Xenon ions were accelerated at 30 kV during ion milling, and the resulting cross-sections were both milled and imaged within the same dual-beam system.

Elemental compositional maps of the ZnO-coated and uncoated regions across the masked boundary were acquired on silicon substrates, flat/unrestructured Pt–10Ir electrodes, and hierarchically surface-restructured (HSR) Pt–10Ir electrodes. In addition, area-based compositional analyses were performed on electrodes mounted on a tilted stage to assess coating penetration depth. These measurements enabled evaluation of the extent to which the ZnO coatings infiltrated the hierarchical structure of the HSR electrodes.

2.5 | Grazing-Incidence X-Ray Diffraction (GIXRD), X-Ray Photoelectron Spectroscopy (XPS), and Raman Analysis

Grazing-incidence X-ray diffraction (GIXRD) measurements were performed using a Rigaku SmartLab X-ray diffractometer (Rigaku, The Woodlands, TX, USA) operated at a fixed incidence angle of 0.5°. The system was equipped with a Cu $K\alpha$ X-ray source powered by a 2.2 kW generator, which was used for all diffraction analyses. Parallel-beam optics were employed for GIXRD measurements, selected from the available Bragg–Brentano and Parallel-Beam configurations. Diffraction scans were collected over a 2θ range of 30°–70°.

X-ray photoelectron spectroscopy (XPS) analysis was performed using a Thermo Scientific K-Alpha⁺ system (Waltham, MA, USA) equipped with a monochromatic Al $K\alpha$ X-ray source (photon energy = 1486.2 eV). The XPS spot size was adjustable between 30 and 400 μm ; all measurements were performed using the maximum spot size of 400 μm . Binding energy calibration was carried out using the C–C component of the C 1s peak at 284.8 eV. Zn 2p spectra were acquired over the binding energy range of 1020–1050 eV. XPS spectral analysis was performed using CasaXPS software, with Shirley-type background subtraction applied.

Lastly, Raman spectra were acquired using a WITec alpha300R system (Oxford Instruments, Concord, MA, USA) equipped with a 532 nm excitation laser operating at 29 mW power, with an integration time of 50 s.

2.6 | Zn²⁺ Ion Release Analysis

Zn²⁺ ion release from the electrodes under static conditions was quantified using inductively coupled plasma mass spectrometry (ICP-MS), following a previously reported methodology [77]. Each electrode was immersed in 10 mL of sterile distilled water and incubated at 37°C with continuous shaking at 225 rpm for 120 min. At predetermined time intervals, 0.5 mL aliquots were collected, diluted with 5 mL of 1% nitric acid, and analyzed for metal ion concentration using an Agilent 7900 ICP-MS system. All measurements were performed using 8 replicates to ensure statistical robustness, and the results are reported as mean values $\pm$ standard deviation.

2.7 | Antibacterial Measurements

Active antimicrobial species (e.g., Zn²⁺ ions) released from the coated electrode surfaces diffuse into the bacteria-laden agar medium. When the local concentration of these released species exceeds a critical threshold, bacterial proliferation is inhibited in the surrounding region, resulting in the formation of a zone of inhibition (ZOI). The ZOI appears as a transparent region surrounding the sample on an otherwise opaque agar surface densely populated with bacterial colonies.

The antibacterial performance of the electrode surfaces was examined using a modified Kirby–Bauer ZOI assay [9, 78]. In this adapted procedure, electrodes were aseptically positioned with the coated surface facing downward on solidified agar (VWR Life Science, Agarose RA, 7.5 g L^{−1}) containing 14 g L^{−1} Luria Broth (LB, RPI L24080) in petri dishes. In this adapted procedure, electrodes were aseptically placed with the coated surface facing downward onto solidified agar (VWR Life Science, Agarose RA, 7.5 g L^{−1}) supplemented with 14 g L^{−1} Luria Broth (LB; RPI, L24080) in Petri dishes.

Escherichia coli (*E. coli*) K12 (Gram-negative) and *Staphylococcus aureus* (*S. aureus*) ATCC 27660 (Gram-positive) were used as representative bacterial strains to assess the bactericidal efficacy of the PEALD ZnO coated electrodes. Electrodes were first placed on the surface of the agar plates and incubated for 2 h at room temperature to promote electrode–agar contact and

facilitate diffusion of released Zn^{2+} ions into the agar surrounding the electrodes. Subsequently, log-phase bacterial suspensions ($0.5\text{--}2 \times 10^6$ CFU per plate) were uniformly spread across the agar and around the electrodes, allowed to dry for 10 min, and then incubated in darkness and aerobic conditions at 37°C for 18 h. Specifically, the glass door of the incubator was covered with aluminum foil to ensure darkness and simulate implanted or dark conditions. After incubation, the antimicrobial efficacy of each electrode was quantified by measuring the ZOI width relative to the known diameter of the disc-shaped electrode. To account for experimental variability, three independent biological replicates were conducted for each electrode type.

2.8 | Electrochemical Measurements

Cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) measurements were performed using a BioLogic VSP-3 potentiostat in a custom-designed three-electrode Teflon electrochemical test cell, consistent with our previously published reports [9, 79]. The electrochemical test cell consisted of the working electrode (uncoated Pt-10Ir, O-PEALD-coated Pt-10Ir, and O-H-PEALD-coated Pt-10Ir HSR electrodes), a coiled platinum wire counter electrode, and an Ag/AgCl reference electrode (Gamry Instruments Inc., Warminster, PA, USA). A phosphate-buffered saline (PBS) solution (Blood Bank Saline, Azer Scientific, USA) was used as the electrolyte.

The potential window was selected to remain within a range that avoids harmful electrochemical reactions at the tissue–electrode interface. CV measurements were conducted between -0.6 and 0.8 V versus Ag/AgCl at a scan rate of 50 mV s^{-1} for 30 cycles, with all potentials reported relative to the Ag/AgCl reference electrode. The electrode–electrolyte contact area (13.8 mm^2), determined from the exposed electrode footprint, was used to calculate specific capacitance and total charge storage capacity from the CV data. EIS measurements were performed at the open-circuit potential (OCP) using a 10 mV (V_{rms}) sinusoidal excitation over a frequency range of $0.1\text{--}10^5$ Hz. EIS spectra were acquired following both 30 and 1500 CV cycles for all working electrode configurations.

3 | Results and Discussion

3.1 | Morphology and Microstructure

A total of 220 ALD cycles were performed for both O-PEALD and O-H-PEALD processes to deposit the ZnO_x layers. Ellipsometry measurements on coated silicon wafers indicated film thicknesses of 30.0 ± 0.1 and 25.0 ± 0.1 nm for the O-PEALD and O-H-PEALD ZnO_x films, respectively. Accordingly, the PEALD coatings correspond to average growth rates of approximately 0.14 nm per cycle and 0.11 nm per cycle for the O-PEALD and O-H-PEALD ZnO_x films, respectively.

The ALD film thicknesses measured by ellipsometry were independently confirmed by atomic force microscopy (AFM) analysis of ALD-coated silicon wafers that had been selectively masked with Kapton tape prior to deposition to define a sharp coating boundary. AFM line-scan analysis revealed

a step height of approximately 30 nm (Figure S1), in good agreement with the ellipsometry measurements. The coated region exhibited a distinct and uniform purple hue (not shown), consistent with the presence of a uniform nanoscale film. The uniform contrast observed across the coated region in the SEM image (Figure S1a) further confirms the conformal growth and excellent morphological homogeneity characteristic of the ALD process.

Figure 5 presents SEM micrographs of uncoated and ALD-coated electrodes, demonstrating that both O-PEALD and O-H-PEALD layers conformally coat the electrode surface without altering the underlying hierarchical structure and morphology of the HSR electrodes. Figure 6a–c shows high-resolution SEM micrographs of the three electrode types—uncoated, O-PEALD ZnO_x -coated, and O-H-PEALD ZnO_x -coated HSR Pt-10Ir electrodes. These micrographs indicate that both O-PEALD and O-H-PEALD films partially cover and blunt nanoscale surface features, particularly at the dome apexes of the hierarchically structured electrode surface. Figure 6d presents an SEM micrograph of a focused ion beam (FIB)-milled electrode cross-section, demonstrating that the PEALD coatings, in general, uniformly and conformally cover the electrode surface.

3.2 | Compositional and Structural Characterization

Figure S2 presents an SEM micrograph and corresponding EDS elemental maps of a partially O-PEALD-coated silicon wafer substrate. Figures 7 and 8 show SEM micrographs and associated EDS elemental mappings of partially coated HSR Pt-10Ir electrodes processed by O-PEALD and O-H-PEALD, respectively. All electrodes were partially masked with Kapton tape during ALD deposition to create well-defined coated and uncoated regions. The EDS elemental maps reveal several consistent trends: (a) signals from the underlying substrates—Si in Figure S2, and Pt and Ir signals in Figures 7 and 8—are slightly attenuated in the ALD-coated regions due to coverage by the ZnO_x layer; (b) Zn is detected exclusively within the ZnO_x -coated regions, confirming spatially selective deposition; and (c) oxygen is present in both coated and uncoated areas, although its intensity is higher in the ZnO_x -coated regions, as expected for ZnO_x films.

Collectively, the EDS elemental mappings indicate that the ALD process achieves uniform deposition of ZnO_x across all substrates and electrode surfaces. Quantitative EDS analysis of the O-PEALD coating on the silicon wafer reveals an approximately 1:1 Zn:O atomic ratio, confirming that the deposited film is predominantly stoichiometric ZnO. Quantitative EDS analysis of the O-H-PEALD coating on the Si wafer shows an approximate Zn:O atomic ratio of 2:1, suggesting that the film consists of a mixture of ZnO and metallic Zn, as further confirmed by the XRD results discussed below.

Figure 9a presents the Zn_{2p} XPS spectra of O-PEALD ZnO_x films deposited on flat Pt-10Ir electrodes, and hierarchically restructured (HSR) Pt-10Ir electrodes, while Figure 9b shows the Zn_{2p} spectrum of an O-H-PEALD ZnO_x film deposited on an HSR Pt-10Ir electrode. Distinct Zn_{2p} doublet peaks are observed for the O-PEALD ZnO_x films at binding energies of 1022.0 eV ($\text{Zn}_{2p_{3/2}}$)

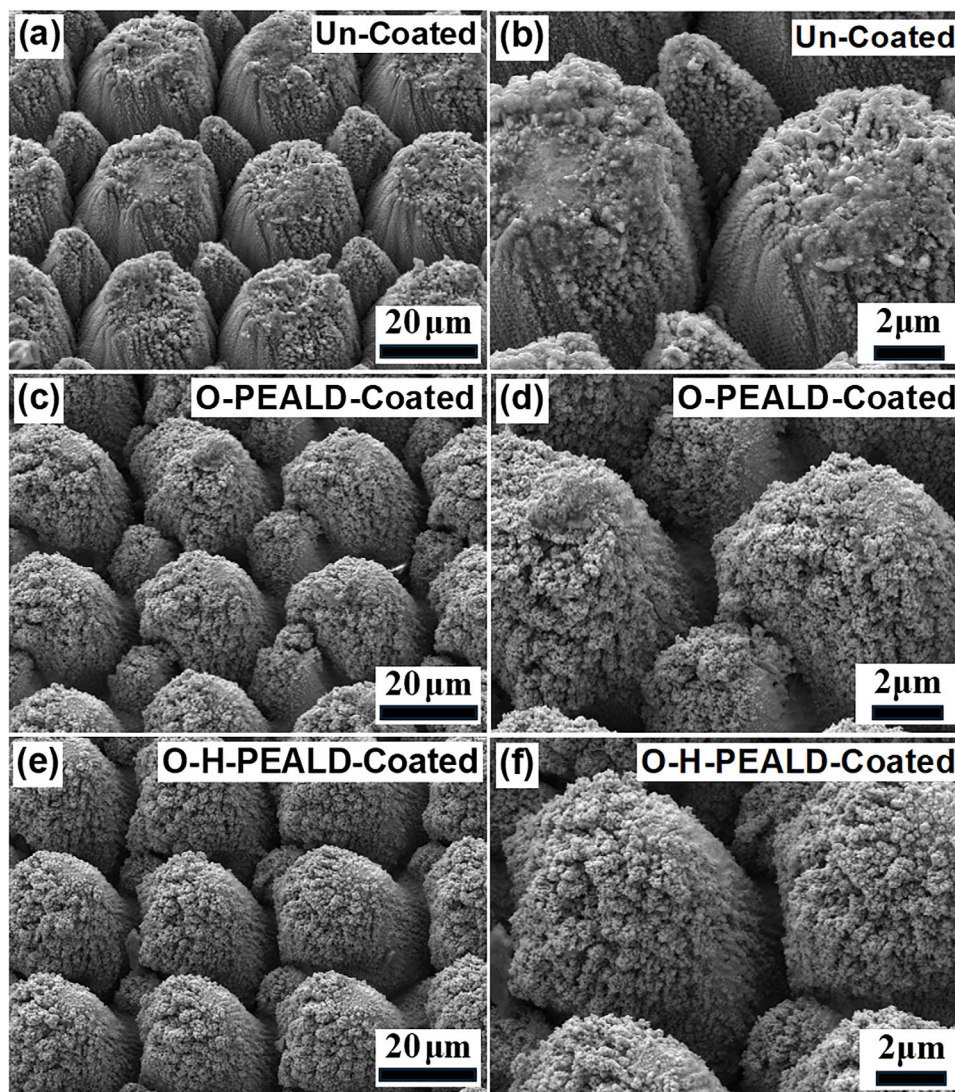


FIGURE 5 | SEM micrographs of HSR Pt-10Ir electrodes: (a,b) uncoated electrodes; (c,d) O-PEALD ZnO_x-coated electrodes; and (e,f) O-H-PEALD ZnO_x-coated electrodes.

and 1045.0 eV (Zn_{2p1/2}), which are characteristic of Zn²⁺ in a fully oxidized ZnO chemical environment [80, 81].

The absence of any detectable peak shift or asymmetry confirms the chemical uniformity and stability of Zn–O bonding across all substrates and electrodes, indicating that the ZnO_x coatings are chemically consistent and independent of surface morphology or underlying substrate type. The narrow full width at half maximum (FWHM) of the Zn_{2p} peaks further indicates the presence of a single Zn oxidation state, with no evidence of metallic Zn or sub-oxide species within the near-surface region of the O-PEALD and O-H-PEALD films [82].

Figure S3 presents the O_{1s} XPS spectra of O-PEALD and O-H-PEALD ZnO_x thin films deposited on HSR Pt-10Ir electrodes. Both spectra exhibit a single, symmetric O_{1s} peak centered at 530.1 ± 0.1 eV, which is characteristic of lattice oxygen (O²⁻) bonded to Zn²⁺ within the wurtzite ZnO crystal structure [83]. Moreover, the O_{1s} spectra exhibit no discernible shoulders or secondary peaks, which are typically associated with oxygen-deficient sites, hydroxyl groups, or adsorbed molecular species [82, 83]. This

further confirms that the near-surface regions of both O-PEALD and O-H-PEALD thin films are chemically stoichiometric ZnO, with negligible incorporation of defect-related oxygen states.

Figure 9c presents the grazing-incidence X-ray diffraction (GIXRD) patterns of ZnO_x thin films deposited on Si substrates via the O-PEALD and O-H-PEALD processes. The O-PEALD ZnO_x film exhibits diffraction reflections corresponding exclusively to the wurtzite ZnO phase (JCPDS No. 36-1451), indicating the formation of phase-pure ZnO. In contrast, the O-H-PEALD ZnO_x film exhibits the coexistence of metallic Zn (JCPDS No. 04-0831) and wurtzite ZnO (JCPDS No. 36-1451), indicating partial reduction of ZnO during the hydrogen plasma exposure step. For the O-PEALD ZnO_x film, the strong diffraction peak at approximately 34.4° corresponding to the (002) plane indicates a pronounced preferential c-axis orientation/growth, which is a characteristic feature of PEALD-grown ZnO films on smooth substrates [84]. For the O-H-PEALD ZnO_x film, the pronounced diffraction peak at approximately 36.2° (101) correspond to wurtzite ZnO, indicating a reoriented growth compared to the O-PEALD film. Additionally, distinct diffraction reflections near

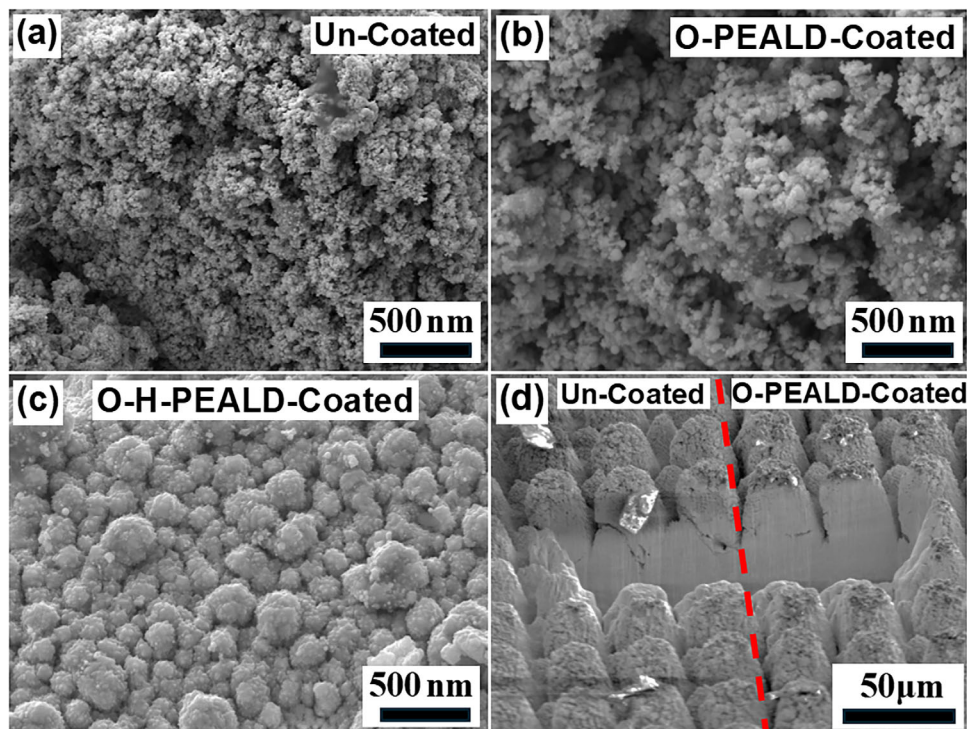


FIGURE 6 | High-resolution SEM micrographs of HSR Pt-10Ir electrodes: (a) uncoated, (b) O-PEALD ZnO_x -coated, and (c) O-H-PEALD ZnO_x -coated electrodes. (d) Focused ion beam (FIB) cross-sectional micrograph of a masked HSR Pt-10Ir electrode, showing the uncoated and O-PEALD ZnO_x -coated regions separated by the dashed red demarcation line.

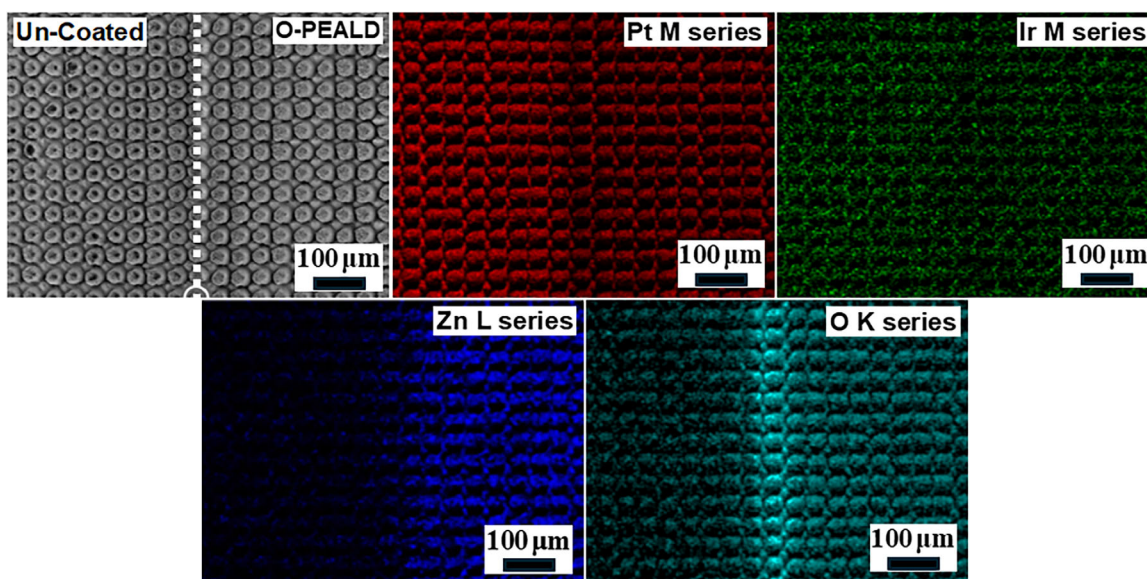


FIGURE 7 | SEM micrograph and corresponding EDS elemental maps of a partially O-PEALD-coated HSR Pt-10Ir electrode.

$\approx 39.0^\circ$ (101) and $\approx 43.2^\circ$ (102) are indexed to metallic Zn (JCPDS No. 04-0831), confirming the presence of crystalline Zn within the O-H-PEALD film.

It should be noted that, although GIXRD reveals the presence of metallic Zn, the corresponding XPS analysis of the O-H-PEALD ZnO_x film does not exhibit signatures of

metallic Zn. This apparent discrepancy is attributed to the fundamentally different probing depths of the two techniques: GIXRD probes the full film thickness (≈ 30 nm), whereas XPS is highly surface-sensitive, with an information depth limited to only a few nanometers. The metallic Zn detected by GIXRD is therefore attributed to subsurface regions of the film, whereas the near-surface region becomes fully

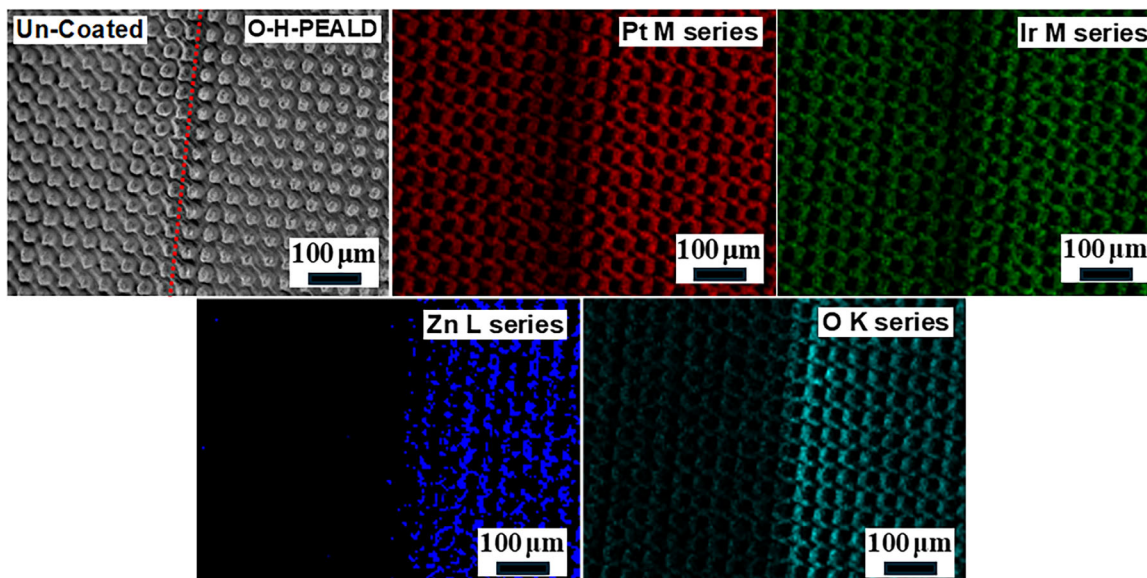


FIGURE 8 | SEM micrograph and corresponding EDS elemental maps of a partially O-H-PEALD-coated HSR Pt-10Ir electrode.

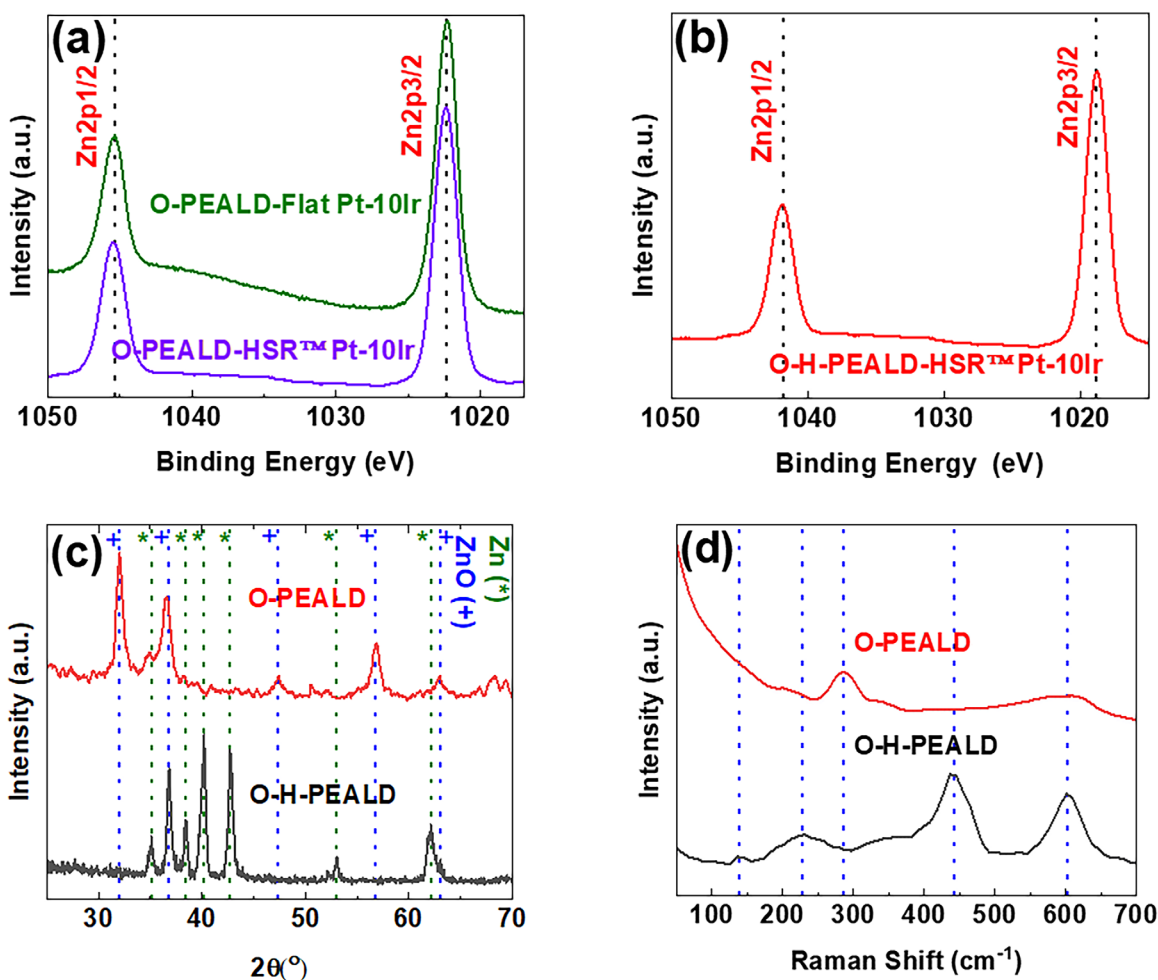


FIGURE 9 | (a) Zn₂p XPS spectra of O-PEALD-coated flat/unrestructured Pt-10Ir and HSR Pt-10Ir electrodes. (b) Zn₂p XPS spectrum of an O-H-PEALD-coated HSR Pt-10Ir electrode. (c) Grazing-incidence X-ray diffraction (GIXRD) patterns of ZnO_x films deposited on Si substrates via O-PEALD (red, top) and O-H-PEALD (black, bottom) processes. Blue crosses denote diffraction peaks corresponding to ZnO, while green stars indicate peaks associated with metallic Zn. (d) Raman spectra of ZnO_x films deposited via O-PEALD (red, top) and O-H-PEALD (black, bottom) processes.

oxidized during the terminal oxidation steps of the ALD process.

Figure 9d presents the Raman spectra of the O-PEALD (top) and O-H-PEALD (bottom) ZnO_x coatings deposited on stainless-steel substrates, revealing distinct differences in their spectral features. The O-H-PEALD ZnO_x film exhibits prominent vibrational modes at approximately 437 cm⁻¹ (E₂ high) and 599 cm⁻¹ (A₁(LO)), which are characteristic of crystalline ZnO and correspond to Zn–O lattice vibrations within the wurtzite structure [85]. The O-PEALD ZnO_x film shows a dominant band near 300 cm⁻¹, suggesting a different wurtzite ZnO orientation [85].

Finally, the Zn LMM Auger spectra shown in Figure S4 for both O-PEALD and O-H-PEALD films deposited on Si substrates exhibit peaks centered at approximately 989–990 eV. These features are characteristic of Zn²⁺ in stoichiometric ZnO and clearly distinguish the oxidized zinc state from metallic Zn, which typically exhibits Zn LMM Auger peaks near ≈992 eV [86].

This confirms that hydrogen plasma exposure does not alter the oxidation state of Zn within the near-surface region of the crystalline ZnO film. Consistent with the XPS results, the apparent discrepancy between the Auger spectroscopy and GIXRD analyses for the O-H-PEALD ZnO_x thin film is most likely attributable to depth sensitivity differences between the techniques. Specifically, any metallic Zn present near the film surface is expected to be oxidized during the terminal oxidation steps of the PEALD process, rendering it undetectable by surface-sensitive techniques such as XPS and Auger spectroscopy, while remaining observable by GIXRD, which probes the full film thickness.

Thus, by combining XPS, GIXRD, Raman, and Auger spectroscopic analyses, it is evident that the O-PEALD ZnO_x film consists predominantly of stoichiometric wurtzite ZnO, with no significant oxygen vacancies or zinc interstitials. In contrast, the O-H-PEALD ZnO_x film is effectively a ZnO–Zn nanocomposite coating, comprising stoichiometric wurtzite ZnO together with a substantial fraction of metallic Zn.

3.3 | Zn²⁺ Ion Release Behavior of ALD-Deposited ZnO_x Films

To quantify Zn²⁺ ion release from the ALD-deposited ZnO_x layer and assess its relevance to antibacterial activity, inductively coupled plasma and mass spectrometry (ICP-MS) measurements were performed on uncoated, O-PEALD-coated and O-PEALD-coated flat/unrestructured and HSR Pt–10Ir electrodes immersed in distilled water, as shown in Figure 10. The uncoated flat and HSR Pt–10Ir electrodes exhibited no detectable Zn²⁺ ion release, with concentrations remaining below the ICP-MS detection limit, as expected. In contrast, PEALD-coated flat and HSR Pt–10Ir electrodes showed measurable and significant Zn²⁺ ion release into the surrounding medium.

For the PEALD-coated HSR Pt–10Ir electrodes, the Zn²⁺ ion concentration increased with immersion time, reaching approximately 0.5 ppm for the O-PEALD-coated electrodes and 0.8 ppm for the O-H-PEALD-coated electrodes after ≈120 min, respec-

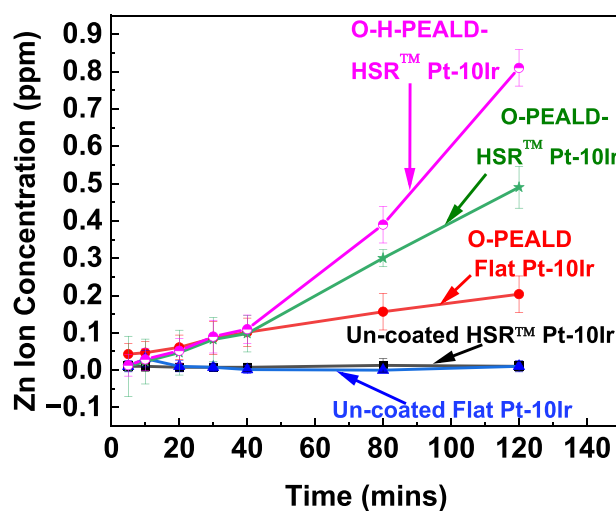


FIGURE 10 | Zn²⁺ ion concentration in water measured by ICP-MS for uncoated, O-H-PEALD and O-PEALD ZnO-coated Pt–10Ir electrodes. Electrodes were immersed in distilled water, and aliquots were collected at defined time intervals to quantify Zn²⁺ ion release as a function of immersion time. Error bars represent the standard deviation from two to eight independent measurements.

tively. In contrast, the Zn²⁺ ion concentration released from O-PEALD-coated flat Pt–10Ir electrodes was approximately threefold-fourfold lower over the same time period. As a function of immersion time, after 60 min of release, the Zn²⁺ ion release rate from the O-PEALD-coated HSR Pt–10Ir electrodes was more than twofold higher than that observed for the O-PEALD-coated flat electrodes. Notably, Zn²⁺ ion release did not reach a plateau within the 2 h measurement window, indicating that continued immersion would likely result in further increases in Zn²⁺ ion concentration.

The substantially higher Zn²⁺ ion release rate observed for the ZnO PEALD-coated HSR electrodes, relative to their flat counterparts, is attributed to the significantly larger specific surface area provided by the hierarchically restructured (HSR) architecture of the electrodes [9]. Thus, the ALD-deposited ZnO coating enables substantial Zn²⁺ ion release into the surrounding medium, with the ZnO-coated HSR electrodes exhibiting significantly higher Zn²⁺ ion release efficiency compared to their ALD-coated flat counterparts.

The maximum Zn²⁺ concentration released from the ZnO-coated electrodes was approximately 0.5–0.8 ppm within a 2-hour releasing time (Figure 10). This concentration is substantially lower than Zn²⁺ levels (in the order of ≈15 ppm) that have been reported to induce observable cytotoxic responses in mammalian cells [87–89]. Previous studies have demonstrated that many mammalian cell types, including fibroblasts and neuronal cells, generally tolerate Zn²⁺ concentrations in the range of 10–100 μM, with appreciable reductions in cell viability occurring only at substantially higher concentrations [89]. Consequently, the relatively low Zn²⁺ release observed in the present study suggests that the antibacterial ZnO coatings may provide antimicrobial functionality while releasing Zn²⁺ within a concentration range that is unlikely to cause significant cytotoxic effects.

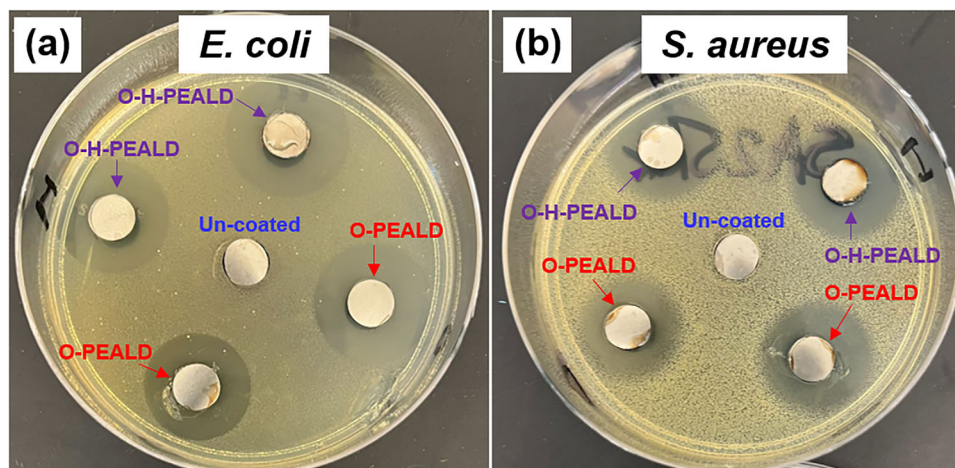


FIGURE 11 | Zone-of-inhibition (ZOI) assays for uncoated HSR-Pt10Ir electrodes and those coated with O- and O-H-PEALD coatings against (a) *E. coli* and (b) *S. aureus* strains.

TABLE 1 | Zone-of-inhibition extension width (ZOI-EW) measurements for ZnO_x-coated (O-PEALD and O-H-PEALD) HSR Pt-10Ir electrodes evaluated under dark, aerobic conditions against *Escherichia coli* (*E. coli*) K12 (Gram-negative) and *Staphylococcus aureus* (*S. aureus*) ATCC 27660 (Gram-positive).

Bacteria ↓	Uncoated HSR Pt-10Ir ZOI-EW (mm)	O-PEALD HSR Pt-10Ir ZOI-EW (mm)	O-H-PEALD HSR Pt-10Ir ZOI-EW (mm)
<i>E. coli</i>	0	6.9 ± 0.4	7.9 ± 0.5
<i>S. aureus</i>	0	6.3 ± 0.6	7.2 ± 0.9

3.4 | Antibacterial Properties

The antibacterial efficacy of ZnO-coated HSR Pt-10Ir electrodes under dark conditions was evaluated using a modified Kirby-Bauer method [78]. As shown in Figure 11, a pronounced zone-of-inhibition (ZOI) formed around both O-PEALD- and O-H-PEALD-coated HSR Pt-10Ir electrodes in cultures of *E. coli* and *S. aureus*, respectively, indicating strong antibacterial activity in the absence of light.

In this study, antibacterial efficacy was estimated using the zone-of-inhibition extension width (ZOI-EW) rather than the conventional ZOI diameter [9]. ZOI-EW provides a direct measure of the inhibited region surrounding the electrode and is independent of electrode size, allowing for consistent comparison between electrodes with different geometries. Furthermore, a ZOI-EW value of zero clearly denotes negligible or absent antibacterial activity.

Table 1 summarizes the ZOI-EW measurements for ZnO_x-coated (O-PEALD and O-H-PEALD) HSR Pt-10Ir electrodes evaluated under dark, aerobic conditions against *E. coli* K12 (Gram-negative) and *S. aureus* ATCC 27660 (Gram-positive). Uncoated HSR Pt-10Ir electrodes exhibit a ZOI-EW of zero, indicating negligible antibacterial activity, which is consistent with our previous findings [9] and confirms that hierarchical surface restructuring alone does not impart antibacterial functionality.

Furthermore, both O-PEALD- and O-H-PEALD-coated HSR Pt-10Ir electrodes exhibit large ZOI-EW values, demonstrating strong antibacterial efficacy against both *E. coli* and *S. aureus*. Compared to O-PEALD, the O-H-PEALD ZnO_x coatings exhibit significantly larger ZOI-EW values (by approximately 14%) against both *E. coli* and *S. aureus*, indicating enhanced antibacterial efficacy relative to their O-PEALD counterparts.

Finally, the ZOI-EW values for both O-PEALD- and O-H-PEALD-coated HSR Pt-10Ir electrodes are slightly (≈10%) larger in *E. coli* cultures than in *S. aureus* cultures, suggesting that the PEALD ZnO_x coatings exhibit modestly higher antibacterial efficacy against *E. coli* compared to *S. aureus*. These findings confirm that conformal ZnO_x films deposited via PEALD impart potent antibacterial activity even under dark conditions, indicating that Zn²⁺ ion release—rather than photoinduced reactive oxygen species (ROS) generation—is the dominant mechanism driving bacterial inhibition. The enhanced antibacterial response observed for the O-H-PEALD films is likely attributable to the presence of metallic Zn domains, which undergo corrosion in aqueous environments and consequently generate higher concentrations of Zn²⁺ ions compared to O-PEALD ZnO films, whose intrinsic solubility in water is extremely low.

Notably, prior studies [90] have shown that the dissolution of antimicrobial metals can be slower in nutrient-rich media than in pure water; therefore, the ability of the O-H-PEALD coatings

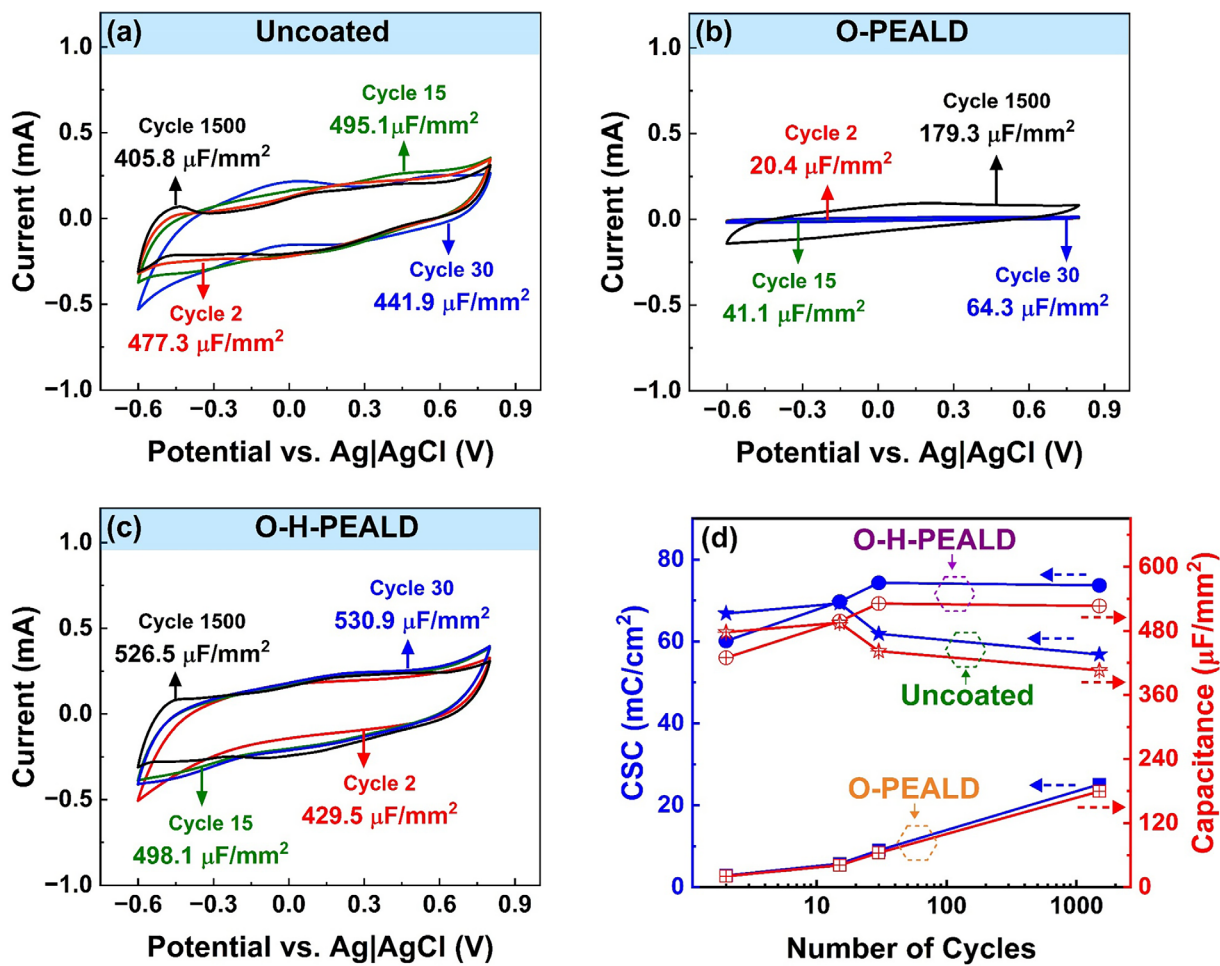


FIGURE 12 | Cyclic voltammograms of (a) uncoated, (b) O-PEALD-coated, and (c) O-H-PEALD-coated HSR Pt-10Ir electrodes recorded at a scan rate of 50 mV s^{-1} , (d) specific capacitance (red) and total charge storage capacity (blue) vs. the CV cycle number, respectively.

to sustain Zn^{2+} release under such conditions further underscores their capacity to deliver an effective antibacterial dose.

Although the modified Kirby–Bauer assay provides a convenient and widely used method for screening antibacterial activity, the resulting zone of inhibition should be interpreted with caution for implantable applications. The extent of bacterial growth inhibition is influenced by the diffusion of antibacterial species through the agar matrix and therefore may not directly represent antibacterial efficacy under physiological conditions. Indeed, implanted materials rapidly become coated with adsorbed proteins and are exposed to complex tissue environments that influence ion transport, bacterial adhesion, and biofilm formation. Therefore, these promising *in vitro* studies will need to be validated with future studies involving different geometries (e.g., coated cylindrical electrodes) and simulated or *in vivo* conditions.

3.5 | Electrochemical Performance

Figure 12a–c presents the cyclic voltammetry (CV) curves of uncoated and ZnO_x -coated HSR Pt-10Ir electrodes. Representative CV curves are shown for the 2nd, 15th, 30th, and 1500th cycles to illustrate the electrochemical evolution with cycling. The corresponding specific capacitance and charge storage capacity

(CSC) values as a function of cycle number are presented in Figure 12d and summarized in Table 2. Figure 12d indicates that the specific capacitance and CSC values slightly ($\approx 4\%$) increase from CV cycle 2 to 15 and then drop significantly ($\approx 11\%$).

For the uncoated HSR Pt-10Ir electrode, Figure 12a shows a nearly semi-rectangular CV profile across all cycles, characteristic of predominantly capacitive charge exchange behavior. As shown in Figure 12d, both the specific capacitance and total charge storage capacity (CSC_t) increase slightly ($\approx 3.7\%$) from the 2nd to the 15th CV cycle, followed by a decrease ($\approx 10.7\%$).

The initial increases in specific capacitance and total charge storage capacity can be attributed to progressive wetting and electrochemical activation of the porous hierarchical surface structure of the electrodes, which facilitates improved electrolyte penetration and more complete utilization of electrochemically active sites.

For the O-PEALD- ZnO_x -coated HSR Pt-10Ir electrodes, Figure 12b shows CV profiles with substantially reduced enclosed areas across all cycles, indicating significantly lower electrochemical activity. Specifically, both the specific capacitance and total charge storage capacity are reduced by approximately 85% at the 30th cycle relative to the uncoated

TABLE 2 | Specific capacitance values in $\mu\text{F mm}^{-2}$ exhibited by uncoated, O-PEALD ZnO-coated and O-H-PEALD ZnO-Zn-coated HSR electrodes at the specified CV cycles.

Specific capacitance ↓ ($\mu\text{F mm}^{-2}$)	CV cycle number →	2	15	30	1500
Uncoated HSR Pt-10Ir		477.35	495.11	441.97	405.87
O-PEALD ZnO-coated HSR Pt-10Ir		20.42	41.10	64.28	179.34
O-H-PEALD-ZnO-Zn coated HSR Pt-10Ir		429.50	498.12	530.94	526.48

electrodes. Figure S5 presents CV profiles plotted on an expanded current scale, more clearly revealing the predominantly capacitive behavior of the O-PEALD-ZnO_x-coated electrodes. As shown in Figure 12d and summarized in Table 2, both the specific capacitance and total charge storage capacity increase markedly from the 2nd to the 1500th CV cycle. The specific capacitance increases substantially from $\approx 20 \mu\text{F mm}^{-2}$ at the 2nd cycle to $\approx 179 \mu\text{F mm}^{-2}$ at the 1500th cycle.

A similar trend was reported in previous studies [63] of ZnO-coated HSR Pt-10Ir electrodes prepared by reactive magnetron sputtering, where extended electrochemical cycling was accompanied by progressive ZnO dissolution, resulting in gradual stripping of the ZnO layer into the electrolyte over time. In the present study, the likely removal of the conformal O-PEALD-ZnO coating, which has inherently low electrical conductivity [91], appears to expose the underlying metallic Pt10Ir HSR surface, restoring electron transport pathways and leading to the observed increase in specific capacitance. Despite slight capacity increase over extended cycling, in general, the O-PEALD-ZnO coating largely compromise the HSR electrode's electrochemical performance [91].

Here, the O-PEALD process deposits an ultra-conformal $\approx 30 \text{ nm}$ ZnO coating across the entire nanostructured surface of the HSR Pt-10Ir electrode, introducing a resistive barrier to electron transport and impeding charge percolation at the Pt-10Ir/electrolyte interface, thereby limiting the electrode's total charge storage capability.

For the O-H-PEALD-coated HSR Pt-10Ir electrodes, Figure 12c shows predominantly capacitive behavior across all cycles. As shown in Figure 12d, both the specific capacitance and total charge storage capacity increase significantly ($\approx 24\%$) from the 2nd to the 30th CV cycle, after which they stabilize and remain essentially constant from the 30th through the 1500th cycle, with no observable degradation. Similar to the uncoated electrodes, the initial increases in capacitance and total charge storage capacity are attributed to progressive wetting and electrochemical activation of the porous hierarchical surface. Notably, compared to the uncoated counterpart, the O-H-PEALD-coated HSR Pt-10Ir electrodes exhibit substantially higher ($\approx 25\%$) capacitance and total charge storage capacity during extended CV cycling (30th–1500th cycles) at a scan rate of 50 mV s^{-1} . This enhancement in electrochemical performance can be attributed to the presence of metallic Zn within the O-H-PEALD ZnO-Zn nanocomposite coating, which improves electron transport and facilitates more efficient charge transfer at the electrode–electrolyte interface.

Both O-PEALD and O-H-PEALD coatings remained electrochemically functional throughout the testing period, indicating that the ALD ZnO films can withstand repeated electrochemical polarization without catastrophic degradation. However, ICP-MS measurements reveal measurable Zn^{2+} release from both coatings, with the O-H-PEALD films exhibiting greater ion release than the O-PEALD films, suggesting comparatively higher dissolution rates.

Although direct mechanical adhesion testing was not performed, no visible signs of coating delamination or failure were observed during electrochemical measurements or handling, qualitatively indicating good adherence of the ALD coatings to the hierarchically restructured Pt-10Ir electrodes. The observed gradual Zn^{2+} release suggests that the coatings are not rapidly depleted and are expected to retain antibacterial functionality during the early post-implantation period, when the risk of implant- and surgery-associated infection is the greatest. Future research involving extended electrochemical cycling, quantitative adhesion measurements, and long-term stimulation experiments under physiologically relevant conditions are required to more rigorously establish the optimal coating thickness, the coating lifetime, and degradation kinetics.

To further corroborate the role of metallic Zn in enhancing electrochemical charge transport and interfacial conductivity, Figure 13a,b presents electrochemical impedance spectroscopy (EIS) Bode plots collected after 30 and 1500 CV cycles, respectively, for uncoated, O-PEALD-coated, and O-H-PEALD-coated HSR Pt-10Ir electrodes. The O-PEALD-coated HSR Pt-10Ir electrodes exhibit substantially higher impedance across the entire frequency range compared to both the uncoated and O-H-PEALD-coated electrodes, indicating that the conformal crystalline ZnO layer grown under oxygen plasma conditions significantly restricts electrical conduction. In contrast, the O-H-PEALD-coated electrodes exhibit the lowest impedance, particularly in the mid- to low-frequency regime, consistent with enhanced electrical conductivity arising from metallic Zn within the ZnO-Zn nanocomposite structure formed during hydrogen plasma processing. Following extended electrochemical cycling up to 1500 CV cycles, all electrodes display an overall reduction in impedance.

As discussed earlier, this decrease is likely attributable to the progressive dissolution of the low-conductivity ZnO coating from the electrode surface [63], which reduces the overall resistance to charge transfer at the electrode–electrolyte interface. More importantly, Figure 13a,b shows that the O-H-PEALD-coated HSR Pt-10Ir electrodes consistently maintain the lowest

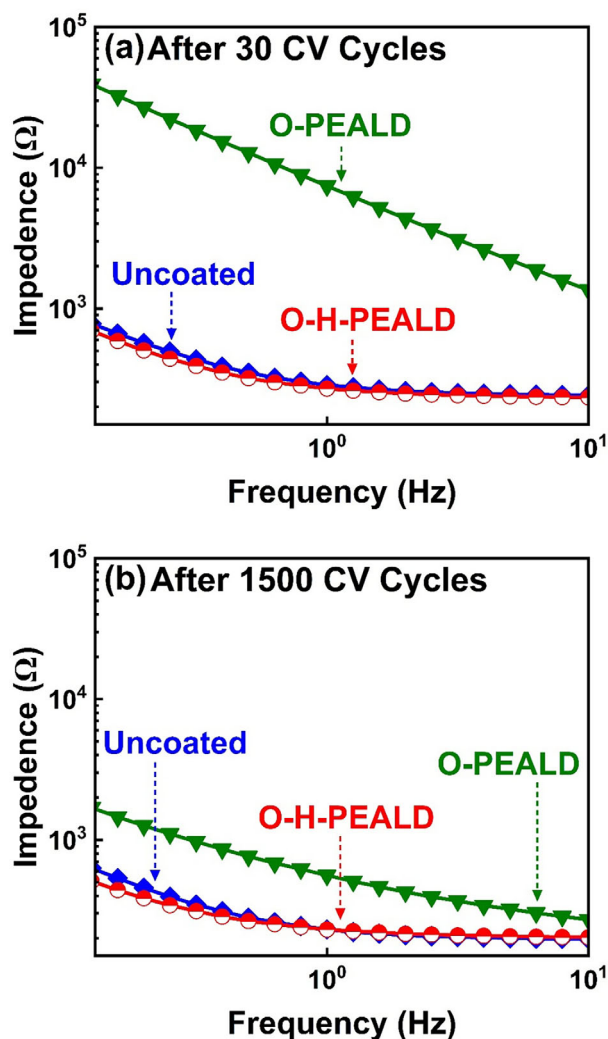


FIGURE 13 | Bode impedance plots for uncoated, O-PEALD-coated, and O-H-PEALD-coated HSR Pt-10Ir electrodes after (a) 30 CV cycles and (b) 1500 CV cycles. Expanded views of panels (a) and (b) are provided in Figure S6.

impedance profile across the measured frequency range. As shown in Figure S6a,b, the O-H-PEALD-coated HSR Pt-10Ir electrodes exhibit approximately 17% lower impedance than the uncoated counterparts, confirming that the incorporation of metallic Zn domains within the ZnO matrix significantly enhances electrical conductivity and facilitates more efficient charge transfer and capacitive behavior.

3.6 | Comparison with State-of-the-Art Antibacterial Electrode Coatings

To benchmark the performance of the developed PEALD ZnO coatings, Table 3 compares the previously reported coating systems with representative antibacterial coatings for implantable electrodes. Although Ag- [92] and Cu-based [93] coatings exhibit excellent antibacterial efficacy, their clinical translation may be limited by concerns regarding cytotoxicity and long-term ion release. TiO₂-based coatings require UV/light to create ROS for antibacterial efficacy so that they are not compatible with antibac-

terial implantation applications in dark conditions. In contrast, the O-H-PEALD ZnO coatings developed in this work provide antibacterial activity under dark conditions while maintaining acceptable electrochemical characteristics and releasing Zn²⁺ concentrations below reported cytotoxic thresholds. Furthermore, the conformal nature of PEALD enables uniform coating of the complex HSR Pt-10Ir electrode architecture, demonstrating a favorable balance between antibacterial performance, electrochemical stability, and biocompatibility for multifunctional implantable neural interfaces.

4 | Concluding Remarks

This work establishes a dual-step surface engineering strategy that decouples and independently optimizes electrochemical performance and antibacterial functionality in implantable Pt-10Ir neural interfacing electrodes. By integrating femtosecond laser-based hierarchical surface restructuring (HSR) with plasma-enhanced atomic layer deposition (PEALD), antibacterial zinc oxide (ZnO) thin films are introduced as a functional overlay onto hierarchically restructured Pt-10Ir electrodes without compromising electrodes' geometric fidelity, mechanical integrity, or manufacturability.

A direct comparison between oxygen plasma PEALD (O-PEALD) and oxygen-hydrogen plasma PEALD (O-H-PEALD) reveals that film chemistry and phase structure play a decisive role in governing coupled electrochemical and antibacterial performance. While stoichiometric crystalline ZnO deposited via O-PEALD provides effective antibacterial activity under dark, aerobic conditions through Zn²⁺ ion release, its intrinsically low electrical conductivity introduces a resistive barrier that degrades electrochemical efficiency. In contrast, O-H-PEALD produces a ZnO-Zn nanocomposite coatings in which conductive metallic Zn domains enable efficient electron transport while sustaining antibacterial efficacy, resulting in enhanced capacitance, increased charge storage capacity, and reduced interfacial impedance relative to uncoated HSR electrodes.

Importantly, antibacterial activity is maintained under dark conditions, confirming that controlled Zn²⁺ ion release—rather than photo-induced reactive oxygen species—dominates the bactericidal mechanism. The enhanced antibacterial response observed for O-H-PEALD coatings is attributed to sustained Zn²⁺ release arising from dissolution of metallic Zn domains, demonstrating a mechanism that is both effective and relevant for long-term implantation.

Beyond the specific ZnO-based system investigated here, this work defines a broadly applicable platform for integrating functional thin-film overlays onto hierarchically restructured Pt-10Ir electrodes without sacrificing electrochemical performance or scalability. Collectively, these findings establish a scalable, commercially viable, and antibiotic-free pathway toward infection-resistant, high-performance neural interfacing electrodes, advancing the translation of next-generation implantable neural interfaces for long-term clinical deployment.

TABLE 3 | Comparison of antibacterial performance, electrochemical characteristics, biocompatibility, and stability of representative antibacterial coatings for implantable electrodes and the O-H-PEALD ZnO-coated HSR Pt-10Ir electrodes developed in this work.

Coating system	Antibacterial mechanism	Antibacterial performance	Electrochemical performance	Biocompatibility	Stability/limitations	Refs.
Ag nano-particles	Ag ⁺ ion release, ROS generation	Excellent against Gram-positive and Gram-negative bacteria	Can increase impedance and alter charge transfer properties	Potential cytotoxicity at elevated Ag ⁺ concentrations	Long-term ion depletion and cytotoxicity concerns	[92]
Cu/CuO	Cu ²⁺ ion release, membrane disruption, oxidative stress	Strong antibacterial activity in dark conditions	May reduce impedance depending on morphology	Dose-dependent cytotoxicity due to Cu ²⁺ release	Oxidation and high ion release may limit chronic use	[9, 93]
TiO ₂	Photocatalytic ROS generation	Effective primarily under UV/light activation	Limited electrochemical performance	Excellent biocompatibility	Limited antibacterial efficacy in dark physiological environments	[94]
ZnO	Zn ²⁺ release, ROS generation, membrane damage	Good antibacterial activity	Electrochemical properties depend on deposition method and thickness	Generally favorable at low Zn ²⁺ concentrations	Stability dependent on film adhesion and dissolution rate	[63, 60]
O-H-PEALD ZnO (current work)	Controlled Zn ²⁺ release and bacterial membrane disruption	Strong antibacterial activity in dark	Good electrochemical characteristics	Zn ²⁺ release below reported cytotoxic thresholds	Conformal ALD coating on hierarchical surfaces and good electrochemical stability	This work

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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