Smart Energy and Sustainable Environment

2026, *29(1)*: 55-64, https://doi.org/10.46390/j.smensuen.29126.467

PLATINUM-BASED ELECTROCATALYSTS PREPARED BY ATOMIC LAYER DEPOSITION FOR PROTON EXCHANGE MEMBRANE FUEL CELLS: FROM FUNDAMENTALS TO PERFORMANCE AND DURABILITY

Silvia-Iustina Pintilie[1*], Luminița Drăgăşanu[2], Cornel Stoica[3], Adriana Marinoiu[1]

[1]National Research and Development Institute for Cryogenic and Isotopic Technologies – ICSI Rm. Valcea, ICSI-Nuclear-Cryogenic Laboratory, 4th Uzinei Street, 240050 Ramnicu Valcea, Valcea, Romania
[2]COMOTI National Institute for Turboengine Research and Development, 220D Iuliu Maniu Boulevard, sector 6, code 061126, PO Box 76, CP174 Bucharest, Romania.
[3]National Institute for Aerospace Research and Development "Elie Carafoli", 220 Iuliu Maniu Boulevard, Sector 6, 061126, Bucharest, Romania

Abstract:

The Atomic Layer Deposition (ALD) method has become, in the last decade, one of the most promising methods for obtaining and optimizing platinum-based electrocatalysts (Pt) for proton exchange membrane fuel cells (PEMFCs), due to the atomic-level control over charge, dispersion and nanometer architecture. This review synthesizes the fundamentals of ALD deposition for obtaining Pt and Pt-alloy catalysts, from growth mechanisms and nucleation, precursor selection and the role of process conditions (surface chemistry, temperature, number of cycles) to their impact on the active structure and composition. Design strategies for ultra-low Pt charge catalysts, including advanced carbon support and conductive oxide depositions, as well as core-shell or Pt-skin approaches to optimize the use of the noble metal, are discussed. The paper analyzes how catalyst structure influences performance in the oxygen reduction reaction (ORR), highlighting how particle size, spatial distribution, degree of alloying, and lattice voltages influence mass and specific activity. Finally, limitations, current gaps, and future directions are identified, including process scaling and standardization of test protocols, to accelerate the transition of laboratory ALD catalysts to commercial PEMFC applications.

Article info:

Received 3 February 2026
Received in revised form 13 May 2026
Accepted 14 May 2026
Available online 18 May 2026

Keywords:
ALD, Electrocatalyst, PEMFC, ORR, Platinum

How to cite: Pintilie S.-I., Drăgăşanu L., Stoica C., Marinoiu A., (2026). Platinum-Based Electrocatalysts Prepared by Atomic Layer Deposition for Proton Exchange Membrane Fuel Cells: From Fundamentals to Performance and Durability, *Smart Energy and Sustainable Environment, 29*(1), 55-64, https://doi.org/10.46390/j.smensuen.29126.467.

[*]*Corresponding author: Silvia-Iustina Pintilie, e-mail: silvia.pintilie@icsi.ro*

1. INTRODUCTION

In the global context of the transition to clean and sustainable energy sources, polymer electrolyte membrane fuel cells (PEMFCs) are a promising technology due to their high efficiency and low emissions [Staffell et al., 2019; Wang et al., 2011]. The principle of these devices relies by converting chemical energy directly, between hydrogen and oxygen, into electricity [Wang et al., 2011; Fuel Cell Handbook, 2004], the only reaction products being water, heat and electricity, according to the reactions below:

$$\textit{Anode:}\quad 2H_2 \rightarrow 4H^+ + 4e^- \tag{1}$$

$$\textit{Cathode:}\quad O_2 + 4H^+ + 4e^- \rightarrow 2H_2O \tag{2}$$

$$\textit{General:}\quad 2H_2 + O_2 \rightarrow 2H_2O + \textit{Electricity} + \textit{Heat} \tag{3}$$

Unlike conventional energy sources, PEM cells avoid combustion and can produce energy with very low local emissions, making them an attractive solution for reducing the carbon footprint of the industrial and transport sectors [Staffell et al., 2019]. Figure 1. describes the main components and principle of operation of these devices.

PEM fuel cells have a variety of applications, including their use in fuel cell electric vehicles (FCEVs), portable devices and stationary power generation systems [Wang et al., 2011; Wu, 2016]. Numerous challenges such as cost, performance at high powers, and especially durability still limit the transition to large-scale deployment of these devices [Staffell et al., 2019]. In the PEMFC architecture, the oxygen reduction reaction (ORR) to the cathode is kinetically slow and requires the use of platinum-based catalysts (Pt), which directly influences cost and sensitivity to degradation [Wang et al., 2011; Stephens et al., 2012; Debe, 2012].

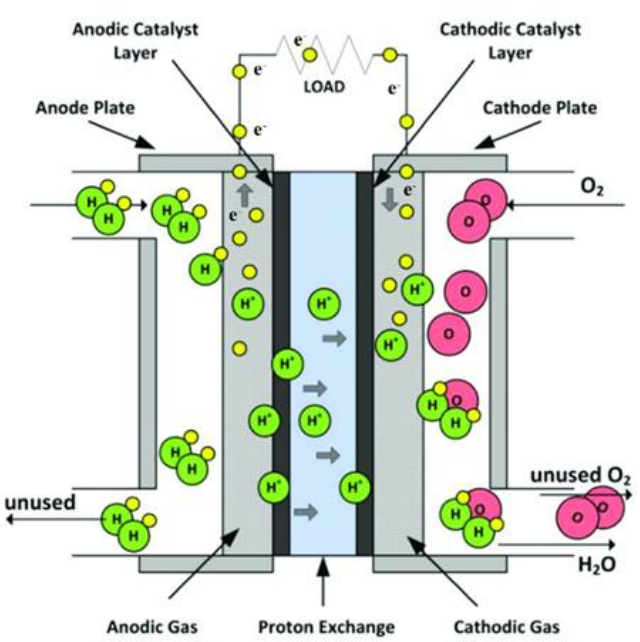

Figure 1. PEM schematic representation [Abbaspour et al., 2014].

Electrocatalysts are indispensable for fuel cells operation, as they accelerate electrochemical reactions at the electrode–electrolyte interface, with reduced efficiency losses. Among the materials used, platinum (Pt) remains the reference catalyst due to its outstanding activity, but the high cost and limited availability are major barriers to the industrial implementation of hydrogen technologies, especially in applications with a high need for active material. Therefore, it is necessary to optimize the consumption of Pt, either by decreasing the catalytic charge or by replacing it with more affordable alternatives. A widely used approach is the distribution of Pt nanoparticles on carbon supports, to raise the active surface area and improve metal utilization. Therefore, the physicochemical properties of the substrate and its interaction with the metal directly determine the activity of the electrocatalyst and long-term stability [Cheng et al., 2016].

Reducing the total amount of Pt needed can be achieved by reducing Pt nanoparticles size to atomic clusters or even isolated atoms, thus considerably increasing the fraction of surface atoms directly involved in electrochemical reactions. But obtaining and maintaining the stability of individual atoms by conventional methods is difficult, because they show a tendency to migrate and aggregate into larger particles [Uzun et al., 2009].

In this sense, the atomic layer deposition (ALD) technique is more and more used to overcome these limitations of the electrocatalysts used in PEMFC, due to the precision in atomic-scale control over thickness, composition and morphology. These methods allow noble metals and metal oxides to be deposited as uniform thin films, and the homogeneous binding of catalytic species, from isolated atoms and subnanometric clusters to integrated nanoparticles, of substrates with complex geometries or porous supports [Cheng et al., 2016; Lifeng, 2017]. Compared to other conventional methods, the ALD technique is appreciated for its versatility, simplicity and the possibility of obtaining small particles with uniform distribution over large areas, including in porous structures. This is possible due to the self-limiting nature of the process, offering a unique control over deposition, thus favoring the formation of homogeneous layers, and functionalization at moderate temperatures also supports the use of materials intended for operation at high temperatures [Nimalan and Begam, 2024].

This paper presents an analysis of the latest advances in platinum-based electrocatalysts (Pt) obtained by ALD for PEMFC, which aim to transition from fundamentals to performance and durability. The objective of this study is to highlight the importance of the ALD method in the synthesis and evolution of Pt electrocatalysts intended for ORR. The level of control of this technique allows the design of advanced electrodes and the optimization of the amount of Pt, with a direct impact on catalytic activity and stability under relevant operating conditions. By comparing the results from the literature with current research directions, this study provides insight into design strategies that can reconcile performance and sustainability requirements, while outlining directions for future optimizations of ALD-prepared Pt electrocatalysts within hydrogen-based energy technologies.

2. PLATINUM PREPARED BY ALD TECHNIQUE

ALD represents a gas-phase deposition procedure based on sequential and self-limiting surface reactions, allowing the controlled growth for thin films and nanostructures having an accuracy close to the atomic scale. In contrast to continuous processes (CVD), ALD separates the overall reaction into successive "half-reactions", so that deposition is dictated predominantly by surface chemistry and the saturation phenomenon of reactive sites, which leads to high uniformity, conformality and repeatability even on substrates that have a high aspect ratio and porous substrates [George, 2010].

2.1. The principle of the ALD technique

Therefore, ALD operates through this sequences of surface half-reactions, and a complete cycle typically includes: (i) the introduction of the first precursor into the reactor, followed by its chemisorption on the reactive sites of the substrate until saturation is reached (self-limitation); (ii) purging/evacuation of the reaction chamber with inert gas (or vacuum) for the removal of the non-adsorbed precursor and by-products; (iii) introduction of the co-reactant or second precursor (oxidizer or plasma), which reacts with the adsorbed species, completes the chemical transformation to the desired phase and regenerates the reactive groups on the surface for the next cycle; (iv) a second purge/discharge stage, aimed at removing the residual co-reactant and the by-products formed, restoring clean conditions for the next cycle. Repeating these cycles a controlled number of times allows fine adjustment of the thickness or catalytic charge, usually quantified by "growth per cycle" (GPC), and by controlling the nucleation and density of reactive sites can orient the deposition either to conformal ultrathin films or to the formation of well-dispersed clusters and nanoparticles [George, 2010; Johnson et al., 2014]. The ALD process is schematically illustrated in Figure 2.

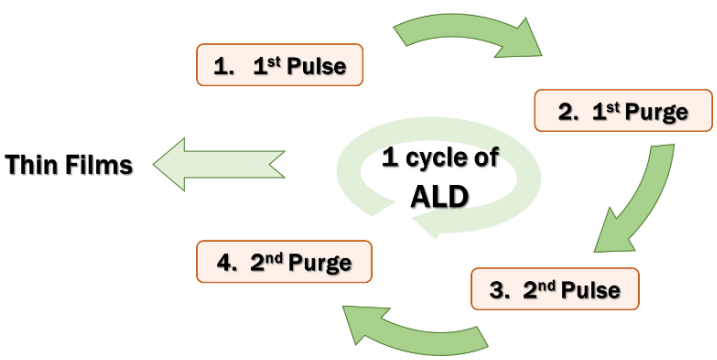

Figure 2. The ALD principle.

2.2. Platinum Precursors for ALD: Fundamental Considerations

The selection of precursors is a determining element in the design of an ALD process, as it directly regulates the temperature window, self-limitation of reactions, purity of layers and reproducibility of growth. In general terms, a suitable precursor for ALD must exhibit (i) sufficient volatility and vapour pressure compatible with transport in the reactor, (ii) gas-phase thermal stability, (iii) selective reactivity to surface sites so that adsorption is self-limiting, (iv) volatile by-products that can be efficiently removed by purging, and (v) minimal tendency to introduce residual impurities (C, O, N, halogens) in the film and non-corrosive to both substrate and materials in the reactor. In practice, these criteria imply a trade-off between volatility, stability and reactivity, and ligand chemistry (type and energy of bond breakage) becomes critical for obtaining clean and controllable deposits [Hagen et al., 2019].

In the case of platinum, the evolution of precursors used in ALD reflects three main objectives: (1) to achieve robust and reproducible chemistry, (2) to reduce the deposition temperature (important for compatibility with sensitive substrates and for nanostructure control), and (3) to control nucleation and growth to nanoparticles/clusters, not just continuous films [Ramos et al., 2013].

The first major and dominant stage in the literature is associated with the organometallic precursor (methylcyclopentadienyl)trimethylplatinum (IV), $MeCpPtMe_3$, classically used with O_2 as a reactant. Early studies demonstrated Pt depositions with good uniformity at typical temperatures on the order of 300°C, establishing $MeCpPtMe_3$ as the reference precursor for thermal processes [Aaltonen et al., 2003].

In order to overcome the limitations imposed by the reactivity of the oxidant and to reduce the process temperature, the use of pure oxygen instead of air was attempted. The use of high purity oxygen and the optimization of exposure conditions lowered the lower limit of the thermal process, more precisely, Pt films were obtained at temperatures up to ~200°C for the $MeCpPtMe_3/O_2$ system [Aaltonen et al., 2004]. Subsequently, the use of a stronger oxidant, ozone (O_3), allowed metallic Pt to be deposited from $MeCpPtMe_3/O_3$ at significantly lower temperatures (reported up to ~100°C under certain conditions), with a process window of 100–300 °C, constant and uniform film growth and low impurity content [Dendooven et al., 2013].

In parallel with the optimization of the reactant in thermal mode, the transition to plasma-assisted ALD for Pt is explicitly documented in remote plasma ALD processes based on $MeCpPtMe_3 + O_2$ plasma. In this framework, it has been shown that the duration of exposure to O_2-plasma can direct deposition to Pt (short exposures) or PtO_x (longer exposures), within a reported temperature window of 100–300°C [Knoops et al., 2009]. Moreover, plasma-assisted ALD processes targeting Pt deposits at room temperature were tried, using $MeCpPtMe_3$ – O_2 plasma – H_2 pulse sequences (gas or plasma) that allow obtaining high purity Pt films, with a resistivity of 18–24 μΩ·cm. The authors show that the introduction of H_2 pulsations is essential for the reduction of PtO_x that tends to form at low temperatures, thus maintaining the metallic character of the deposition [Mackus et al., 2013].

Over time, this combination of $MeCpPtMe_3/O_2$ has been studied in much more detail, including by *in-situ* techniques, to elucidate the mechanism of surface reactions involved in the deposition of platinum by ALD. Therefore, the thermal process based on alternating exposures to $MeCpPtMe_3$ and O_2 is considered a model system used as a benchmark in the analysis of ALD processes for other noble metals [Vandalon et al., 2022]. Figure 3 illustrates the detailed mechanism.

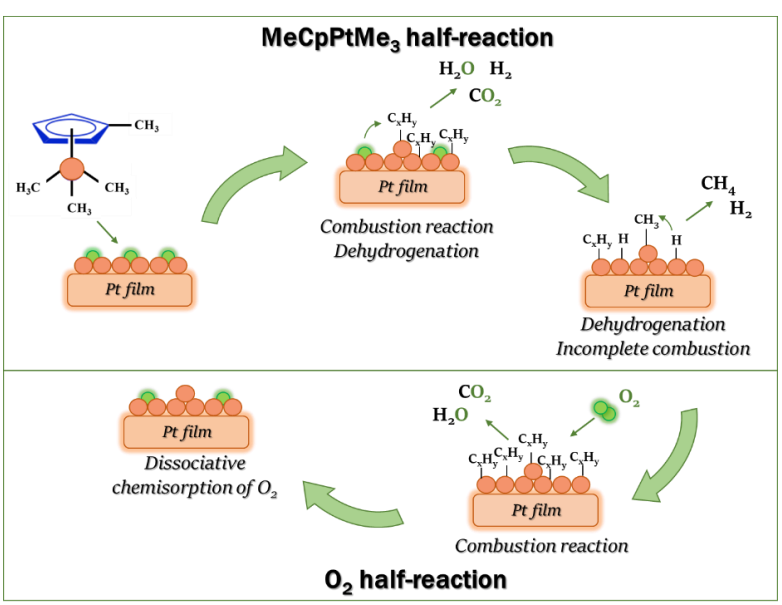

Figure 3. Surface reactions mechanism in thermal Pt-ALD process using $MeCpPtMe_3/O_2$ system.

A second direction of evolution focused on the alternative of β-diketone precursors, in particular $Pt(acac)_2$ (acac = acetylacetone), motivated by cost and availability, but also by the possibility of accessing controlled PtO_x deposits. By combining $Pt(acac)_2/O_3$, it has been reported that both Pt oxide films can be obtained at lower temperatures (120–130°C) and metallic Pt at higher temperatures (≥140°C), highlighting the control of chemical status by temperature. The authors point out that the lower temperature limit for oxide deposition is determined by the sublimation temperature of $Pt(acac)_2$, which has direct implications for the process window [Hämäläinen et al., 2008].

A third line, explicitly oriented towards nanoparticles and "particle ALD", often uses fluorinated precursors of the type $Pt(hfac)_2$ (hfac = hexafluoroacetylacetonate) together with a reducing agent such as formalin, reported to be effective for the growth of nanoparticles on oxide carriers such as TiO_2/WO_x. The role of the ligand in site blocking and nucleation control is highlighted: hfac ligands can induce longer incubation periods and alter the initial density of nuclei, which allows to adjust nanoparticles size by varying the cycles number, but requires increased attention on surface cleaning and residue removal [Anderson et al., 2014].

More recently, the evolution of Pt precursors has also included engineered precursors designed to overcome classical limitations (e.g., growth rate, electrical properties, impurity control) and expand the temperature range. An example is the precursor DDAP [dimethyl (N,N-dimethyl-3-butene-1-amine-N) platinum], used with O_2, reported to obtain Pt films with relatively high growth rate and low resistivity, suggesting that optimizing ligand architecture can reduce the constraints associated with "classical" precursors (including kinetic limitations or incomplete ligand cleaning) [Lee et al., 2019].

2.3. Bimetallic and Pt alloy ORR electrocatalysts prepared by ALD: Performance-durability potential in PEMFCs

Bimetallic catalysts and Pt–M alloys (M = transition metals) are considered a central direction for the PEMFC cathode, as they can increase the activity of ORR while reducing the Pt charge and improving durability, by controlling the composition and nanostructural architecture [Wu et al., 2025]. Bimetallic nanoparticles can have distinct catalytic properties from component metals, due to synergistic effects, and oxide or carbon supported variants are widely used in catalysis and electrocatalysis, including in fuel cell applications. Traditional synthesis methods for bimetallic nanoparticles often offer limited control over composition and structure, and can produce mixtures of mono- and bimetallic particles with variable compositions [Lu et al., 2024].

In this context, ALD confers a major advantage in the design of bimetallic catalysts and alloys, as it allows rigorous control of metal particle formation by alternating deposition sequences and fine-tuning of nucleation and growth processes at surface level. At the same time, this approach facilitates the adjustment of the spatial distribution and composition of the active phases, which are essential for optimizing catalytic performance [Lu et al., 2024; Sairanen et al., 2014].

One of the first studies to explicitly demonstrate the use of ALD to prepare carbon-supported catalysts, both monometallic (Pt) and bimetallic Pt-Co is that of Sairanen et al., in which small metallic particles showing a narrow size distribution and high dispersion were produced. In same study, control of metal positioning on the surface is achieved by alternating ALD cycles, and the formation of bimetallic Pt-Co particles on the support is noticed as a result of this strategy. The catalysts thus synthesized are reported to show activity for both oxygen reduction and methanol oxidation in an acidic media, reactions relevant to fuel cells, including PEMFC, where ORR constitutes the kinetic limiting reaction. Furthermore, the authors show that durability in electrochemical oxidation can be improved by changing the sequence of metal

cycles, and deposition of an ALD cycle of Pt after Co deposition favors the durability of the catalyst in the methanol oxidation reaction [Sairanen et al., 2014].

In another study, ALD is used for TiO_2 and Pt deposition on carbon-based substrates, followed by heat treatment in H_2 to form a Pt_3Ti alloy/intermetallic. In this case, the ALD technique provides close contact with Pt–TiO_2 and fine control of the amount deposited, and the heat treatment produces Pt_3Ti with Pt-enriched surface, which combines the high activity of Pt with the better thermodynamic stability of the Pt–Ti phase. From the electrochemical analyses, the authors report ORR performance for Pt_3Ti a mass activity (MA) of 1.84 A mg/Pt and a specific activity (SA) of 5.99 mA cm^{-2} Pt, values much higher than commercial Pt/C and even higher than polycrystalline Pt. From the accelerated durability/stress test, of 10,000 cycles (0.6–1.0 V vs RHE in O_2-saturated 0.1 M $HClO_4$) it is observed that Pt_3Ti keeps its ORR and electrochemical surface area (ECSA) almost unchanged, while commercial Pt/C loses significantly, for example, reported losses of 21% in kinetic current density and 14% in active electrochemical area [Kim et al., 2022].

However, the activity advantage of Pt–M alloys is often accompanied by a vulnerability to degradation in an acidic environment, specific to PEMFC: the less noble metal can undergo leaching/dissolution under electrochemical conditions, which destabilizes the "skeleton" of the alloy. That is why a recurring direction in design is the shift from alloys with random structures to core–shell / Pt-skin / ordered (intermetallic) architectures, in which a surface layer rich in Pt can protect the M component and simultaneously preserve, or even amplify, the effects of electronic activation [Zhang et al., 2021].

In this regard, PtCu@Pt/C catalysts were obtained by combining a wet technique (for $PtCu_3$/C) with ALD, (for Pt coating) and the shell thickness was controlled by changing the number of cycles from 1 to 6, and the authors state that four ALD cycles deposit approximately one atomic layer of Pt on the surface of the $PtCu_3$ nucleus. Experimentally, $PtCu_3@Pt_{ALD\text{-}4}$/C exhibited the MA and SA of 3.2 and 2.6 times higher than Pt/C respectively and also having superior durability compared to $PtCu_3$/C and Pt/C [Zhao et al., 2025].

Lee et al. developed PtNi alloy electrocatalysts for ORR by fluidized bed reactor–ALD (FBR-ALD), using a super-cycle strategy to control the composition ($Pt_{75}Ni_{25}$–$Pt_{85}Ni_{15}$), followed by heat treatment in H_2 at 700°C to promote the formation of an alloy and a Pt-skin Pt-rich surface over a phase close to Pt_3Ni. The ORR activity was electrochemically evaluated by rotating disk electrode (RDE) analyses in 0.1 M $HClO_4$, reporting comparable or even higher values of ECSA, and the best values of MA and SA at 0.9 V vs RHE, compared to commercial Pt. Practical relevance in PEMFC was also demonstrated by membrane electrode assembly (MEA) tests at 70°C, where $Pt_{78}Ni_{22}$ provided higher current density at 0.6 V and higher MA at 0.9 V than commercial Pt and commercial Pt_3Ni, confirming RDE analyses. Durability was supported by 10,000-cycle accelerated durability test (ADT), in which $Pt_{78}Ni_{22}$ retained a significantly higher fraction of ECSA compared to commercial benchmarks, suggesting superior structural stabilization for the architecture obtained by ALD [Lee et al., 2022].

3. CONCLUSIONS AND DISCUSSION

Overall, this paper highlights that ALD represents a robust and flexible platform for the design of platinum-based electrocatalysts for PEMFCs, as it provides atomic-scale control over the charge, thickness, composition and morphology of the active phase, with a direct impact on Pt utilization, ORR activity and stability over time. The integration of process fundamentals with electrochemical results highlights the potential of ALD to generate high-performance architectures such as well-dispersed nanoparticles, ultra-thin films, core-shell and alloy/bimetallic structures, which are able to support the transition from performance to

durability. However, implementation at the relevant scale remains limited by challenges such as scaling on porous supports, uniformity of deposition in structures with complex mass transport, control of precursor residues and structural/compositional stability under degradation (coalescence, dissolution, dealignment, support corrosion). As future perspectives, optimization of the ALD technique on particles and layered porous supports, standardized correlation between RDE and MEA tests, and development of Pt-alloy/intermetallic and core–shell systems resistant to degradation are required to accelerate the validation and sustainable adoption of ALD catalysts in PEMFCs.

Funding: This work has been funded by the Core Program within the National Research Development and Innovation Plan 2022-2027, carried out with the support of Ministry of Research, Innovation, Digitization from Romania by the National Plan of R & D, project no. 20N/2023, PN 23 15 01 03.

Conflict of Interest Statement: The authors declare no conflicts of interest associated with this manuscript.

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