

Synthesis and properties of polyfunctional coatings on aluminum and titanium alloys

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This work presents the synthesis of highly active electrode materials for the electrolytic production of hydrogen from aqueous solutions, which can also serve as photocatalysts. Composite coatings produced by plasma-electrolytic oxidation on aluminum and titanium alloy substrates, doped with vanadium and tungsten compounds, were investigated. The electrolysis processes of aqueous solutions using these composites as electrode materials were analyzed. Linear voltammetry was applied to determine the Tafel equation constants for the systems $\text{WO}_3 - \text{V}_2\text{O}_5 - \text{Al}_2\text{O}_3 / \text{Al}$ and $\text{WO}_3 - \text{V}_2\text{O}_5 - \text{TiO}_2 / \text{Ti}$, which are proposed as electrode materials for hydrogen evolution reactions. The study found that the dopant content, the metallic substrate characteristics, and surface morphology strongly influence the coatings' functional properties. The Tafel coefficients a and b indicate a high level of electrocatalytic activity for the synthesized coatings, confirming their suitability as electrode materials for electrolytic hydrogen production. A comparison of photocatalytic activity in the methyl orange degradation reaction revealed higher activity of the aluminum-based coating than the titanium-based coating, highlighting the relevance of these materials for ecological technologies. The presence of nonstoichiometric oxides, compositional variability, morphological differences, and the developed surface area accounts for their exceptionally high electrocatalytic activity and, therefore, their strong potential for functional applications.

Keywords: plasma electrolytic oxidation, composite coatings, metallic substrates, vanadium and tungsten oxides, photocatalysis, electrocatalysis, hydrogen evolution reaction, degradation.

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1. Introduction

Plasma electrolytic oxidation (PEO) is a well-established surface treatment technique for the formation of oxide coatings on valve metals such as Al, Ti, Zr, Ta, and Nb. This method enables the synthesis of multicomponent surface layers incorporating transition metal oxides, thereby offering broad possibilities for tailoring functional properties [1]. A key characteristic of the PEO process is the continuous and dynamic restructuring of the coating across the entire metal surface. Under PEO anodizing conditions, dielectric breakdown events give rise to numerous transient microdischarges that are repeatedly gener-

ated over the surface of the substrate. These localized discharge channels are associated with extreme temperature gradients, reaching approximately 2000–10 000 K, which facilitate the transformation of initially amorphous oxide phases into crystalline structures. As a result, PEO-derived coatings exhibit highly tunable morphologies, strong interfacial adhesion, elevated microhardness, and excellent resistance to wear and corrosion. In recent years, significant research attention has focused on the photocatalytic performance of modified coatings based on titanium (IV) and aluminum oxides. Although alternative semiconductor materials, including $\alpha\text{-Fe}_2\text{O}_3$, CdS, SnO₂, and SrTiO₃, have been explored for photocatalytic ap-

plications, their widespread implementation is often constrained by toxicity and environmental concerns. In contrast, titanium (IV) and aluminum oxides are non-toxic, chemically stable, cost-effective, and readily produced on an industrial scale, making them particularly attractive for sustainable and large-scale technological applications.

Photocatalytic performance of semiconductor materials can be enhanced through several approaches: doping with transition metals (Pt, Pd, Au, Ni, Cr, Mo, Nb, W, Mn, Fe, Ce, Co, etc.), modifying surfaces with polymers or dyes, or employing mixed binary and ternary metal oxide systems [2]. A critical factor in improving coating performance is the incorporation of *d*- and *p*-elements into the oxide matrix [3]. In this regard, particular attention is directed to electrode materials based on vanadium and tungsten oxides [4–7]. These systems hold great promise for diverse applications, including heterogeneous catalysis, photocatalysis, semiconductor devices, and as electrode materials for electrolytic hydrogen and oxygen evolution reactions [8–10]. This context has defined the focus of the present study. The purpose of this study is to investigate the properties of hetero-oxide coatings formed on titanium and aluminum substrates, with the aim of expanding their use as photocatalytic and electrode materials. This requires examining the positive effects of incorporating complex vanadium and tungsten oxides into titanium and aluminum oxide films.

It is well established that the crystal lattices of tungsten and vanadium oxides often deviate from ideal stoichiometry, resulting in oxygen deficiencies that significantly enhance their electrochemical activity [11]. The high concentration of oxygen vacancies, together with the existence of multiple structural polymorphs—including cubic, hexagonal, orthorhombic, and monoclinic phases—suggests that coatings based on these oxides can exhibit pronounced catalytic and photocatalytic behavior. The efficiency of photocatalytic processes is strongly influenced by several factors, such as the specific surface area of the catalyst, the nature and density of surface-active sites, and the spatial localization and lifetime of photogenerated charge carriers. These properties are, in turn, governed by the crystalline structure of the material, the synthesis route employed, and any subsequent post-treatment of the active layer [12,13].

2. Experimental

PEO coatings were prepared on titanium alloy VT1-0 and aluminum A0 (Al – 99 wt.%). Sample

preparation for anodizing included degreasing in a sodium carbonate solution at 40–60 °C, followed by etching: titanium samples in a nitric acid–hydrofluoric acid mixture (1:3) and aluminum samples in nitric acid at room temperature. Samples were then rinsed in running and distilled water.

Previous studies [14] indicated that coatings formed in potassium pyrophosphate solutions with concentrations ≥ 0.5 mol/dm³ exhibit optimal functional properties. Therefore, further experiments were carried out using electrolytes with constant pyrophosphate content and varying Na₂WO₄ (0.05–0.15 M) and NH₄VO₃ (0.1–0.15 M) concentrations. Electrolytes were prepared by dissolving tungsten and vanadium salts in distilled water, mixing them, and gradually adding the mixture to the base pyrophosphate solution under stirring. The pH was monitored with a pH-meter pH-150M and glass electrode ESL-6307. Due to vanadate's low solubility, dissolution was performed at 40–50 °C. At pH 8–10, sodium tungstate and ammonium metavanadate are fully hydrolyzed, with the [HVO₄]²⁻ anion in equilibrium with the pervanadate ion [V₂O₇]⁴⁻. Under PEO conditions, high temperatures induce transformations of these anions into V₂O₅ and WO₃, which are incorporated into the coating surface layers [15].

Oxidation was performed in a single stage at current densities of 10–40 A/dm² and a maximum voltage $U_{\text{phase}} = 80$ –140 V for 10–20 min, with continuous stirring and electrolyte cooling. A B5-50 DC power supply was used for polarization. After anodizing, samples were rinsed with distilled water and dried at room temperature.

The chemical composition of the coatings was determined by X-ray fluorescence using a portable “SPRUT” spectrometer, with a relative standard deviation of 10⁻³–10⁻². Measurements were taken at a minimum of three points per sample and averaged, with an error of ± 1 wt.%. For verification, energy-dispersive X-ray spectroscopy was performed using an Oxford INCA Energy 350 electron probe micro-analyzer (15 keV electron beam). Surface morphology was examined using a ZEISS EVO 40XVP scanning electron microscope (G.V. Karpenko Institute of Physics, NASU). Secondary electron imaging allowed high-resolution and high-contrast visualization at 100–5000 \times magnification. Images were processed with SmartSEM software [16].

Photocatalytic activity was tested via the model oxidation of methyl orange (MO) in a 50 mL thermostatic glass photoreactor with magnetic stirring (100 rpm) at room temperature under air. Initial MO concentrations ranged from (0.02–2.0) \times

10^{-4} mol/dm³. Samples coated with hetero-oxide films were irradiated with a UV lamp DRT-125-1 (230–400 nm, 125 W) placed 5 cm above the solution. Solutions were pre-equilibrated in the dark for 60 min to establish adsorption equilibrium, then irradiated for 80 min. MO concentration was measured every 10 min using a KFK-2 spectrophotometer at $\lambda = 490$ nm. The degree of MO decomposition was calculated as $(C_0 - C_t)/C_0$, where C_t is the current concentration and C_0 is the initial concentration.

Electrocatalytic properties were studied by linear voltammetry in a neutral model medium (1 M Na₂SO₄, pH 7) using a MTech PGP-550S potentiostat–galvanostat in a thermostated three-electrode cell. A platinum mesh was used as the auxiliary electrode, and a saturated Ag/AgCl electrode (EVL-1M1) as the reference. All potentials were converted to the standard hydrogen electrode (SHE) scale. Electrocatalytic activity in hydrogen evolution was characterized by the exchange current density $j_0(H_2)$, determined from linearized Tafel plots.

3. Results and discussion

Previous studies have demonstrated that the incorporation of d-block elements into metal oxide matrices significantly enhances the catalytic performance of hetero-oxide coatings [17–19]. In the present work, the observed synergistic interaction between tungsten and vanadium oxides in improving the photo- and electrocatalytic activity of oxide films motivated a detailed investigation into the influence of electrolysis conditions, electrolyte composition, and substrate material on these properties.

The formation mechanisms of the composite oxides can be rationalized by considering the atomic radii of the constituent elements: titanium (0.068 nm), aluminum (0.143 nm), vanadium (0.052 nm), and tungsten (0.135 nm). Owing to its relatively small atomic radius, vanadium can readily incorporate into the TiO₂ lattice, partially substituting titanium ions within the oxide structure [20–23]. In contrast, the larger atomic radius of tungsten restricts its incorpo-

ration into the bulk oxide lattice, leading to its preferential localization in the near-surface region of the coating.

The presence of tungsten oxides in the surface layer, which are intrinsically less catalytically active, facilitates the formation of vanadium-rich oligomeric domains and intermediate oligomeric structures. These species enhance catalytic performance primarily through structural rather than electronic effects. As a result, tungsten oxides are stabilized in the surface layer in the form of W_xO_y, enabling the formation of multicomponent heterostructured systems such as WO₃–V₂O₅–TiO₂/Ti and WO₃–V₂O₅–Al₂O₃/Al. Previous investigations [24–27] have shown that the emergence of highly reactive surface sites arises from the combined effect of tungsten and vanadium oxides, leading to the generation of surface centers with enhanced catalytic activity.

During PEO of aluminum, high-temperature regions form at breakdown sites, where γ -Al₂O₃ transforms into α -Al₂O₃ (corundum) with a stable cubic lattice, increasing the spark-discharge voltage. Electrolyte penetration into the aluminum matrix at these sites is slower than in titanium, yet the resulting coatings still contain vanadium and tungsten oxides [28, 29].

The formation of complex oxides differs between the two metals, likely due to the differing electrical resistivities of the oxide layers: Al₂O₃ films have resistivity in the range 10^{12} – 10^{18} Ω·cm, while titanium films are 10^{10} – 10^{12} Ω·cm. At temperatures above 400 °C, Al₂O₃ resistivity decreases by several orders of magnitude [30–38]. PEO monitoring shows that discharge intensity is lower on aluminum samples than on titanium. For titanium, sparking begins at $U_{\text{spark}} = 60$ –70 V within 30 s, and the transition to the microarc regime occurs faster at lower voltages (105–120 V) than for aluminum, where $U_{\text{spark}} = 80$ –100 V after 60–90 s.

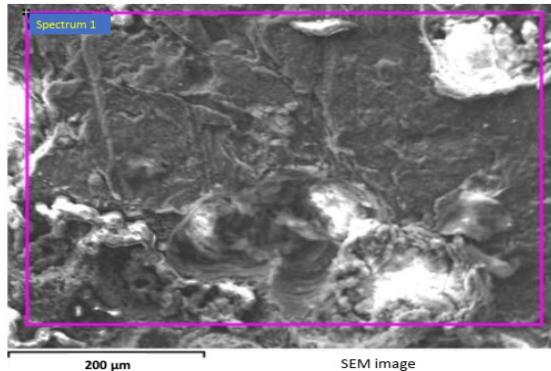
The presence of α -Al₂O₃ complicates the incorporation of electrolyte components into the oxide film, resulting in a lower amount of incorporated elements (Table 1) and influencing the surface morphology of the coatings (Figures 1 and 2).

Table 1 – Composition of hetero-oxide coatings on aluminum and titanium substrates.

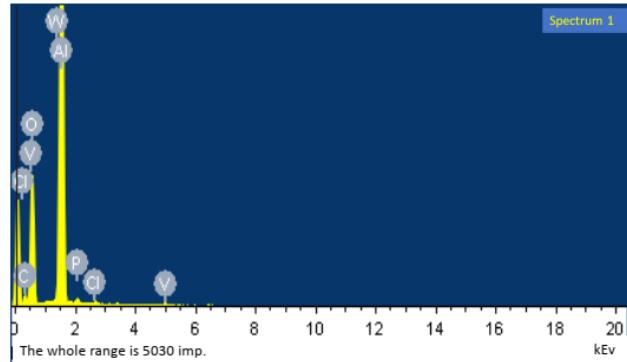
Composition of the oxide coating	Elemental mass fraction, %						
	V	W	Al	Ti	O	P	K
WO ₃ –V ₂ O ₅ /Al ₂ O ₃	0,29	0,12,	46,35	-	52,65	0,59	-
WO ₃ –V ₂ O ₅ /TiO ₂	1,51	2,83	-	27,59	60,99	6,76	0,32

Figure 1 shows that the surface of the $\text{WO}_3 - \text{V}_2\text{O}_5 - \text{Al}_2\text{O}_3 / \text{Al}$ composite is uneven, with crater-like inclusions. In contrast, increasing the tungsten

content in the $\text{WO}_3 - \text{V}_2\text{O}_5 - \text{TiO}_2 / \text{Ti}$ coating produces a uniform, fine-grained structure without large agglomerates (Figure 2).

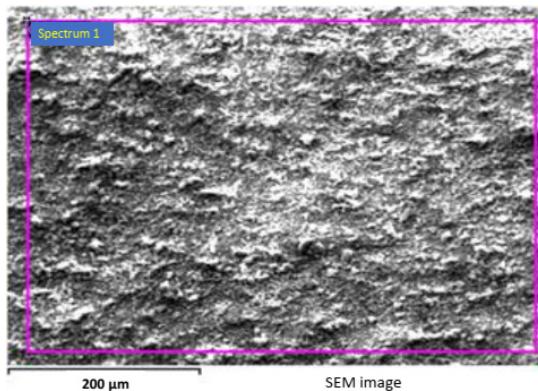


a)

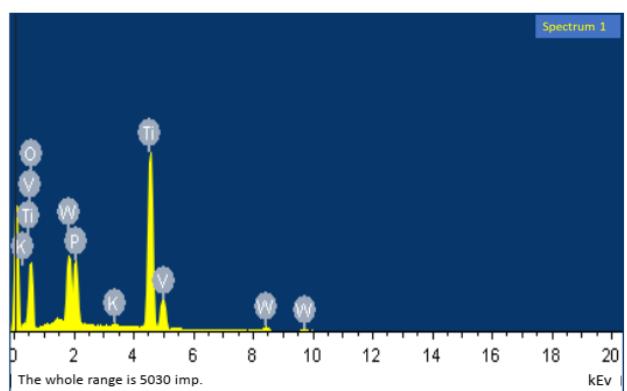


b)

Figure 1 – Morphology (a) and X-ray spectrum (b) of the composite surface on an aluminum substrate.



a)



b)

Figure 2 – Morphology (a) and X-ray spectrum (b) of the composite surface on a titanium substrate.

The photocatalytic performance of the hetero-oxide coatings was assessed by tracking the temporal degradation of methyl orange (MO) in aqueous solutions. The results of these photocatalytic activity tests are presented as histograms in Figure 3.

The results indicate that the ratio of dopant metals has a significant impact on the photocatalytic activity of hetero-oxide coatings on aluminum substrates. Increasing the vanadium content in the electrolyte, however, reduces the photocatalytic activity of the hetero-oxide coatings on both aluminum and titanium. This effect can be explained by the sequence

of oxide layer formation on the metal surfaces. V_2O_5 forms much more rapidly than WO_3 during oxidation reactions [22]. At high VO_3^- concentrations in the electrolyte, the accelerated formation of V_2O_5 hinders the uniform deposition of WO_3 on the coating surface, while WO_3 predominantly forms inside pores, reducing the exposed catalytically active surface area. Therefore, the optimal dopant ratio is 1:1.

The time-dependent degradation profiles of MO for mono-oxide coatings and hetero-oxide coatings prepared from electrolytes with a V:W ratio of 1:1 are shown in Figure 4.

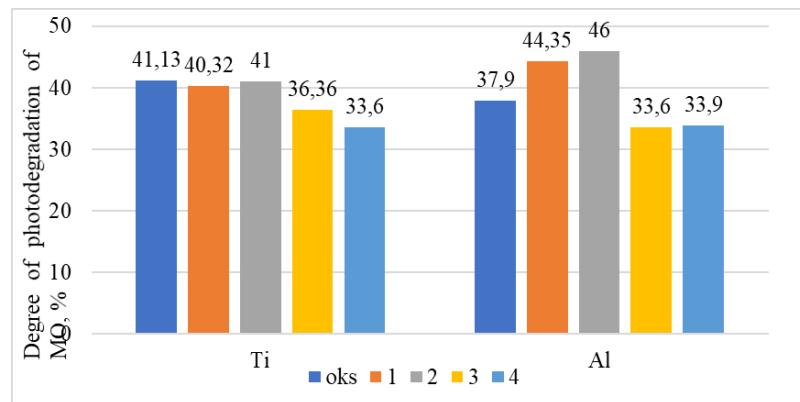


Figure 3 – Photocatalytic properties of coatings on aluminum and titanium substrates: mono-oxide coatings (ox) and hetero-oxide coatings obtained by PEO in electrolytes with V/W ratios of 1:3 (1), 1:1 (2), 2:1 (3), and 3:1 (4).

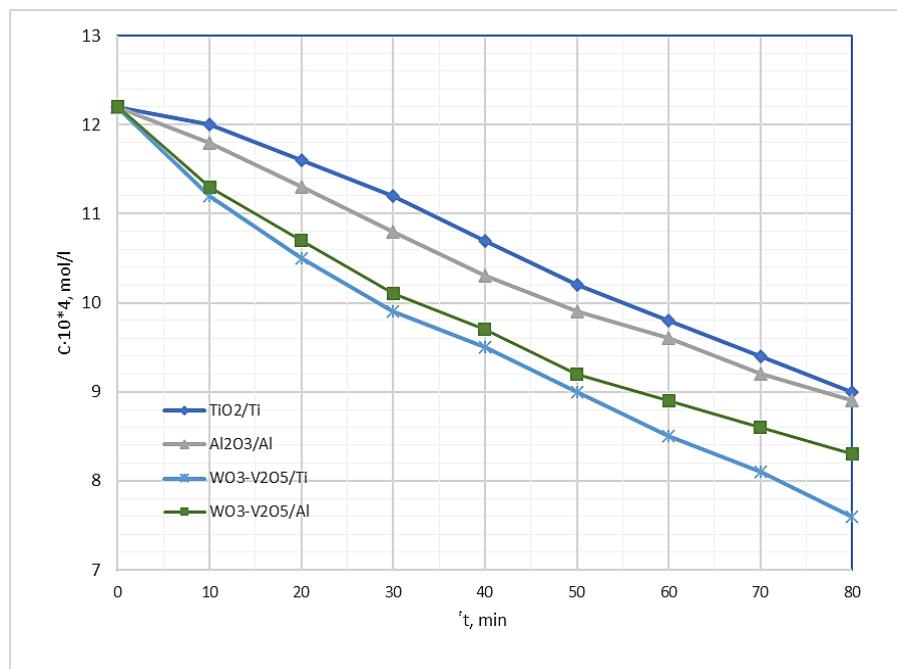


Figure 4 – Time-dependent profiles of photocatalytic degradation of MO in contact with mono- and hetero-oxide coatings (V:W ratio = 1:1).

Comparison of the coatings in the MO degradation reaction shows that doped hetero-oxide coatings exhibit significantly higher photocatalytic activity than mono-oxide coatings, demonstrating their potential for eco-technological applications.

The electrocatalytic activity of the hetero-oxide coatings was assessed using kinetic parameters from a model hydrogen evolution reaction. The hydrogen reduction potential and polarization behavior were found to depend on the coating composition, the nature of the substrate metal, and the pH of the medium.

The presence of non-stoichiometric oxides, compositional variability, surface morphology, and the degree of surface development result in electrocatalytic properties of the hetero-oxide coatings that differ markedly from those of the pure metals (Figure 5). In a neutral medium, hydrogen evolution begins at -1.15 V on aluminum coatings and at -0.95 V on titanium coatings, indicating a reduction in hydrogen overpotential. The electrocatalytic activity of the coatings is characterized by the Tafel slope (b), determined from the linear portion of the Tafel plot (Figure 5, Table 2).

Lower b values indicate faster reaction kinetics. In this study, the observed b values differ from classical trends relating hydrogen evolution overpotential

to current density and electrode material, reflecting the unique electrocatalytic behavior of the hetero-oxide coatings.

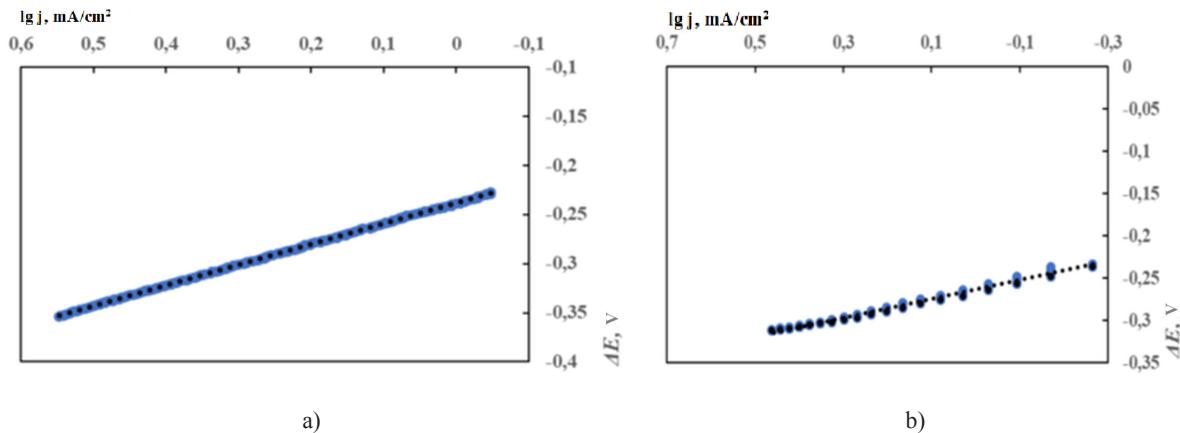


Figure 5 – Linear regions of the polarization curves for the hydrogen evolution reaction in Tafel coordinates on $\text{WO}_3 - \text{V}_2\text{O}_5 - \text{TiO}_2 / \text{Ti}$ (a) and $\text{WO}_3 - \text{V}_2\text{O}_5 - \text{Al}_2\text{O}_3 / \text{Al}$ (b) coatings in a neutral 1 M Na_2SO_4 solution. Scan rate: 2 mV/s.

Table 2 – Electrocatalytic parameters of the coatings for hydrogen evolution.

Electrode material	$-a$, B	$-b$, B
Pt	0,31	0,10
Ti	0,82	0,14
Al	0,64	0,14
$\text{WO}_3 - \text{V}_2\text{O}_5 - \text{Al}_2\text{O}_3 / \text{Al}$	0,26	0,11
$\text{WO}_3 - \text{V}_2\text{O}_5 - \text{TiO}_2 / \text{Ti}$	0,23	0,20

In multicomponent oxide systems, unlike pure metals, the Tafel slope (b) can exceed 0.12 V. In this study, b for the $\text{WO}_3 - \text{V}_2\text{O}_5 - \text{TiO}_2 / \text{Ti}$ composites was -0.20 V. For many metals, b is higher in alkaline solutions than in acidic media during hydrogen evolution; to avoid this effect, a neutral electrolyte was used.

The variation in b is influenced by several factors: the compositional diversity of non-stoichiometric oxides, differences in hydrogen adsorption on the oxide matrix (which can slow electrochemical desorption), and the formation of active centers from dopants (vanadium and tungsten). These factors complicate the hydrogen evolution mechanism on hetero-oxide surfaces and merit further investigation.

The Tafel constant a for $\text{WO}_3 - \text{V}_2\text{O}_5 - \text{Al}_2\text{O}_3 / \text{Al}$ was -0.26 V (Table 2), close to that of $\text{WO}_3 - \text{V}_2\text{O}_5 - \text{TiO}_2 / \text{Ti}$ (-0.23 V), and both values fall within the

range typical for catalytically active materials. The slight difference in a arises from variations in the hetero-oxide matrix and the tungsten and vanadium oxide content. A higher proportion of non-stoichiometric tungsten and vanadium oxides in titanium-based coatings further lowers a . Overall, these results indicate the exceptionally high electrocatalytic activity of the hetero-oxide composites in hydrogen evolution, confirming their strong potential for practical applications.

4. Conclusion

Highly active hetero-oxide photocatalytic coatings were successfully synthesized on aluminum- and titanium-based substrates via plasma electrolytic oxidation incorporating vanadium and tungsten spe-

cies. The resulting materials demonstrated excellent functional performance across multiple application domains.

Evaluation of photocatalytic activity using methyl orange degradation revealed significantly enhanced reaction rates, confirming the strong potential of these coatings for environmentally oriented and eco-technological applications.

Electrochemical studies of hydrogen evolution reactions showed that the synthesized hetero-oxide composites exhibit exceptionally high electrocata-

lytic activity, underscoring their promise as efficient electrode materials for electrolytic hydrogen production.

The functional properties of the coatings exhibited a strong dependence on the dopant content, the characteristics of the metallic matrix, and the surface morphology.

(iv) Analysis of the Tafel parameters further confirmed the superior electrocatalytic characteristics of the coatings, supporting their applicability in practical hydrogen generation systems.

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