

# NANODIAMOND ELECTRODES FOR HYDROGEN PRODUCTION BY ELECTROLYSIS

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## Abstract

Hydrogen production by water electrolysis is limited by the efficiency of current catalytic materials and the overall process cost. High overpotentials and expensive catalysts raise energy use and capital expenditures, constraining scale-up. Nanodiamond composite electrodes for the hydrogen evolution reaction were developed using two routes: a muffle furnace method and a bell jar chemical reactor. Formulations incorporated nanodiamond powder into a composite matrix with aluminum titanate, lithium chloride, boron, tantalum, and graphene, then were pressed into cylindrical pellets. Structural integrity and catalytic activity were tested in alkaline media prior to electrolysis. Volumetric gas collection was used to quantify hydrogen generation rates and benchmark against graphite and Pt controls. This study offers insights into the use of nanomaterials for hydrogen evolution, showing that nanodiamond electrodes exhibit strong catalytic response at low overpotential.

## Introduction

The transition to clean and sustainable energy sources is crucial for mitigating climate change, with hydrogen emerging as a promising solution due to its high energy potential, nearly three times greater than gasoline. Hydrogen is increasingly seen as a key energy source for electricity generation, transportation, and energy storage. Hydrogen fuel cells generate electricity by combining hydrogen and oxygen, producing water and heat as byproducts, enabling zero-emission vehicles, though adoption is hampered by high fuel cell costs and the limited availability of refueling stations, largely in California. Current hydrogen production methods, particularly for "green hydrogen," remain costly and inefficient. The main challenge lies in improving the efficiency of the electrolysis process, which depends heavily on the materials used in the electrodes; conventional materials have not yet achieved the desired balance between cost-effectiveness and performance.

One of the cleanest approaches to hydrogen production is electrolysis, in which water is split into hydrogen and oxygen with an electric current. The efficiency of this process largely depends on the electrode materials, and previous research has demonstrated that nanodiamonds have significant potential for hydrogen-related applications, including hydrogen storage. Building on the principle that materials efficient in hydrogen storage may also be effective as catalysts in its production, this research proposes that combining nanodiamonds with metals could enhance the efficiency of the electrolysis process. Recent work has highlighted the electrochemical properties of nanodiamonds and their effect on redox reactions: undoped detonation diamond nanoparticles can enhance redox reactions in solution. Nanodiamond electrodes exhibit a wide working potential window in aqueous solutions while maintaining a low and stable background current; due to these properties, nanodiamonds are considered a promising support material for electrocatalysts. The selection of suitable metals for improving efficiency will be guided by a base matrix, with metals added incrementally based on chemical and physical properties, focusing on transition metals. Experimental tests will determine performance in terms of efficiency and durability; several candidates show promise, including boron, lithium, and tantalum.

## Objective

The general objective of this research is to develop high-performance electrodes by combining nanodiamonds with selected metals to enhance the efficiency of the electrolysis process. By optimizing this combination, the study aims to make green hydrogen production more economical and sustainable, ultimately contributing to the broader adoption of hydrogen as a clean energy source.

## Methodology

For electrode fabrication, several powder compositions were prepared, as summarized in the following table.

Material (grams)	Electrode Number			
	1	2	3	4
Nanodiamond	2.22	2.22	2.22	2.22
Lithium Chloride	7.06	7.06	7.06	0
Aluminum Titanate	4.22	4.22	4.22	4.22
Boron	2.00	2.00	2.00	2.00
Tantalum	4.22	4.22	4.22	4.22
Graphene	0	1.00	0	0

One or several drops of polyvinyl butyral (PVB) diluted in 1 ml of an organic solvent (ethanol) was added to each powder blend to impart structural integrity during pellet molding. The mixture was then pressed into cylindrical pellets at 3,000 psi and dried for 48 hours at ambient temperature.



Figure 1. First Pellet Mixture

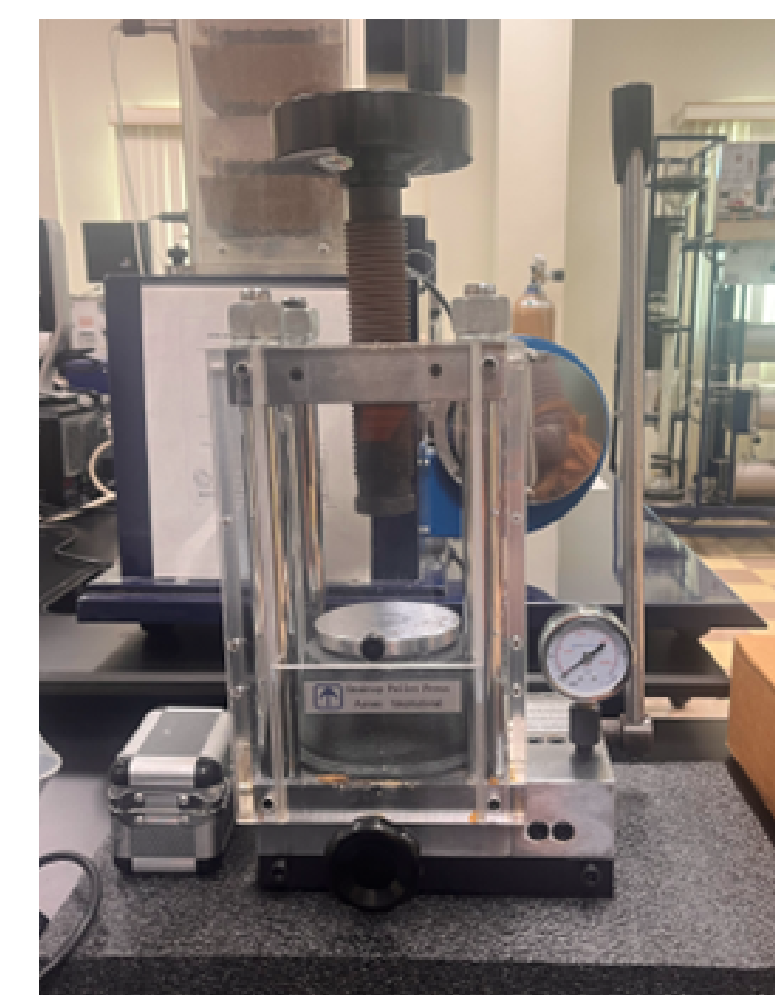


Figure 2. Pellet Press



Figure 3. First pellets

The first and second pellet mixtures were treated in a Muffle Furnace at 610°C for 4 hours. The fourth pellet mixture was treated in a Bell Jar Chemical Reactor at 600°C for 4 hours under an argon atmosphere to prevent or reduce oxidation. Argon, as an inert gas, does not participate in the reaction and effectively displaces oxygen.



Figure 4. Muffle Furnace

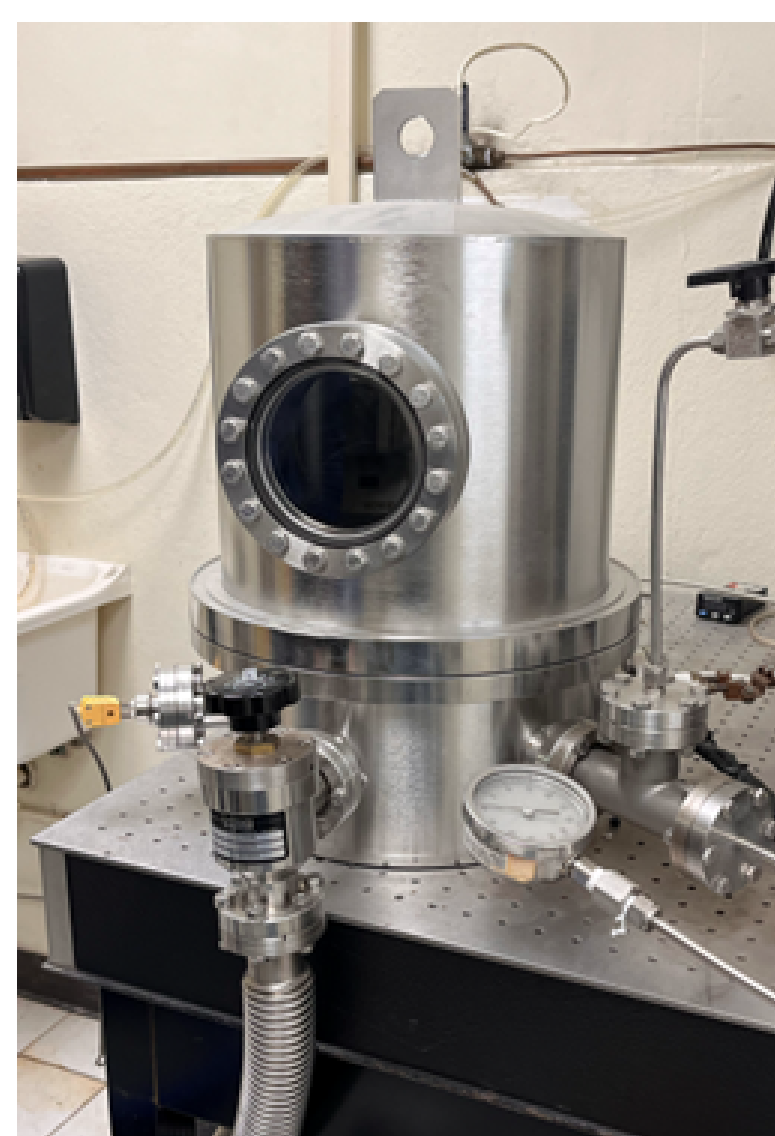


Figure 5. Bell Jar Chemical Reactor



Figure 6. Electrolysis

Following heat treatment to promote sintering, pellets were immersed in 1M potassium hydroxide (KOH) to assess structural integrity under aggressive alkaline conditions. Electrolysis tests were subsequently performed.

Graphite and platinum electrodes were employed as performance benchmarks for the nanodiamond electrodes.

## Results

Electrode #1, treated in a muffle furnace under an oxygen atmosphere, exhibited low catalytic activity and underwent surface oxidation that compromised its structural integrity in 1M KOH, causing the pellets to fracture and preventing electrolysis testing. Electrode #2, which was also muffle-treated but without planetary ball mill processing to minimize oxidation exposure, withstood immersion in KOH but deformed due to oxidation and showed low hydrogen production rates. Electrode #3 was discarded prior to testing because it continued to absorb moisture from the environment and could not be pressed into pellets, likely due to lithium chloride not being treated with boron in the planetary ball mill. Electrode #4, treated in an argon atmosphere reactor to prevent oxidation, exhibited good catalytic activity, maintained structural stability in KOH and achieved a hydrogen production rate of 1.210 ml/min, with an average lap time of 5:47 min/ml. The same electrode was then intentionally oxidized by immersion in hydrogen peroxide for 48 hours, resulting in a performance decline to 0.1720 ml/min and an average lap time of 6:47 min/ml.

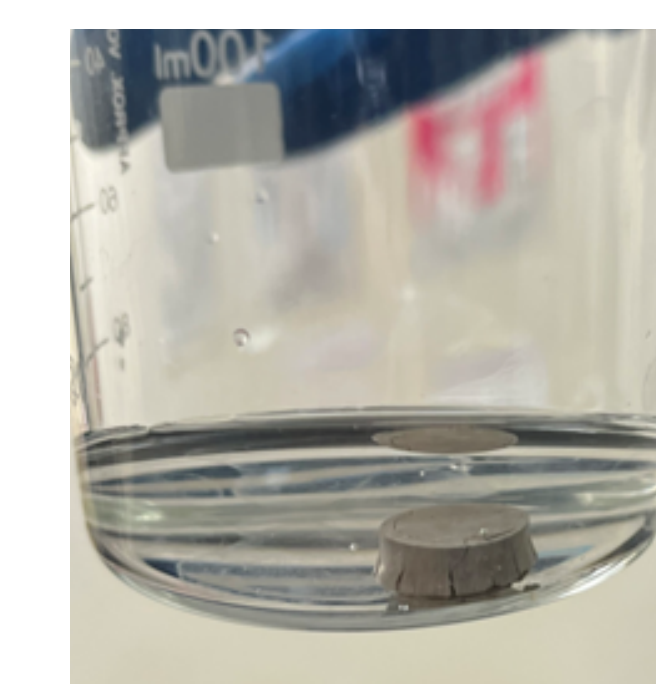


Figure 7. Electrode #1 - Catalytic Activity

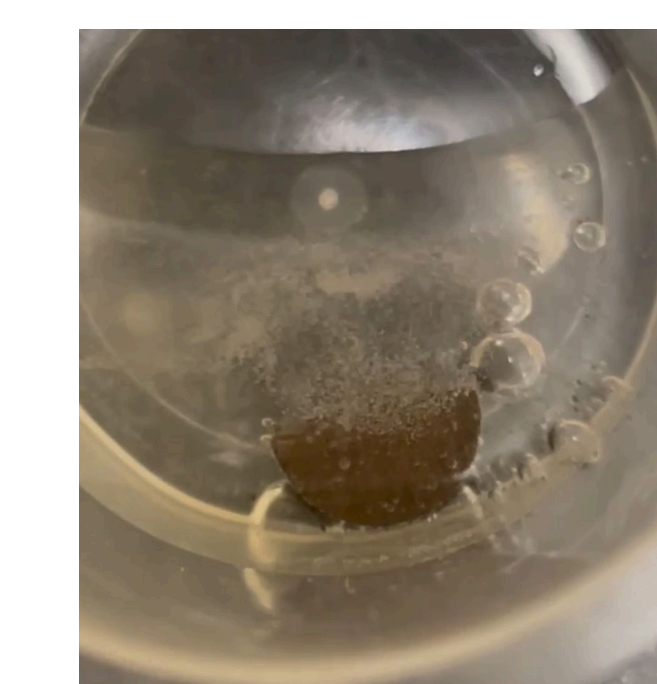


Figure 8. Electrode #4 - Catalytic Activity

## Conclusions

Treatment in an inert argon atmosphere effectively prevented oxidation, preserved pellet integrity, and maximized hydrogen production performance, as observed with Electrode #4. Direct comparison of pre- and post-oxidation results for the same electrode confirmed that oxidation significantly reduces electrical conductivity and the availability of active catalytic sites, leading to lower hydrogen generation rates. These findings indicate that the specific combination of reactants used with nanodiamond is effective for hydrogen production via electrolysis, and that oxidation prevention is a critical factor influencing overall electrode efficiency.

## References

- Dincer, I. (2012). Green methods for hydrogen production. *International Journal of Hydrogen Energy*, 37(2), 1954-1971. <https://doi.org/10.1016/j.ijhydene.2011.03.173>
- Elsayed, A. T., et al. (2022). "Large-scale hydrogen production via water electrolysis: a techno-economic and environmental assessment." *Energy & Environmental Science*, 15(9), 3778-3795. <https://doi.org/10.1039/D2EE01023B>
- Su, L.-X., Cao, Y., Hao, H.-S., Zhao, Q., & Zhi, J. (2021). Emerging applications of nanodiamonds in photocatalysis. *Functional Diamond*, 1(1), 93-109. <https://doi.org/10.1080/26941112.2020.1869431>

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