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Thin film surface modifications of thin/tunable liquid/gas diffusion layers for high-efficiency proton exchange membrane electrolyzer cells

Zhenye Kang[a], Jingke Mo[a], Gaoqiang Yang[a], Yifan Li[a], Derrick A. Talley[a], Scott T. Retterer[b], David A. Cullen[b], Todd J. Toops[b], Michael P. Brady[b], Guido Bender[c], Bryan S. Pivovar[c], Johney B. Green Jr[c], Feng-Yuan Zhang[a,*]

[a] Nanodynamics and High-Efficiency Lab for Propulsion and Power (NanoHELP), Department of Mechanical, Aerospace & Biomedical Engineering, UT Space Institute, University of Tennessee, Knoxville, Tullahoma, TN 37388, USA
[b] Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA
[c] National Renewable Energy Lab, Golden, CO 80401, USA

HIGHLIGHTS

- Novel TT-LGDLs with different surface treatments are investigated for the first time.
- Superior PEMEC performance with a value of 1.63 V at 2.0 A/cm^2 and 80 °C is obtained.
- Ohmic losses is reduced from 0.0925 Ω cm^2 to 0.0700 Ω cm^2.
- Hydrogen production rate can be greatly increased by 28.2%.
- Au thin film surface treatment on titanium material shows good stability.

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ABSTRACT

A proton exchange membrane electrolyzer cell (PEMEC) is one of the most promising devices for high-efficiency and low-cost energy storage and ultrahigh purity hydrogen production. As one of the critical components in PEMECs, the titanium thin/tunable LGDL (TT-LGDL) with its advantages of small thickness, planar surface, straight-through pores, and well-controlled pore morphologies, achieved superior multifunctional performance for hydrogen and oxygen production from water splitting even at low temperature. Different thin film surface treatments on the novel TT-LGDLs for enhancing the interfacial contacts and PEMEC performance were investigated both *in-situ* and *ex-situ* for the first time. Surface modified TT-LGDLs with about 180 nm thick Au thin film yielded performance improvement (voltage reduction), from 1.6849 V with untreated TT-LGDLs to only 1.6328 V with treated TT-LGDLs at 2.0 A/cm^2 and 80 °C. Furthermore, the hydrogen/oxygen production rate was increased by about 28.2% at 1.60 V and 80 °C. The durability test demonstrated that the surface treated TT-LGDL has good stability as well. The gold electroplating surface treatment is a promising method for the PEMEC performance enhancement and titanium material protection even in harsh environment.

1. Introduction

Sustainable energy resources, including solar, wind, and tide, generate electricity intermittently, which leads to challenges in supplying continuous power to the electrical grid and end users [1–8]. High-efficiency and robust electrochemical energy storage or conversion systems coupled with the sustainable energy resources would accommodate seasonal, daily or even hourly changes and have showed promising solutions for future smart grids [7–15]. The ideal energy storage system should exhibit both high performance and low cost to better meet the future requirements [16]. At present, battery is the most widely used method to store and mitigate the intermittent energy sources [17]. Hydrogen can also be an ideal energy carrier due to its high energy density and zero emission, which will not generate any greenhouse gases, like CO_2, during its usage [18–24]. Water electrolysis is the foremost technology for producing high purity hydrogen with its the ability to rapidly follow an intermittent load for grid-balancing that is interrupted due to the supply and demand change for energy

* Corresponding author.
E-mail address: fzhang@utk.edu (F.-Y. Zhang).

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generation and consumption [25]. Proton exchange membrane electrolyzer cells (PEMECs), which act as the reverse proton exchange membrane fuel cells (PEMFCs), have been regarded as a very promising method of hydrogen production for energy storage by water splitting [26–33]. PEMECs also yield very high purity hydrogen gas, which is beneficial for storage and future energy production by multiple methods [34–38].

A typical PEMEC mainly consists of a catalyst coated membrane (CCM), which is sandwiched between two electrodes (anode and cathode). At the anode side, water is split into molecular oxygen, protons, and electrons, which leads to a harsh operating environment for the PEMEC components, i.e. high potential, oxidization and humidity [22,39]. Therefore, the components of the anode electrode, such as LGDL and bipolar plate (BP), require very high corrosion resistance. Titanium (Ti) based LGDLs are widely used as anode LGDL due to their good bulk conductivity, high corrosion resistivity, and excellent mechanical strength. The conventional Ti based LGDLs, including Ti felt, Ti mesh, and sintered Ti powers, have been widely used and investigated in PEMECs [37,39–44]. However, resistance to corrosion in such systems is achieved by surface oxide formation, which can increase surface electrical resistivity and detrimentally impact cell performance [22].

Ito et al. carried out PEMEC experiments focusing on the porosity and pore diameter of titanium felt LGDLs. Their results showed that the electrolysis performance can be improved by decreasing the LGDL mean pore diameter (MPD) when the MPD was larger than 10 μm. And they also pointed out that the porosity had no significant effect on the electrolysis performance if the porosity was greater than 0.5 [42]. In another study, the influence of porosity and pore diameter of LGDLs on PEMEC performance was also conducted. The results showed that the oxygen bubbles produced at the anode may block the LGDL when the MPD is less than 50 μm [43]. Hwang et al. tested titanium felt by loading titanium powder or polytetrafluoroethylene (PTFE) in the PEMECs. They investigated the effects of pore properties and PTFE content on the PEMEC performance [45]. Grigoriev et al. experimentally optimized various parameters of the sintered titanium powder LGDLs. They found that the optimum titanium sphere particle sizes were from 50 to 75 μm, and the pore sizes were around 12 μm [46]. Ioroi et al. investigated the wettability of the titanium mesh by loading TiO_2 powder and PTFE. They concluded that the LGDL with hydrophilic property showed better performance than the hydrophobic one [47]. Millet et al. stated that the interfacial ohmic resistance between the LGDLs and BPs should be reduced in order to improve the performance of the PEMECs [48]. Oh et al. introduced a pore size gradient structure inside the LGDL in order to improve the performance of a PEMFC, concluding that the pore size gradient structure can enhance the steady-state and transient response [49]. Siracusano et al. used a novel short-side chain (SSC) perfluorosulfonic acid (PFSA) membrane to develop low catalyst loading membrane electrode assemblies (MEAs). They achieved a high current density > 3.0 A/cm^2 and demonstrated a very good durability of the low catalyst loading MEAs [25]. Mo et al. have studied the effect of parameters of Ti felt and Ti mesh LGDLs in a PEMEC, and they found that the thickness of LGDLs have a significant impact on the PEMEC performance. They also conducted surface treatments such as thermal nitridation and sputter coating, which can improve the PEMEC performance significantly [50].

Previous efforts have been focused on investigation of different kinds of conventional titanium felt/form/mesh LGDLs and their modification methods to improve PEMEC performance. We recently reported a novel thin, planar titanium LGDL with straight-through pores and tunable pore morphologies that was fabricated using advanced micro/nano manufacturing techniques [51–53]. These novel thin/tunable LGDLs (TT-LGDLs) exhibited superior multifunctional performance with values of 1.66 V at 2.0 A/cm^2 and 80 °C, which to our knowledge exceed the best values reported in the open literature [1,54]. The TT-LGDLs remarkably reduce ohmic and activation losses with its advantages of planar surface and thin features, which are different from the conventional LGDLs. By further reducing the interfacial electrical surface resistance of the TT-LGDLs using surface treatment with the mature and low cost micro/nano technologies, it is anticipated that the performance can be further promoted [27].

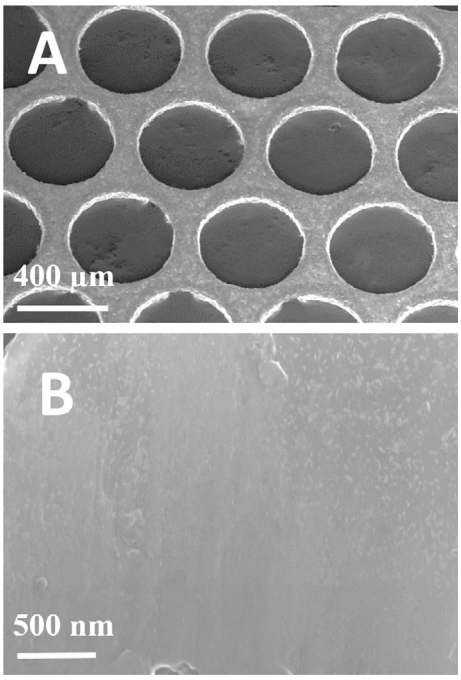

Fig. 1. SEM images of the untreated TT-LGDLs (A) low magnification for untreated TT-LGDLs with a pore diameter about 414 μm and porosity about 0.62 (B) high-resolution surface characterization.

In this study, different gold surface treatment methods were applied on TT-LGDLs for the first time in order to gauge the potential to improve the PEMEC performance and enhance its application. The surface modified TT-LGDLs were evaluated *in-situ* by polarization and EIS techniques and *ex-situ* performed by SEM and EDS with micro/nano-structural characterization of the pre/post testing LGDLs. Furthermore, the 100 h stability test of the surface treated TT-LGDLs showed no obvious degradation.

2. Experimental details

The circular pore shape TT-LGDLs with a pore diameter about 414 μm and porosity about 62%, as shown in Fig. 1, were fabricated by a combination of conventional contact photolithography and chemical wet etching. The detailed fabrication procedures can be found in our previous work [51,54]. It can be seen that the novel TT-LGDLs have a planar surface and straight-through pores, which is completely different from conventional LGDLs, such as titanium felt or mesh [1,44]. The TT-LGDLs can significantly improve the PEMEC performance by reducing the mass transport, ohmic and activation losses. The SEM of the TT-LGDL surface characteristics is shown in Fig. 1(B) and it can be seen that the surface of the untreated titanium TT-LGDL is very smooth, which contribute to the smaller ohmic resistances in PEMECs compared with conventional Ti felt LGDLs.

The TT-LGDLs were used as a substrate for gold sputter deposition. Plasma modifications and sputter deposition augmentations were both completed using a BIO-RAD Polaron Division SEM Coating System E5150. A potential of 2.4 kV and a current of 20 mA was maintained to control the deposition for gold. The thickness of the coating was controlled by adjusting the operating time. The coated Ti foil was weighed before and after sputter deposition to determine coating thickness.

Gold electroplating was conducted mainly with three steps: electro-

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