



Short communication

Unveiling the crucial role of temperature on the stability of oxygen reduction reaction electrocatalysts

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ABSTRACT

The stability of Pt-based/C electrocatalysts used in proton exchange membrane fuel cell (PEMFC) systems is commonly evaluated *via* accelerated stress testing in half-cell configuration at temperature close to ambient ($20 \leq T \leq 25$ °C), and 100% relative humidity (liquid electrolyte). Those conditions are by far different from those encountered in PEMFC systems (solid electrolyte, $60 \leq T \leq 80$ °C, $0 \leq$ relative humidity $\leq 100\%$), and fail in reproducing the morphological changes and the performance losses encountered during real life. Here, using a high surface area Pt/C electrocatalyst, we show that the gap between half-cell and real PEMFC configurations can be bridged by considering the pronounced effect of the temperature. The accelerated stress tests (ASTs) conducted in liquid electrolyte at $T = 80$ °C more accurately reflect the changes in morphology and surface reactivity occurring in real PEMFC environment, and provide gain in time. Due to massive release of Pt^{2+} ions in the electrolyte during ASTs performed at $T = 80$ °C, using fresh electrolyte is strongly recommended for correct determination of the oxygen reduction reaction (ORR) kinetics.

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1. Introduction

State-of-the-art proton exchange membrane fuel cell (PEMFC) cathodes employ carbon-supported platinum-based nanoparticles as electrocatalysts. Various strategies are used to tune their catalytic activity for the sluggish oxygen reduction reaction (ORR), improve their stability, and reduce their overall cost. The most common consists in finely controlling the physical (crystallite size and shape) and the chemical (alloying Pt with transition metals) structure of the Pt nanoparticles [1–8]. Once a new material is synthesized, accelerated stress tests (ASTs) bring about its long-term stability. The degradation protocols recommended by the American Department of Energy (DOE), and the Fuel Cell Commercialization Conference from Japan (FCCJ) mimic load cycles of an operating PEMFC by linear potential sweeps or potentiostatic square-wave pulses between 0.60 and 1.00 V vs. the reversible hydrogen electrode (RHE). Excursions to high electrode potential faced during start-up/shutdown and fuel starvation events are reproduced by linear potential sweeps between 1.0 and 1.5 V vs. RHE [9,10] or potential holds at high electrode potential [11,12]. To lower the development cost of the catalysts, these ASTs are first performed at a thin film of catalyst in half-cell configuration, and only then in a membrane-electrode assembly (MEA) [12–14]. However, this approach has limits as revealed by

the very different conclusions drawn from half-cell and MEA measurements [14]. The nature of the electrolyte (liquid vs. solid in half cell/MEA configuration, respectively) [15], the water content (0 to 100% relative humidity in a MEA vs. 100% in liquid electrolyte), [16] and the incomplete utilization of the catalyst (utilization factor close to 60–80% in MEA vs. nearly 100% in liquid electrolyte) [16] partially account for the observed differences. Recently, Gilbert et al. [14] also showed that ASTs conducted with flowing electrolyte (rather than stagnant) better reproduce the electrochemically surface area (ECSA) losses occurring in a real MEA environment, while retaining the main degradation mechanisms. However, the role of temperature remains understudied, and is the focus of the present communication.

2. Materials and methods

A high surface area Pt catalyst supported on Vulcan XC72 (Pt/C) with a weight fraction (wt.%) of 20% was purchased from E-Tek, and used as received without any further treatment. The number-averaged Pt nanoparticle size was 2.9 ± 0.6 nm.

10 mg Pt/C was suspended in 54 μL of 5 wt% Nafion[®] solution (Electrochem. Inc.), 20 μL of isopropanol and 2.4 mL (18.2 M Ω cm) of ultrapure water (MQ-grade, 18.2 M Ω cm, total organic compounds <3 ppb). After sonication for 15 min, an aliquot of 10 μL of the suspension was drop casted on a glassy carbon disk (0.196 cm²), and dried in air yielding a thin-film rotating disk electrode with a Pt loading of 40 $\mu\text{g}_{\text{Pt}} \text{ cm}_{\text{geo}}^{-2}$.

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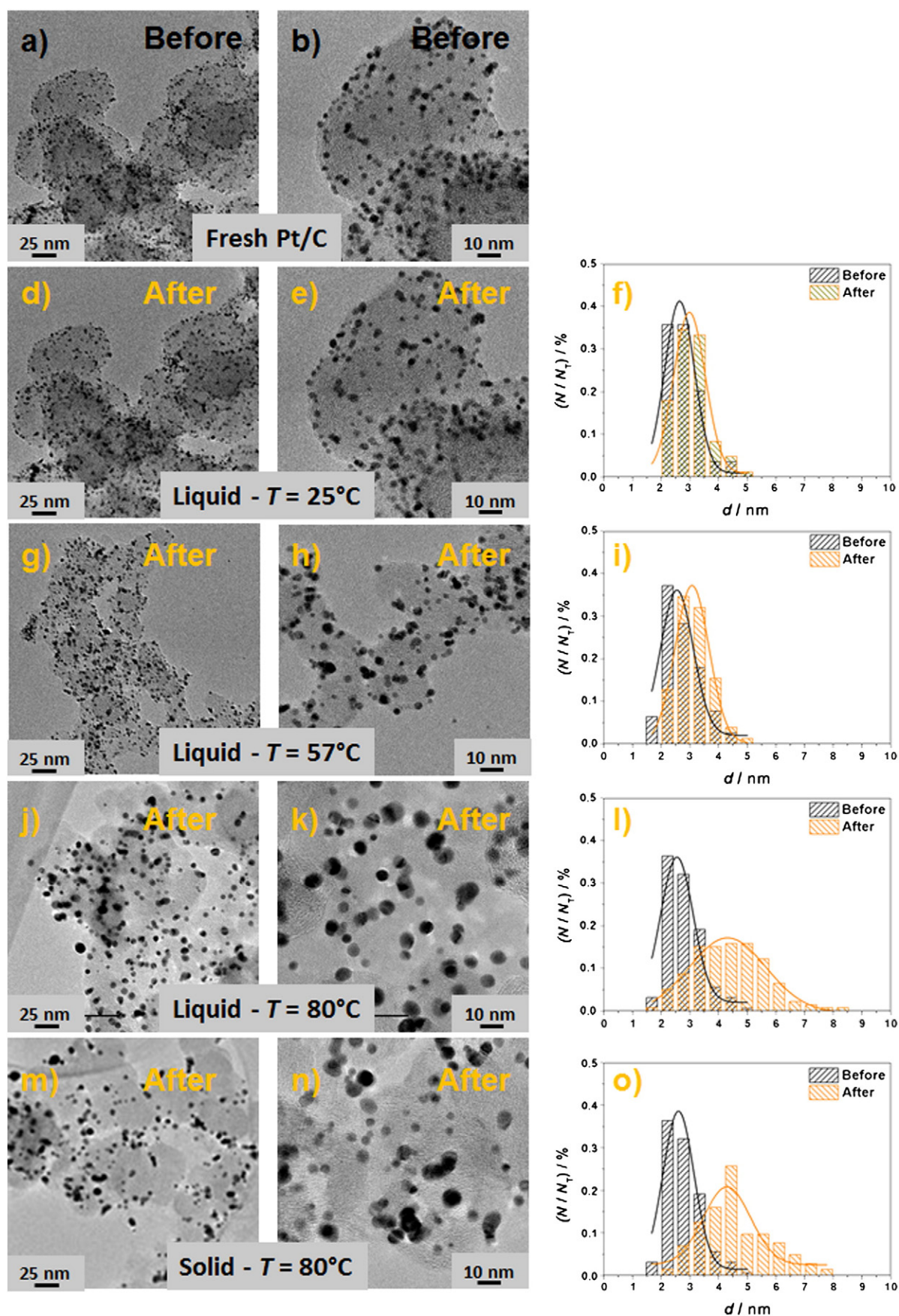


Fig. 1. IL-TEM and TEM images of Pt/C pre- and post-AST at (d, e, f) $T = 25^\circ\text{C}$, (g, h, i) $T = 57^\circ\text{C}$ or (j, k, l) $T = 80^\circ\text{C}$ in 0.1 M H_2SO_4 or (m, n, o) in a single PEMFC (solid electrolyte — $T = 80^\circ\text{C}$), and corresponding Pt particle size distributions.

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