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ORIGINAL PAPER

Temperature effect on the recovery of SO_2-Poisoned GC/Nano-Pt electrode towards oxygen reduction

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Abstract The SO_2 poisoning of Pt nanoparticle (n-Pt) modified glassy carbon (GC/n-Pt) electrode and the recovery of its activity for the oxygen reduction reaction (ORR) were studied using cyclic voltammetry at ambient (25 °C) and elevated (70 °C) temperatures. Recovery of the GC/n-Pt electrode by cycling the potential within the ORR range (1.0 to 0.2 V (standard hydrogen electrode)) in 0.1 M H_2SO_4 was not effective at 25 °C, but at 70 °C the onset potential of the ORR was almost the same as that at the fresh GC/n-Pt electrode. For the two different temperatures used here, the recovery on cycling the potential between 0.4 and 1.7 V was efficient. However, the number of cycles and the amount of charge required for the recovery at 70 °C were the smallest, which is of great interest for the proton exchange membrane fuel cell performance. The recovery using such a wide potential range at 70 °C resulted in an enhancement of the electrocatalytic activity of the GC/n-Pt electrode over a non-poisoned (bare) GC/n-Pt electrode.

Keywords ORR · Platinum · Nano · SO_2 · Recovery · Fuel cell

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Introduction

The potential for commercialization of low-temperature fuel cells faces several problems. Among those is their costly platinum electrocatalyst which is susceptible to poisoning by adsorbed contaminants. These platinum electrodes are sensitive to the poisoning effects of many molecular species, such as sulfur and nitrogen compounds [1, 2]. It has long been known that the presence of air-borne poisons such as SO_2 and NO_2 in proton exchange membrane fuel cells (PEMFCs) dramatically affects their performance [3–6]. The oxygen reduction reaction (ORR) is much slower than the hydrogen oxidation reaction and the effects of poisoning substances on the activity of the Pt electrode for ORR require more attention. The electrochemical behavior of SO_2 at polycrystalline platinum (Poly-Pt) electrodes has been studied [7–9]. However, a little attention has been paid to the poisoning effect of SO_2 on Poly-Pt or platinum nanoparticles (n-Pt) electrodes towards the ORR [10]. The danger of sulfur contamination in the fuel cells arises from the relative ease of the change in the oxidation states of sulfur compounds, which may lead to the formation of several sulfur species (e.g., SO_2, $S_4O_6^{2-}$, S, $S_2O_3^{2-}$, and SO_4^{2-}) within the operational potential window of fuel cells, i.e., from 1.0 to 0.2 V (standard hydrogen electrode, SHE) [11].

Although the highest performance of H_2/air PEM fuel cell has usually been achieved at elevated temperatures (70–90 °C), no work has been reported on the poisoning of Pt electrodes at these temperatures in electrolyte solutions. Many researchers have studied and reviewed the effect of different sulfur species on the PEMFCs performance [12–16]. The aim of the present work is to explore the effects of the SO_2 poisoning on the ORR at n-Pt electrodes in 0.1 M H_2SO_4 at ambient and elevated temperatures (25 °C and 70 °C). Also, processes such as short-range recovery (by cycling potential within the ORR range) and long-range recovery of poisoned electrodes (by cycling potential in a higher range) are examined under these experimental conditions. In the present study, we used 0.1 M H_2SO_4 as an electrolyte solution as is very often used in the electrochemical studies for developing PEMFCs, especially the fundamental electrocatalysis of their electrocatalysts, while in PEMFCs Nafion is typically used as a polymer electrolyte membrane. Although the present case is not exactly the real one, it may help to give better understanding of SO_2-poisoning effects and recovery approaches (both short and long range) that may be used in the recovery of PEMFC performance.

Experimental

A working electrode consisting of platinum nanoparticles-electrodeposited glassy carbon (GC) electrodes (φ=3 mm) was used for the cyclic voltammetric (CV) experiments. The counter and the reference electrodes were, respectively, a spiral Pt wire and Ag/AgCl/KCl (sat.) (0.198 V vs. SHE at 25 °C). All the potential values throughout this work are expressed vs. SHE. Prior to the electrodeposition of Pt nanoparticles, the GC electrode was polished first with no. 2000 emery paper, then with aqueous slurries of successively finer alumina powder (particle size down to 0.06 mm) with the help of a polishing microcloth. The bare GC electrode was then sonicated for 10 min in Milli-Q water. Pt nanoparticles were electrodeposited from acidic bath of 0.1 M HCl containing 3 mM H_2PtCl_6 by applying bi-potential steps from the open-circuit potential to −1.3 V (1 s) and then to −0.04 V (6 s). Holding the potential at −1.3 V was to initiate the nucleation at a high rate followed by stepping the potential to −0.04 V to continue the growth of the platinum nanoparticles. The average size of the Pt particles was 35 nm. The surface area of the Pt nanoparticles in the modified GC electrode was 0.3±0.01 cm^2. It was estimated from the hydrogen adsorption/desorption peaks in N_2-saturated 0.1 M H_2SO_4 where 1 cm^2 is equivalent to 210 µC [17]. Also, by knowing that the density of Pt equals 21.46 g cm^{-3}, the Pt loading on the GC electrode can be estimated to be 0.02 mg cm^{-2}. This value matches well with the *i-t* measurements during the deposition.

The ORR was studied in 0.1 M H_2SO_4 using a conventional two-compartment Pyrex glass container. Milli-Q water (Millipore, Tokyo, Japan) was used to prepare all the solutions. Prior to each experiment, O_2 gas was bubbled directly into the cell for 30 min to obtain an O_2-saturated solution, and during the measurements O_2 gas was flushed over the cell solution. Mel'nichenko et al. [18] reported that the solubility of O_2 gas in distilled water at 25 °C and 70 °C is 6.5 and 2.9 ml l^{-1}, respectively. These amounts are equivalent to 8.5 and 3.3 mg l^{-1}.

SO_2 poisoned solution was prepared by introducing a specific volume of a solution containing 0.1 M Na_2SO_3 in 0.1 M H_2SO_4, in which Na_2SO_3 was completely converted to soluble SO_2, thus giving a desired concentration of the latter in 0.1 M H_2SO_4. This means that the largest amount of SO_2 in solution cannot exceed 0.32 g l^{-1}. The solubility of gaseous SO_2 in distilled water at 25 and 70 °C is 97 and 27.9 g l^{-1}, respectively [19], so the SO_2 concentrations that have been used in this work are about two orders of magnitude lower than the solubility of SO_2 at the higher temperature. This eliminates the possibility that a possible change in the behavior at a higher temperature might be attributed to the change in SO_2 solubility at that temperature.

The measurements were carried out at 25 and 70 °C (±1 °C). Electrochemical measurements were performed using a BAS 100 B/W electrochemical analyzer (Bioanalytical system, Inc., Kent Avenue, West Lafayette, IN, USA). The current density was calculated on the basis of the geometrical surface area of the underlying GC electrode. Deaeration of the electrolyte solutions was, if necessary, carried out by bubbling N_2 gas for at least 30 min prior to the electrochemical measurements. The CV measurements were repeated to assure the reproducibility of the results.

Results and discussion

SO_2 poisoning

Figure 1 shows a typical SEM image of the GC/n-Pt electrode prepared by the method given in the "Experimental" section. It shows the distribution of the electroplated Pt nanoparticles

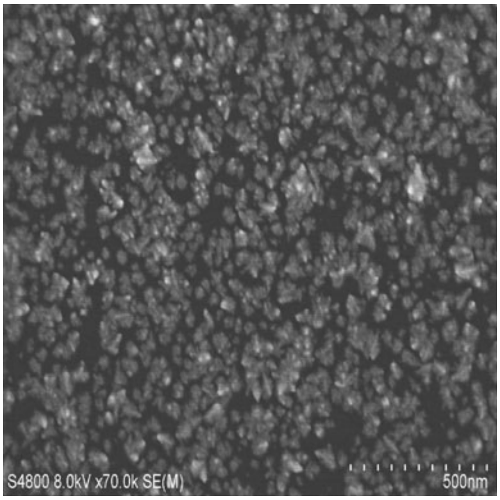


Fig. 1 SEM image for GC/n-Pt electrode showing the Pt nanoparticle distribution above the GC electrode

at the surface of the GC electrode. The rather uniform size and density distribution of these nanoparticles can be confirmed from this micrograph. The size of the particles is in the range of 30–40 nm. Poisoning of the GC/n-Pt electrode was done by repeating the potential cycle between 1.0 and 0.2 V three times in N_2-saturated 0.1 M H_2SO_4 containing 5.0×10^{-5} M SO_2. Note that an equilibrium between SO_2 and $SO_3{}^{2-}$ exists in the solution, according to the chemical equation;

$$\begin{array}{c} SO_2{+}H_2O \rightleftharpoons HSO_3^- + H^+ \rightleftharpoons SO_3^{2-}{+}2H^+ \\ pK_a = 1.86 \qquad pK_a = 7.2 \end{array} \qquad (1)$$

From the above equation, it is concluded that at the present low pH, the predominant species in solution is the SO_2 molecule [20].

Figure 2 shows the poisoning of the GC/n-Pt electrode at the two temperatures (one cycle is shown here for simplicity). In this case an adsorbed sulfur species layer is formed on the electrode surface. In previous studies, many research groups have reported the formation of sulfur and sulfur compounds on Pt surface as a result of SO_2 reduction [10, 16, 21, 22]. An ill-defined cathodic peak was obtained starting from 0.6 V which corresponds to the reduction of SO_2 to SO_x and S [21, 22]. The charge of adsorption (Q_{ads}) was estimated by integrating the cathodic part of the CVs in Fig. 2 at 25 and 70 °C. The values of Q_{ads} were estimated to be 5.7×10^{-5} and 2.7×10^{-4} C at 25 °C and 70 °C, respectively. It can be seen that the Q_{ads} at 70 °C is higher than that at 25 °C. Although the solubility of SO_2 decreases with temperature, the rate of reduction of SO_2 on nano-Pt at 70 °C is much higher than that at 25 °C and hence the charge of reduction (Q_{ads}) at the higher temperature. That is to say, the greater reduction kinetics at 70 °C has a greater effect on the rate of reduction than the higher solubility of SO_2 at 25 °C.

Electrode recovery

The poisoned electrodes prepared above were transferred to fresh SO_2-free oxygen-saturated 0.1 M H_2SO_4 to study its recovery in two different potential ranges. Figures 3 and 4 show the ORR on a SO_2-poisoned GC/n-Pt in clean (SO_2-free) solution of 0.1 M H_2SO_4 at 25 °C and 70 °C, respectively.

Note that at the fresh (bare) electrode (curve 8 in Figs. 3 and 4) the peak current (at ca. 0.6 V) obtained at 70 °C is much higher than that obtained at 25 °C. There is a contradiction in the literature [23–25] regarding the effect of temperature on the ORR. Although some authors have reported higher wave currents at higher temperatures [23], others have reported lower currents [24, 25]. In Ref. 25, the ORR current was low at low temperatures, then increases up to temperature of 60 °C and subsequently decreases at 80 °C. According to this reference, as the temperature increases between 20 °C and 60 °C, higher diffusivity of O_2 prevails and gives higher currents. However, at 80 °C the lower solubility of O_2 becomes predominant and lower wave currents were obtained. However, the current at 80 °C was still higher than that at 20 °C. In our case, comparing the current peaks at both temperatures studied shows that the increase in the diffusivity of molecular oxygen overweighed the lower solubility of O_2 at higher temperatures, which led to an enhancement of the ORR current at the non-poisoned GC/n-Pt electrode, i.e., the current is almost doubled at 70 °C, but the onset potential of the ORR is almost the same as that at 25 °C.

Fig. 2 The poisoning of the GC/n-Pt electrode in N_2-saturated 0.1 M H_2SO_4 containing 5×10^{-5} M SO_2 at different temperatures

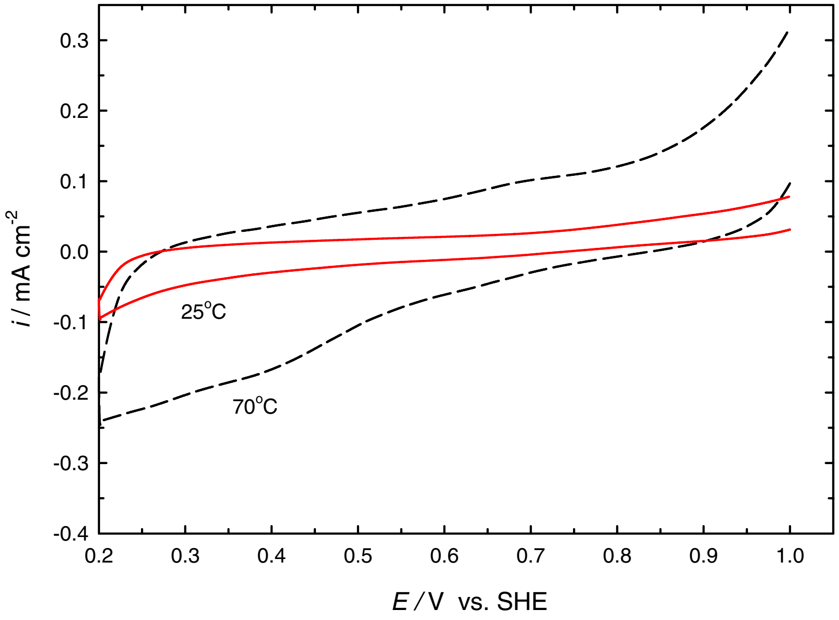
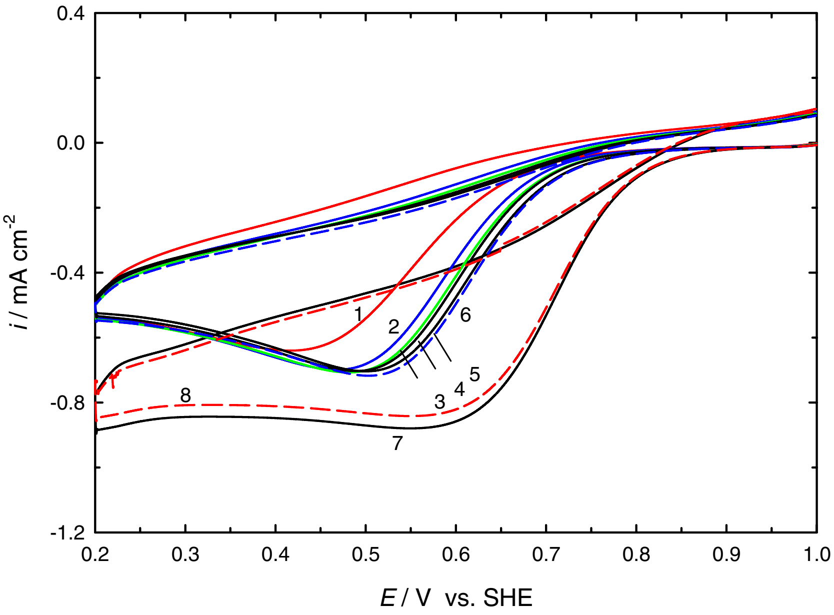


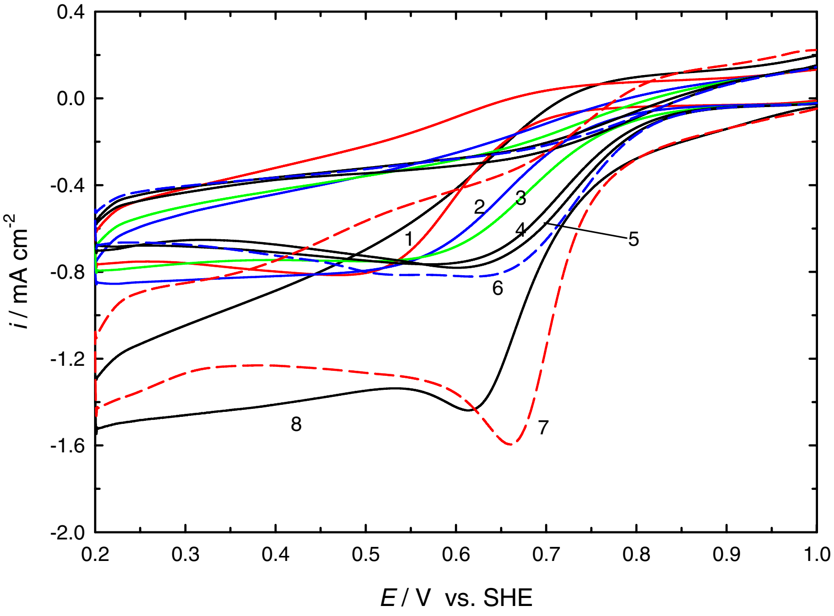
Fig. 3 Comparison of the behavior of a poisoned GC/n-Pt electrode towards the ORR in O_2-saturated SO_2-free 0.1 M H_2SO_4 at 25 °C (*1*–*8*) during potential cycling between 1.0 and 0.2 V (*1*–*6* cycles) and (*7*) after the potential scanning from 1.0 to 0.4 V and further to 1.7 V and then back to 0.4 V. Voltammogram *8* was obtained at non-poisoned (fresh) GC/n-Pt electrode. Potential scan rate: 0.1 V s^{-1}



The poisoned electrodes at 25 °C and 70 °C were recovered using two methods. Short-range recovery was achieved by cycling the potential between 0.2 and 1.0 for several cycles and long-range recovery was achieved by scanning the potential to 1.7 V (see Fig. 5). At 25 °C, cycling the potential between 1.0 and 0.2 V (see cycles 1–6 in Fig. 3) did not significantly improve the onset potential or the ORR current. However, longer-range cycling (*cf* Fig. 5) gave almost full recovery of electrode activity. The voltammetric curve for the electrode recovered by long-range cycling (curve 7) almost coincides with that of the performance of a fresh electrode (curve 8).

At 70 °C (Fig. 4), on potential cycling between 1.0 and 0.2 V (i.e., within the range of the ORR) the onset potential of the ORR is gradually improved. The current in the diffusion-limited region is still much less than that at the non-poisoned (fresh) electrode. However, the electrode does show partial recovery. The improvement in the onset potential was continuously observed until the sixth potential cycle and no further improvement was obtained on further cycling. Thus, short-range cycling fully recovers the onset potential of the ORR, while retaining the diffusion current of the poisoned electrode. This gradual improvement was not observed at the lower temperature studied

Fig. 4 Comparison of the behavior of a poisoned GC/n-Pt electrode towards ORR in O_2-saturated SO_2-free 0.1 M H_2SO_4 at 70 °C (*1*–*8*) during potential cycling between 1.0 and 0.2 V (*1*–*6* cycles) and (*7*) after the potential scanning from 1.0 to 0.4 V and further to 1.7 V and then back to 0.4 V. Voltammogram *8* was obtained at non-poisoned (fresh) GC/n-Pt electrode. Potential scan rate: 0.1 V s^{-1}



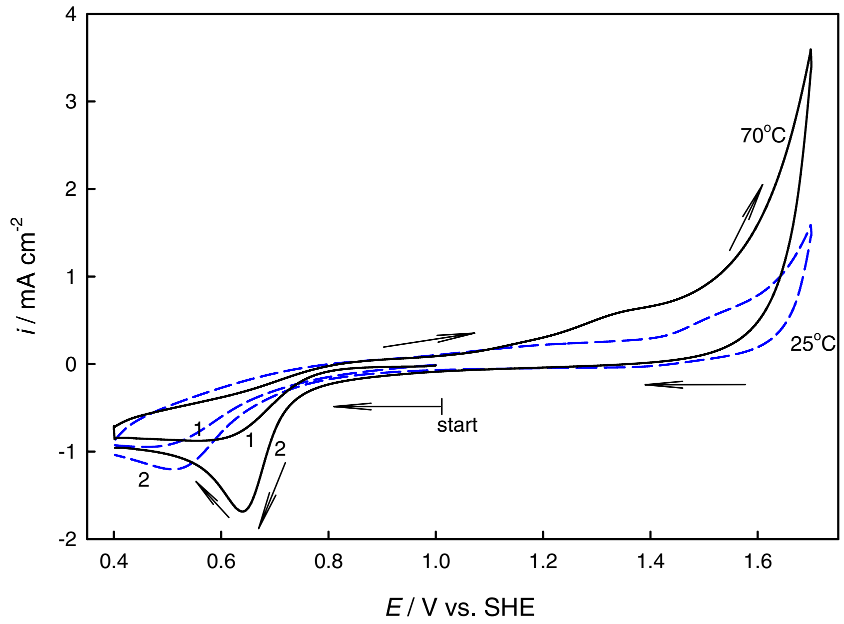


Fig. 5 Recovery by potential scanning (1.0→0.4→1.7→0.4 V) of a poisoned GC/n-Pt electrode in O_2-saturated 0.1 M H_2SO_4 at 25 °C and 70 °C

(*cf.* Fig. 3). The improvement in the recovery at the higher temperature may be attributed to the desorption of the weakly bound adsorbed sulfur species from the surface of the GC/n-Pt electrode. Operation at a higher temperature enhances the desorption of sulfur and sulfur species from the GC/n-Pt, allowing O_2 molecules to adsorb on the nano-Pt surface. However, a full recovery of the activity of the Pt surface was obtained by potential scanning in the range of 0.4 to 1.7 V, as shown in Fig. 5. The potential was scanned from 1.0 to 0.4 and further to 1.7 and then back to 0.4 V. This scan is shown in Fig. 5 at both 25 °C (dashed line) and at 70 °C (solid line). After the activity recovery shown in Fig. 5, the ORR at this electrode was recorded (see Figs. 3 and 4, curve 7). The recovery with potential cycling in the higher potential range (long-range recovery) is clearly due to the oxidation of strongly adsorbed sulfur to soluble sulfate ion according to the following equation:

$$S + 4H_2O \rightarrow SO_4^{2-} + 8H^+ + 6e^- \qquad (2)$$

Another interesting observation from Fig. 4 is the higher electrocatalytic activity for the ORR of the “recovered” GC/n-Pt electrode at 70 °C compared to that of the corresponding non-poisoned (bare) electrode. For instance, comparing curves 7 and 8 in Fig. 4, the cathodic peak

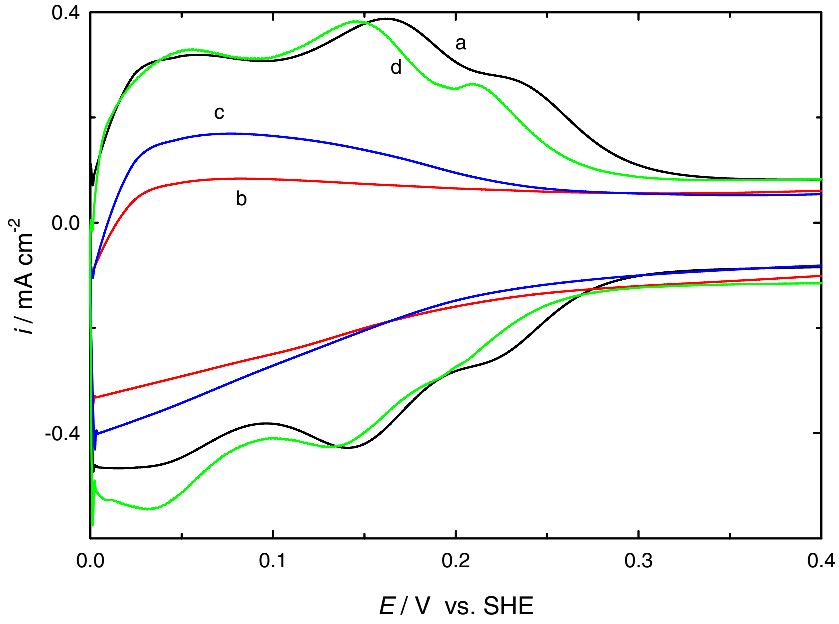
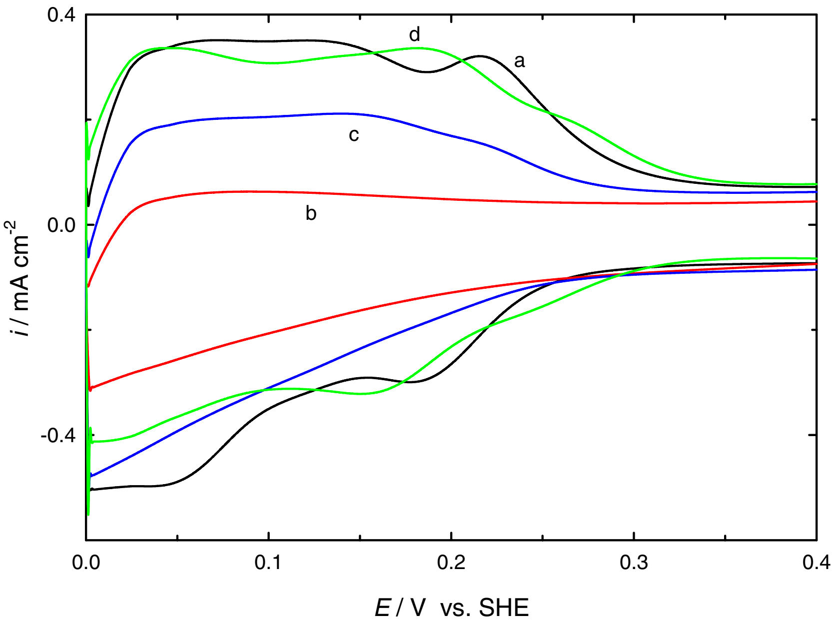


Fig. 6 H-adsorption/desorption of the GC/n-Pt electrodes: *a* fresh electrodes; *b* after poisoning; *c* after short-range recovery; and *d* after long-range recovery at 25 °C

Fig. 7 H-adsorption/desorption of the GC/n-Pt electrodes: *a* fresh electrodes; *b* after poisoning; *c* after short-range recovery; and *d* after long-range recovery at 70 °C



potential is shifted; 50 mV positive compared with the non-poisoned (fresh) one. This may be attributed to the presence of trace amounts of sulfur on the nano-Pt particles. Some reports have shown that traces of adsorbed sulfur can have enhanced electrocatalytic effects [26]. We may conclude that there are trace levels of sulfur that may enhance the ORR due to electronic effects. However, when the amount of adsorbed sulfur exceeds these levels the ORR is not enhanced. In accordance with this speculation, the ORR is not enhanced at 25 °C in the presence of relatively high levels of sulfur (θ=6% at 25 °C, θ=3% at 70 °C where θ is the sulfur surface coverage estimated from the charge consumed during the hydrogen desorption (cf Figs. 6 and 7). The slight positive shift in the oxygen reduction peak may be attributed also to the partial dissolution of the platinum particles at high positive potential. This dissolution may lead to a new surface morphology of the nanoparticles which could be behind the electrocatalysis of the oxygen reduction. However, it should be stressed that scanning the potential to higher values may corrode the underlying substrate. Further details regarding this point will be reported in the near future.

One can explain the different behaviors of the electrodes studied under various conditions of recovery by considering the electrochemical active areas of the electrodes. The active areas of the different electrodes were estimated from the data shown in Figs. 6 and 7 which show the hydrogen adsorption/desorption ($H_{ads/des}$) patterns at 25 °C and 70 °C, respectively. These are for the as-prepared GC/n-Pt (curves a), the poisoned electrode (curves b) and the recovered electrode (curves c, short range) and curves d, long range). The active area is an expression for the electroactive Pt sites on the surface. Real surface areas (A) for the bare, poisoned, and recovered electrodes were estimated from the charge consumed (Q) in the hydrogen desorption. Then the surface

Table 1 Real surface area (A), charge consumed in the hydrogen desorption (Q), and the surface coverage of sulfur (θ)

Electrodes	Real area (A)/cm^{2a}		Charge (Q)/μC		Surface coverage (θ)	
	25°C	70°C	25°C	70°C	25°C	70°C
Bare GC/n-Pt	0.31	0.31	65.5	65.3		
Poisoned GC/n-Pt[b]	0.08	0.06	17.0	13.5	0.74	0.84
Short-range-GC/n-Pt[c]	0.12	0.18	26.7	37.9	0.59	0.42
Long-range-GC/n-Pt[d]	0.29	0.30	60.6	63.0	0.06	0.03

[a] As estimated from the charge consumed in the hydrogen desorption using 210 μC/cm^2 [17].

[b] The GC/n-Pt poisoned by cycling the potential between 1.0 and 0.2 V (SHE) in deaerated 0.1 M H_2SO_4 containing 5.0 × 10^{-3} M Na_2SO_3.

[c] As reactived by short-range recovery, i.e., by cycling the potential between 1.0 and 0.2 V (SHE).

[d] As reactivated by the long-range recovery, i.e., by scanning the potential from 1.0 to 0.4 and further to 1.7 and then back to 0.4 V (SHE).

coverage of sulfur (θ) was calculated from the following relation:

$$\theta = 1-(Q/Q^{o}) \quad (3)$$

where Q^{o} and Q are the charges for hydrogen desorption measured at the fresh (bare) and at different conditioned electrodes (poisoned or recovered), respectively. The charge consumed in the hydrogen desorption, real surface area, and the surface coverage of the different electrodes, i.e., fresh, poisoned, and recovered are given in Table 1. Inspection of Figs. 6 and 7 and Table 1 reveals several interesting points:

i. The real surface areas of the fresh GC/n-Pt electrodes used for ORR at both temperatures are the same. This indicates that the higher current of the ORR at 70 °C is not attributed to a difference in area.
ii. Long-range recovery of the GC/n-Pt electrode almost completely reactivates the voltammetric response at both 25 °C and 70 °C: the percentages of the recovered areas at 25 °C and 70 °C are 94 and 97%, respectively.
iii. The area recovered by short-range recovery at 70 °C (58% recovery) is higher than at 25 °C (41% recovery). Thus it is obvious together with (ii) that the most effective reactivation is obtained by using long-range recovery at both temperatures.

The charge of recovery (Q_{rec}) is given by the amount of charge consumed in the oxidation of the sulfur species to soluble SO_4^{2-} using the CVs shown in Fig. 5. This is given by the charge passed in the potential range from 1.0 to 1.44 V in which sulfur is oxidized to soluble sulfate ion [10] and before the onset of the oxygen evolution reaction (OER). The values of Q_{rec} were estimated to be 6.4×10^{-5} and 1.1×10^{-4} C at 25 °C and 70 °C, respectively. This result indicates that at 70 °C, Q_{rec} is by ca. three times less than Q_{ads} (see results of Fig. 2), while at 25 °C Q_{rec} is almost equal to Q_{ads}. One can conclude that the recovery at 70 °C is much more efficient due to not only the sulfur oxidation but also the mechanical removal of sulfur species by evolved O_2.

It is believed that the efficient recovery at high temperature speeds up by the oxidation of adsorbed S to the soluble SO_4^{2-}. This may be attributed to the higher OER kinetics at 70 °C which are obvious from a consideration of the CVs in Fig. 5 where the onset potential and the current of the OER at 70 °C are more negative and larger than those at 25 °C, respectively. The higher rate of O_2 gas evolution may enhance the mechanical detachment of the adsorbed sulfur species and thus enhance the recovery of the poisoned electrode.

At the present stage, our results may be, though not directly, related to practical PEMFCs. For example, the results presented here show that the coverage by sulfur species is stable (depending on temperature) within the potential region of the ORR i.e., 0.2 to 1.0 V, implying that the sulfur species cannot be removed from the Pt/Vulcan carbon surface under PEMFC steady-state operating conditions, where the cathode potential does not exceed 1.0 V. However, potentials up to 1.7 V have been measured during PEMFC start up [27] and some practical measurements have shown that S is removed from the MEA cathodes at open-circuit potentials [10]. The present results may help to understand some of these practical processes.

Summary and conclusions

The SO_2 poisoning of the GC/n-Pt electrode in the ORR was examined at two different temperatures. Although the recovery behavior of the poisoned GC/n-Pt electrode at 25 °C is similar to that at 70 °C, recovery is easier at 70 °C than at 25 °C. Recovery of the GC/n-Pt electrode by cycling the potential within the ORR range (between 1.0 and 0.2 V) is not effective at 25 °C, but at 70 °C the recovery of the onset potential of the ORR is achieved. For all the electrodes and at both temperatures used here, the reactivation process by potential cycling between 0.4 and 1.7 V was efficient and the number of cycles required for full recovery was fewest at 70 °C. This would keep the dissolution of the Pt catalyst during the recovery of poisoned PEM fuel cells to a minimum level. It is worthy to mention that starting the recovery using the wide potential window resulted in an increase in the catalytic activity of the GC/n-Pt electrode towards the ORR by shifting the E_p for the ORR by 50 mV in a positive direction.

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