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ABSTRACT

A quantitative research on S and SO$_2$ poisoning Pt/Vulcan carbon (Pt/VC) catalysts for fuel cells was conducted by the three-electrode method. Pt/VC electrodes were contaminated by submersion in a SO$_2$-containing solution made up of 0.2 mM Na$_2$SO$_3$ and 0.5 M H$_2$SO$_4$ for different periods of time, and held at 0.05 V (vs. RHE) in 0.5 M H$_2$SO$_4$ solutions in order to gain zero-valence sulfur (S^0) poisoned electrodes. The sulfur coverage of Pt was determined from the total charge consumed as the sulfur was oxidized from S^0 at 0.05 V (vs. RHE) to sulfate at >1.1 V (vs. RHE). The summation of initial coverage of S^0 (θ_S) and coverage of H (θ_H) are approximately equal to 1 ($\theta_H + \theta_S = 1$) when $0.5 < \theta_H < 1$, which gives an easy way to figure out the quantity of sulfur adsorbed on Pt/VC. As to SO$_2$ poisoned Pt/VC, the catalytic activity of oxygen reduction reaction (ORR) decreases linearly with the amount of SO$_2$ adsorbed on the Pt nanoparticles when $0.45 < \theta_H < 0.81$. When the adsorbed SO$_2$ was reduced to S^0, the mass activity was greatly recovered, and the area specific activity was even higher than the unpoisoned Pt/VC.

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1. Introduction

As one of the most promising power sources for automotive applications, proton exchange membrane fuel cells (PEMFCs) are expected to use ambient air as the oxidant. Thus the cathode may be exposed to trace undesirable contaminants in the air, such as SO$_2$, NO$_x$ and organic compounds, which are supposed to be adsorbed by Pt nanoparticles and degrade the performance of PEMFC [1–4]. Among the ordinary contaminants, sulfur-contained compounds are one of the most severely poisons that affect the performance [3–5]. Thus a lot of studies have been performed on the poisoning behavior of SO$_2$, and exploring the impacts of temperature, humidity, potential and concentration of SO$_2$ under operating conditions in order to get the recovery strategies [5–11].

There are literatures reporting the poisoning behaviors of different kinds of sulfur-contained species on PEMFC [12], but the effects of sulfur with different valences on the performance of Pt only draw minimal attentions. Since the catalytic layers are affected by SO$_2$ most severely, literatures have also paid lots of attentions to catalysts [10,13–17]. Garsany et al. [13] quantitatively studied the impact of SO$_2$ on the ORR at Pt/VC catalysts, and found that H$_2$O$_2$ was produced when Pt/VC was covered with S^x species. The

quantitative method they used was a bit different from ours used here, since they did not consider the recovery of the area of adsorption/desorption of hydrogen in each cycling of cycle voltammetry (CV). Gould et al. [12] studied the effects of SO$_2$, COS and H$_2$S on the performance of Pt/VC, and found they had the similar poisoning behaviors, which indicated a similar poisoning mechanism. Baturina et al. [14] studied the SO$_2$ poisoning behavior on Pt$_3$Co/VC and Pt/VC fuel cell catalysts, and found that Pt$_3$Co/VC was easier to recover the performance, despite that it was more susceptive to SO$_2$ poisoning than Pt/VC at a given platinum loading. However, only a few literatures quantitatively studied the effects of SO$_2$ on ORR [13–15], probably due to a lack of convenient method to calculate the amount of SO$_2$ adsorbed on Pt nanoparticles.

Here we presented a method that can easily calculate the quantity of SO$_2$ adsorbed on Pt surfaces with the potentiostatic method and cycle voltammetry (CV) in conventional three-electrode system. In addition, the effects of S^0 and SO$_2$ on the performance of Pt/VC towards ORR were quantitatively evaluated with the rotating disk electrode (RDE) methodology.

2. Experimental

2.1. Preparation of Pt/VC coated electrodes

A commercially available catalyst comprising 40 wt% Pt supported on Vulcan carbon XC-72 (Johnson Matthey) was used. Thin

film electrodes were prepared using a method adapted from the literature [18]. For short, 5 mg catalyst was dispersed in a mixture of 50 μL 5 wt% Nafion solution and 1 mL isopropanol. The mixture was agitated by ultrasonic for more than 30 min to form an ink. Then 3 μL of this ink was dropped on the glassy carbon disk (0.1256 cm^2) of RDE and dried to yield a thin film electrode.

2.2. Preparation of three-electrode system

All of the electrochemical measurements were conducted using a three-electrode method in a glass cell at room temperature. A Pt mesh was used as the counter electrode. A saturated calomel electrode (SCE) was used as the reference electrode. A 0.2 mM Na_2SO_3/0.5 M H_2SO_4 solution prepared by dissolving a corresponding quantity of anhydrous Na_2SO_3 in a 0.5 M H_2SO_4 solution was used as the contaminant source. There is an equilibrium between SO_3^{2-} and SO_2 in the acidic solution, and in this case the Na_2SO_3 is completely converted to SO_2 soluble in the aqueous solution. The H_2SO_4 solution was bubbled with N_2 for more than 30 min before Na_2SO_3 was added. The mixed solution was shaken up before used.

2.3. Electrochemical characterization

A method for the adsorption of aqueous SO_2 on Pt surfaces described by Quijada and co-workers was used in this paper [19]. The Pt catalysts were poisoned by holding the electrodes at the potential of 0.65 V in the 0.2 mM Na_2SO_3/0.5 M H_2SO_4 solution for 0.5, 1, 2 and 5 min, respectively. Then the electrodes were withdrawn covered with a droplet of this solution and rinsed with more than 100 mL de-ionized water. After that, the following experiments were conducted: (1) For determining the sulfur coverage of Pt, the electrodes were introduced into clean N_2-saturated 0.5 M H_2SO_4 electrolyte and held at 0.05 V for 10 min to deoxidize the adsorbed SO_2, and then cycled from 0 to 1.4 V at a sweep rate of 50 mV s^{-1} for more than 10 cycles. (2) The ORR polarization curves of the SO_2 poisoned electrodes were performed at a sweep rate of 5 mV s^{-1} from 1.0 to 0.2 V in O_2-saturated 0.5 M H_2SO_4 at a rotating speed of 1600 rpm. (3) For the S^0 poisoned electrodes, the electrodes were held at a potential of 0.05 V for 10 min in the clean N_2-purged 0.5 M H_2SO_4 electrolyte in order to deoxidize the SO_2 to S^0, then the ORR polarization curves were conducted as described above.

3. Results and discussion

3.1. Determination of the quantity of SO_2 adsorbed on Pt

Fig. 1 shows a sequence of 10 CV cycles of a Pt/VC that has been deoxidized after poisoning in the 0.2 mM Na_2SO_3/0.5 M H_2SO_4 solution at 0.65 V for 2 min. The curve of the unpoisoned Pt/VC is shown in the same figure as a reference. The CV characteristic is quite similar to those described else-where [8,13,20], except for the shift of some peaks of potential. The adsorbed sulfur species modify the cyclic voltammetric curve of the Pt/VC catalysts. During the first positive sweep from 0 to 1.4 V (CV 1), the electrochemical oxidation of the adsorbed S^0 to SO_4^{2-} causes a wide anodic peak at 1.15 V. During the first negative sweep from 1.4 to 0 V, reduction peaks appear at about 0.72 V and 0.08 V, which is probably attributed to the reduction of adsorbed oxygen species, protons in the solution and a small amount of sulfur species that are still adsorbed on the surface, respectively. As cycling progresses, the cathodic peaks near 0.72 and 0.08 V increase slowly, implying that sulfur species are removed from the Pt/VC surface. The increase of anodic peak currents between 0 and 0.4 V indicates that more and more electrochemical active surface areas (ECA) are exposed, which results in the adsorption of more hydrogen in each cycle.
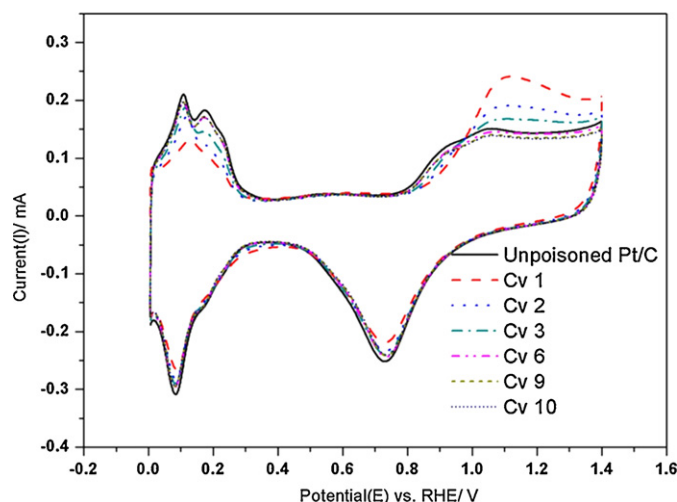

Fig. 1. Cyclic voltammetry of Pt/VC standard compared to selected from voltammograms from 1 to 10 sequential cycles of a Pt/VC electrode after poisoning in 0.2 mM Na_2SO_3/0.5 M H_2SO_4 solution for 2 min: 45.5 μg$_{Pt}$ cm^{-2}, E_i = 0 V; polarization program 0 V → 1.4 V → 0 V at 50 mV s^{-1}.

The anodic peak around 1.15 V decreases slowly and shifts toward more negative potentials implying that the Pt surface is becoming more dominated by the adsorption of oxygen rather than the oxidation of sulfur species. The voltammograms for CV 9 and 10 are essentially coincided and identical to that of the unpoisoned Pt/VC, implying that the sulfur species are wholly removed.

Using the data in Fig. 1, we can determine the amount of sulfur species adsorbed on the Pt/VC. First of all, according to Garsany et al. [13], the adsorbed SO_2 are wholly on the surface of Pt, and the Vulcan carbon does not adsorb SO_2. Moreover, we already know the relativity between potential and the oxidation state of sulfur [19,21–26]. At the potential of 0.65 V, an unchanged product of SO_2 adsorption was formed on Pt surfaces. At the potential of 0.05 V (vs. RHE), sulfur is in a zero-valence state (S^0), which causes an adsorption state of Pt–S, or rather, one Pt atom on the surface adsorbing one S^0 atom [8,21]. At high potential (>1.0 V) (vs. RHE), sulfur adsorbed on Pt begins to be oxidized to sulfate (SO_4^{2-}), which desorbs from the Pt surface. This process can be described by the following equation [27]:

$$S^0 + 4H_2O \rightarrow SO_4^{2-} + 8H^+ + 6e^- \qquad (1)$$
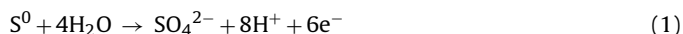
The number of electrons transferred for each S atom is 6. Furthermore, during the cycle n (n = 1–10) of the cycling progress, the current is comprised of the charge current for electric double layer, current for the adsorption/desorption of hydrogen and oxygen species and for the reduction/oxidation of adsorbed sulfur species. The integrated charge for electric double layer and the adsorption/desorption of oxygen species are essentially equal in cycle n. However, the integrated charge for adsorption of hydrogen in cycle n does not equal to that for desorption of hydrogen in the same cycle, but equal to that for desorption of hydrogen in cycle n + 1, because in the range of high potential of cycle n, some sulfur species are oxidized and desorbed from the Pt surface, which causes an increase of ECA, and consequently results in an increase of the amount of integrated charge for the adsorption of hydrogen in cycle n. S^0 absorbed on the Pt nanoparticles is slowly oxidized to SO_4^{2-} and desorbs from Pt surface, and the total charge of the oxidization of S^0 in cycle n can be calculated as follows:

$$Q_{Sox}(n) = Q_{n+} - Q_{n-} - Q_{Hupd\,n} + Q_{Hupd\,n+1} \qquad (2)$$

where $Q_{Sox}(n)$ is the total charge of the oxidization of S^0 to SO_4^{2-} in cycle n, Q_{n+} is the integrated charge of positive scan of cycle n,

Table 1
Sulfur and hydrogen charge and coverage of a Pt/VC electrode over 10 voltammetric cycles.

Cycle no., n	$Q_{Hox,n}/10^{-4}C$	$Q_{Sox,n}/10^{-4}C$	$\sum_n Q_{Sox,n}/10^{-4}C$	$\theta_{H,n}$	$\theta_{S,n}$	$\theta_{H,n} + \theta_{S,n}$
1	3.46	1.04	2.41	0.58	0.40	0.98
2	4.46	0.46	1.37	0.74	0.23	0.97
3	5.19	0.28	0.92	0.86	0.15	1.02
4	5.62	0.18	0.64	0.93	0.11	1.04
5	5.84	0.13	0.45	0.97	0.07	1.05
6	5.96	0.10	0.32	0.99	0.05	1.04
7	6.01	0.07	0.22	1.00	0.04	1.04
8	6.01	0.05	0.15	1.00	0.03	1.03
9	6.01	0.05	0.10	1.00	0.02	1.02
10	5.99	0.05	0.05	1.00	0.01	1.01

Cycle no., n: cycle number of cycling process, range from 1 to 10;
$Q_{Hox,n}$: charge of hydrogen oxidization of cycle n;
$Q_{Sox,n}$: charge of sulfur oxidization of cycle n;
$\sum_n Q_{Sox,n}$: total charge of sulfur oxidization from cycle 10 to cycle n;
$\theta_{H,n}$: ECA left in cycle n;
$\theta_{S,n}$: S^0 left in cycle n.

Q_{n-} is the integrated charge of negative scan of cycle n, $Q_{Hupd\,n}$ is the integrated charge of desorption of hydrogen from 0 to 0.4 V in cycle n, $Q_{Hupd\,n+1}$ is the integrated charge of desorption of hydrogen from 0 to 0.4 V in cycle $n+1$.

The quantity of S^0 (Coulomb as the unit) that are oxidized in cycle n can be calculated as follows:

$$N_{S^0}(n) = \frac{Q_{Sox}(n)}{6} \tag{3}$$

The total amount of S^0 that are oxidized during the whole cycling process is:

$$N_{S^0}(\text{total}) = \sum N_{S^0}(n), \quad n = n_{\text{end}} \text{ to } 1 \tag{4}$$

where n_{end} is identified as the cycling in which the area of H_{upd} stops increasing as the cycling progresses.

Then the coverage of S^0 is:

$$\theta_S = \frac{N_{S^0}(\text{total})}{Q_{Hupd}} \tag{5}$$

where $N_{S^0}(\text{total})$ is the total quantity of the sulfur adsorbed on Pt surfaces, Q_{Hupd} is the ECA (Coulomb as the unit) of the Pt nanoparticles after desorbing all the sulfur species. θ_H is the remaining ECA of each cycle. The sulfur and hydrogen charge and coverage of the Pt/VC electrode over 10 voltammetric cycles are shown in Table 1.

It can be seen from Table 1 that as the cycling progresses, $\theta_{H,n}$ increases and $\theta_{S,n}$ decreases, which indicates that ECA is recovered slowly. Moreover, $\theta_{H,n} + \theta_{S,n}$ always equals to 1 in each cycle indicating that S^0 is oxidized to SO_4^{2-} and then desorbs from the Pt surface, and that Pt—S is formed at the potential of 0.05 V, or rather, one S atom takes the place of one hydrogen atom to seize one Pt site. In order to find out the range of $\theta_{H,1}$ where initially $\theta_{H,1} + \theta_{S,1} = 1$ is satisfied, we change the poisoning time while holding the electrode in the same 0.2 mM Na$_2$SO$_3$/0.5 M H$_2$SO$_4$ solution at 0.65 V. With the same experiment procedures and computational processes, we get the results listed in Table 2.

As shown in Table 2, when $\theta_{H,1}$ is greater than 0.58, $\theta_{H,1} + \theta_{S,1}$ essentially equals to 1 in error range. When the initial hydrogen coverage ($\theta_{H,1}$) is less than 0.45, $\theta_{H,1} + \theta_{S,1}$ is always lower than 1.

Table 2
Initial hydrogen and sulfur coverage as a function of poisoning time.

Poisoning time	$\theta_{H,1}$	$\theta_{S,1}$	$\theta_{H,1} + \theta_{S,1}$
0.5 min	0.81	0.16	0.97
1 min	0.72	0.24	0.96
2 min	0.58	0.40	0.98
5 min	0.45	0.45	0.90

We also increased the concentration of Na$_2$SO$_3$ in the contaminant solution to get a lower initial $\theta_{H,1}$, and we found that $\theta_{H,1} + \theta_{S,1}$ became lower with the lowering of $\theta_{H,1}$. We would like to discuss more about the results. Obviously the validity of equation $\theta_{H,1} + \theta_{S,1} = 1$ comes from the assumption that one S atom adsorbs at one Pt atom (single coordination). However, when a part of S atom adsorbs at two Pt atoms (a complex of single coordination and multiple coordination), $\theta_{H,1} + \theta_{S,1}$ would become lower than 1, since the hydrogen coverage become lower. In our experimental conditions, the concentration of Na$_2$SO$_3$ in 0.5 M H$_2$SO$_4$ is never higher than 0.5 mM, so we do not get the result that $\theta_{H,1} + \theta_{S,1} > 1$, which might be result from several S atoms adsorbs at one Pt atom. Therefore, we get a convenient way to figure out the amount of sulfur adsorbed on the Pt surface when the Pt nanoparticles are partly poisoned, i.e., holding the SO$_2$ poisoned electrode at the potential of 0.05 V for enough time, then measuring the remaining ECA of the electrode, and the sulfur coverage of Pt (θ_S) is $1 - \theta_H$ if the remaining ECA is more than half of the initial ECA ($\theta_H > 0.5$).

3.2. SO$_2$ poisoning Pt/VC towards ORR

Since we can easily figure out the amount of SO$_2$ adsorbed on the Pt nanoparticles, we can quantitatively study the effect of SO$_2$ on the performance of Pt/VC. Fig. 2 shows the ORR curves of Pt/VC
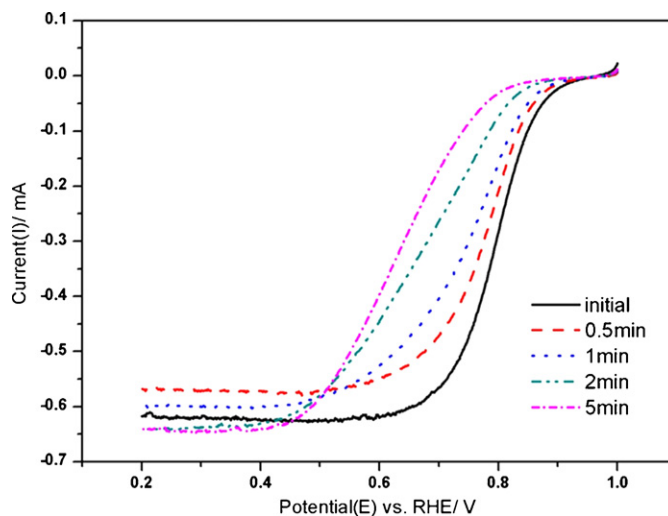

Fig. 2. Polarization curves for the ORR in 0.5 M H$_2$SO$_4$ solution of unpoisoned Pt/VC and Pt/VC covered with sulfur species as a function of poisoning time. 45.5 μg$_{Pt}$ cm^{-2}, 1600 rpm, room temperature, polarization program: 1.0 V → 0.2 V at 5 mV s^{-1}.
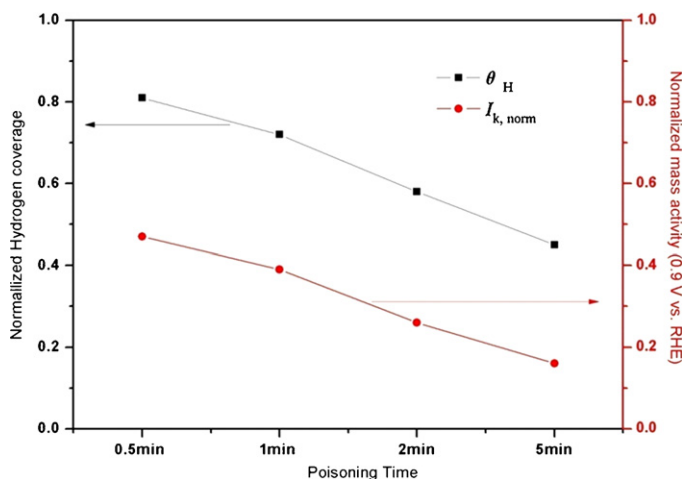
Fig. 3. Hydrogen coverage of Pt/VC after holding at 0.05 V for 10 min in 0.5 M H$_2$SO$_4$ solution after poisoning for different time in 0.2 mM Na$_2$SO$_3$/0.5 M H$_2$SO$_4$ solution and the normalized mass activity at 0.9 V vs. RHE as a function of poisoning time.

Table 3
Pt/VC mass activities at 0.9 V as a function of θ_H.

θ_H	$I_{k,norm}$
0.81	0.47
0.72	0.39
0.58	0.26
0.45	0.16

by the ECA consequently. However, when extrapolate the θ_H to 1, $I_{k,norm}$ is still lower than 1, which means that at the beginning stage of poisoning, there should be other issues besides of the decreasing ECA that cause the weakening of ORR activity. These issues might be the change of the electronic structures of Pt caused by the adsorbed SO$_2$ and the different adsorption state of SO$_2$ at the beginning stage. As a quantitatively research on the performance of SO$_2$ poisoning Pt/VC, Table 3 shows the remaining ORR activity and the remaining θ_H. When the hydrogen coverage (θ_H) is 0.81, the remaining ORR is 0.47; when the remaining θ_H is 0.58, the remaining ORR is 0.26. They are linearly dependent to each other in the range of 0.45 < θ_H < 0.81.

3.3. S^0 poisoning Pt/VC towards ORR

After holding the electrode at the potential of 0.05 V for 10 min, the SO$_2$ adsorbed at 0.65 V would be deoxidized to S^0, and one S^0 atom takes one Pt site. Since that one SO$_2$ molecule takes two Pt site at 0.65 V, the performance of ORR would be partly recovered. Fig. 5 shows the ORR curves of a Pt/VC coated electrode at three different states: unpoisoning, poisoning at 0.65 V for 1 min and holding at 0.05 V for 10 min after poisoning at 0.65 V for 1 min. It can be seen that the performance of ORR is partly recovered after deoxidized. In addition, we compare the area specific activity (ASA) of the unpoisoned Pt/VC (at 0.9 V vs. RHE) and S^0 poisoned Pt/VC, the result is shown in Fig. 6. It can be seen that the ASA of the S^0 poisoned Pt/VC is higher than the unpoisoned Pt/VC, and the ASA increases with the increase of the amount of S^0 in the referred range. The reasons for the improvement might be the electrophilic effect of S^0, which would decrease the d-band center of Pt and weaken the interaction between Pt and the OH$_{abs}$. Therefore, the reduction of the adsorbed SO$_2$ can partly recover the performance, which could be attributed to two issues. One is the recovery of the ECA, since one sulfur atom takes one Pt site when reduced, compared to the

electrodes which have been poisoned at 0.65 V for different periods of time. The ORR limiting currents might be affected by sulfur compounds covering parts of the Pt surfaces and the additional reduction current of the adsorbed SO$_2$ species in low potential range. The former would decrease the limiting currents and the latter would increase them. It can be seen that with the increase of poisoning time, the performance of Pt/VC became poorer and poorer. Fig. 3 shows the normalized initial hydrogen coverage and the normalized mass activity at 0.9 V as a function of poisoning time. In order to make figures comparable, the activity of ORR and the hydrogen coverage were normalized to those of the unpoisoned Pt/VC ($I_{k,norm}$ and θ_H). It can be seen that with the increase of poisoning time, both hydrogen coverage (θ_H) and ORR activity ($I_{k,norm}$) decrease, and they looks like parallel to each other. The relativity between ORR activity ($I_{k,norm}$) and hydrogen coverage (θ_H) was also introduced by curve fitting method in Fig. 4. It can be seen that $I_{k,norm}$ and θ_H are linearly dependent, which means that when the θ_H lies in the range between 0.45 and 0.85, the performance is linearly determined by the amount of SO$_2$ adsorbed. As we already know that at the potential of 0.65 V, one SO$_2$ molecule seizes two Pt sites, so the ECA left might be linearly dependent on the amount of SO$_2$ absorbed, and the activity of ORR are linearly determined
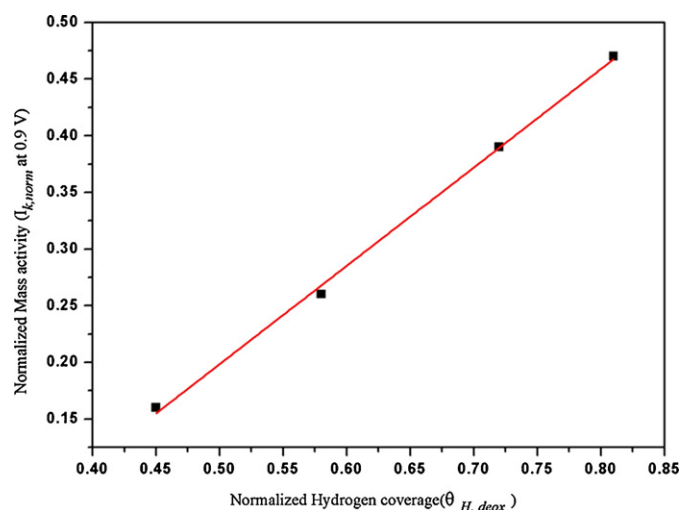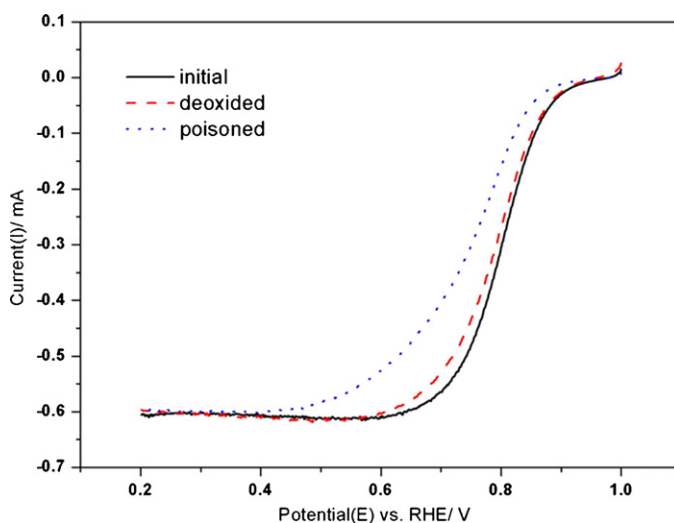

Fig. 4. Fitting curve of the relationship between normalized mass activity at 0.9 V and hydrogen coverage.

Fig. 5. Polarization curves for the ORR in 0.5 M H$_2$SO$_4$ solution of unpoisoned Pt/VC and Pt/VC covered by the same amount of SO$_2$ and S^0. 45.5 µg$_{Pt}$ cm^{-2}, 1600 rpm, room temperature, polarization program: 1.0 V → 0.2 V at 5 mV s^{-1}.
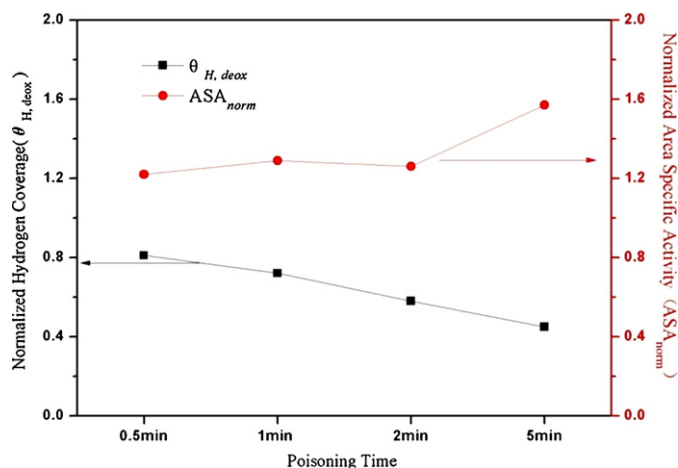
Fig. 6. Normalized area specific activity (0.9 V vs. RHE) and hydrogen coverage after deoxidization as a function of poisoning time.

SO_2 adsorbed Pt/VC catalysts where one SO_2 molecule takes two Pt sites; and the other is the increasing area specific activity of the uncovered electrochemical active area. However, the increase of the ASA could not balance the negative effect of S^0 covering parts of the Pt surfaces, so the ORR performance could not be fully recovered as a whole.

4. Conclusion

(1) A convenient method to measure the amount of SO_2 adsorbed on the Pt nanoparticles is proposed in this paper. Holding the SO_2 poisoned Pt/VC at 0.05 V for enough time, if the hydrogen coverage of Pt (θ_H) lies in the range between $0.5 < \theta_H < 1$, then the sulfur coverage (θ_{S^0}) is $1 - \theta_H$.

(2) A quantitative study of the impacts of adsorbed sulfur on the performance of Pt/VC toward ORR is carried out in this paper. When $0.45 < \theta_H < 0.81$, the activity of ORR decreases linearly with the amount of SO_2 adsorbed, and at the beginning stage of the poisoning process ($\theta_H \rightarrow 1$), some other negative factors besides of the lessening ECA cause the weakening of the ORR.

(3) The deoxidization of the adsorbed SO_2 could partly recover the performance, which could be attributed to the recovery of electrochemical active area and the improvement of the area specific activity of the uncovered Pt sites.

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