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Gd_{0.2}Ce_{0.8}O₂ 包覆 LaNi_{0.6}Fe_{0.4}O_{3.8} 阴极制备及性能

任睿轩, 黄波*, 朱新坚, 胡一星, 丁小益, 刘宗尧, 刘烨彬

(上海交通大学 机械与动力工程学院, 燃料电池研究所, 上海 200240)

摘要: 应用丝网印刷和共烧结制备 LaNi_{0.6}Fe_{0.4}O_{3.8}/Sc_{0.1}Zr_{0.9}O_{1.95}/LaNi_{0.6}Fe_{0.4}O_{3.8} 对称电池. 以硝酸铈和硝酸钕为原料, 柠檬酸作燃料, 燃烧合成 Gd_{0.2}Ce_{0.8}O₂(GDC)包覆的 LaNi_{0.6}Fe_{0.4}O_{3.8}(LNF)阴极. 实验表明, 在 750 °C 工作温度下, 纯 LaNi_{0.6}Fe_{0.4}O_{3.8} 阴极的极化电阻为 0.70 Ω·cm², 而 21.3% (by mass, 下同, 如无特殊标注均为质量分数)GDC 包覆的 LNF-GDC 复合阴极的极化电阻最小(0.13 Ω·cm²), 活化能最低(136.80 kJ·mol⁻¹), 故其阴极性能最佳. GDC 的包覆加速了气体/阴极/电解质三相界面反应区的扩散过程, 降低了阴极极化电阻.

关键词: 固体氧化物燃料电池; LaNi_{0.6}Fe_{0.4}O_{3.8} 阴极; Gd_{0.2}Ce_{0.8}O₂ 包覆; 极化电阻; 交流阻抗

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在固体氧化物燃料电池 (Solid Oxide Fuel Cell, SOFC) 的发展过程中, 人们越来越认识到降低电池工作温度的重要性. 若能将电池工作温度降低到中温 (600 ~ 800 °C), 则能提高电极的稳定性, 减小热应力, 延长电池寿命, 还可使用廉价的金属合金作为电池的双极板材料^[1-3]. Cr 基合金因具有成本低、易加工、电子电导率和热导率高、机械稳定性高、耐高温以及抗氧化等优点而成为最有前景的双极板材料^[4-6]. Cr 基合金用于 SOFC 的双极板时, 高价态 Cr 化合物易挥发. 而低导电性的 Cr₂O₃(s) 在阴极/电解质界面的生成与聚集又会降低 SOFC 的电输出性能^[7-10], 且 CrO₃(g) 和 Cr₂O₃(s) 可与阴极材料如 (La,Sr)MnO₃ 反应生成绝缘的尖晶石相 (Cr,Mn)₃O₄(s), 即发生阴极“Cr 中毒”现象^[11], 使电池性能急剧下降.

据 Chiba 等^[12]报道, LaNi_{0.6}Fe_{0.4}O_{3.8}(LNF) 800 °C 电导率为 580 S·cm, 为传统阴极 La_{0.8}Sr_{0.2}MnO₃(LSM) (180 S·cm) 的 3 倍, 其室温 ~ 1000 °C 热膨胀系数为 11.4 × 10⁻⁶ K⁻¹, 更接近于电解质 YSZ (Yttria Stabilized Zirconia, Y_{0.08}Zr_{0.92}O_{1.95}) 的热膨胀系数 10.0 × 10⁻⁶ K⁻¹. Zhen 等^[13]研究也表明 LNF 比 LSM 具有更稳定的电化学性能, Fe-Cr 合金存在时, 其界面上没有 Cr 沉积, LNF 有抗 Cr 毒化作用. 然在相同的烧结温度 LNF 较 LSM 更易与 ZrO₂ 基电解质反应^[14-15], 如高于 1000 °C 时 LNF 与 ZrO₂ 基电解质反应生成

绝缘的 La₂Zr₂O₇, 降低电池性能. 由于 LNF 阴极材料在工作温度下的再氧化以及随后的氧空位浓度的下降^[16], 致使 LNF 阴极的初始性能下降. Stodolny 等^[17-18]研究发现, 由于 CrO₃(g) 或 CrO₂(OH)₂(g) 等挥发性的物质与阴极 LaNi_{0.6}Fe_{0.4}O_{3.8} 发生反应而使其欧姆电阻和极化电阻增加. 本文应用燃烧合成法, 在已烧结的 LNF 阴极表面包覆一层 Gd_{0.2}Ce_{0.8}O₂(GDC)纳米颗粒制成 LNF-GDC 复合阴极, 该阴极的导电性降低不显著, 可阻止 LNF 与电解质 ScSZ (Scandia Stabilized Zirconia, Sc_{0.1}Zr_{0.9}O_{1.95}) 生成绝缘的 La₂Zr₂O₇, 以期增大阴极材料-电解质材料-反应气体三相界面, 改善 LNF 阴极性能.

1 实验

1.1 LNF-GDC 复合阴极的制备

按 La:Ni:Fe = 1:0.6:0.4 (by mole) 称取一定量的 La(NO₃)₃·6H₂O、Ni(NO₃)₂ 和 Fe(NO₃)₃·9H₂O 配成混合溶液, 加入一定比例的柠檬酸 (柠檬酸与硝酸盐的化学配比计算方法参见文献^[19]), 用氨水将混合液调至中性, 将其 120 °C 加热蒸发, 得褐色干凝胶, 置于坩埚电炉 (300 °C 预热) 燃烧, 升温至 600 °C 煅烧 2 h 可得黑色 LaNi_{0.6}Fe_{0.4}O_{3.8} (LNF) 粉体材料. 详细的粉体制备方法参见文献^[20].

将 LNF 阴极浆料通过丝网印刷覆盖于 ScSZ 片 (厚约 200 μm) 两侧, 1050 °C 煅烧 2 h 印制成

LNF/ScSZ/LNF 对称电池 (面积为 $1\text{ cm} \times 1\text{ cm} = 1\text{ cm}^2$).

GDC 溶液(见文献^[21]),按 $\text{Gd}:\text{Ce} = 1:4$ (by mole)取一定量的 $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ 和 $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ 与一定化学计量比的柠檬酸溶于蒸馏水中,配制成 Gd^{3+} 浓度为 $0.4\text{ mol} \cdot \text{L}^{-1}$ 的溶液.将上述 LNF 阴极浸入此溶液,在低于 200 MPa 真空度下保持 30 min ,使溶液充分浸润 LNF,又将其移置于烘箱 $75\text{ }^\circ\text{C}$ 烘干,移入预热至 $450\text{ }^\circ\text{C}$ 的坩埚电炉内即升温至 $600\text{ }^\circ\text{C}$,保温 2 h .按上述步骤重复数次,即得 $\text{Gd}_{0.2}\text{Ce}_{0.8}\text{O}_2$ (GDC)($\leq 50\text{ nm}$) 包覆的 LNF 对称电池,铂网为电流收集体.

1.2 LNF-GDC 复合阴极的表征

采用阿基米德排水法测试 GDC 包覆的 LNF 阴极样品的孔隙率,使用 Solartron 1260 阻抗仪测试电池交流阻抗 (开路),测试频率 $10\text{ mHz} \sim 100\text{ kHz}$,交流阻抗输入信号 20 mV ,工作温度: $650\text{ }^\circ\text{C}$ 、 $700\text{ }^\circ\text{C}$ 、 $750\text{ }^\circ\text{C}$ 、 $800\text{ }^\circ\text{C}$ 和 $850\text{ }^\circ\text{C}$.使用扫描电子显微镜(SEM,PHILIPS 515 型)观察材料微观形貌.

2 结果与讨论

2.1 LNF-GDC 复合阴极表征

表 1 给出不同 GDC 包覆量的 LNF-GDC 复合阴极的孔隙率.可以看出,纯 LNF 阴极的孔隙率为 38.1% ,经 1 次包覆后,其孔隙率为 32.2% ,GDC 包覆量为 4.7% (5.9% , by volume). 包覆次数增加, GDC 包覆量也逐渐递增,其阴极孔隙率降低. 经 6 次包覆 GDC,其阴极孔隙率降为 0.05% .

表 1 不同 GDC 包覆量的 LNF-GDC 复合阴极的孔隙率参数
Tab. 1 Porosity and GDC loading of the impregnated LNF cathode

Coating cycle	0	1	2	4	5	6
Porosity/%	38.1	32.2	24.6	13.1	6.2	0.05
Loading/% (by mass)	0	4.7	8.1	15.9	21.3	26.9
Loading/% (by volume)	0	5.9	13.5	25.0	31.9	38.1

图 1 示出 21.3% (A)和 26.9% (B) GDC 包覆量的 LNF-GDC 复合阴极的 SEM 照片.可以看出,包覆量 21.3% 时,GDC 颗粒覆盖于 LNF 颗粒表面,彼此联通,形成网络. 包覆量增至 26.9% 时,GDC 颗粒堵塞阴极气孔,使阴极孔隙率下降.

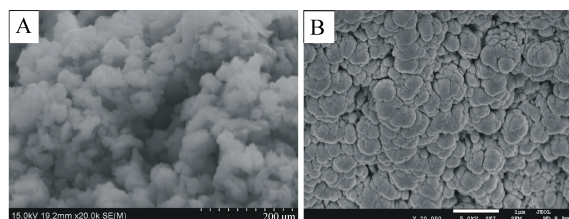


图 1 21.3% (A)和 26.9% (B) GDC 包覆量的 LNF-GDC 复合阴极的 SEM 照片

Fig. 1 SEM images of 21.3% (A) and 26.9% (B) GDC-impregnated LNF-GDC composite cathodes

2.2 LNF-GDC 复合阴极电化学性能

图 2 给出不同 GDC 包覆量的 LNF-GDC 复合阴极分别在 $750\text{ }^\circ\text{C}$ (A)和 $800\text{ }^\circ\text{C}$ (B)工作温度下的交流阻抗谱图和等效电路图(C). 由图可知,该阻抗谱图是由高频弧和低频弧组成. 其高频弧对应于阴极/电解质界面的电化学反应过程 (界面极化电阻或电荷传递电阻 R_{ct}),低频弧对应于气体扩散过程 (浓差极化电阻 R_d),阴极极化电阻 $R_p = R_{ct} + R_d$ ^[22-25], R_{Ω} 为对称电池的欧姆电阻. 随 GDC 包覆量增加,其阴极的电荷传递电阻 R_{ct} 变化甚微,而浓差极化电阻 R_d 渐减,欧姆电阻 R_{Ω} 几乎不变. 包覆量增至 26.9% ,欧姆电阻 R_{Ω} 显著增加,阴极浓差极化电阻 R_d 也递增. $650\text{ }^\circ\text{C}$ 、 $700\text{ }^\circ\text{C}$ 以及 $850\text{ }^\circ\text{C}$ 工作温度下阴极的交流阻抗谱图具有同样的趋势.

图 3 和图 4 分别给出不同工作温度下 GDC 包覆量与对称电池的欧姆电阻 R_{Ω} 和阴极极化电阻 R_p 的关系图. 由图发现,同工作温度下 GDC 包覆量由 0 增至 21.3% ,其对称电池的欧姆电阻几乎不变,而阴极极化电阻逐渐降低. 显然,GDC 包覆极大地改善了该电极的电化学性能,GDC 的存在加速气体/阴极/电解质三相界面反应区的扩散过程,降低阴极极化电阻^[26]. GDC 包覆量为 21.3% 的 LNF-GDC 复合阴极,其极化电阻最低,在 $850\text{ }^\circ\text{C}$ 、 $800\text{ }^\circ\text{C}$ 、 $750\text{ }^\circ\text{C}$ 、 $700\text{ }^\circ\text{C}$ 和 $650\text{ }^\circ\text{C}$ 工作温度其值分别为 $0.03\text{ }\Omega \cdot \text{cm}^2$ 、 $0.06\text{ }\Omega \cdot \text{cm}^2$ 、 $0.13\text{ }\Omega \cdot \text{cm}^2$ 、 $0.29\text{ }\Omega \cdot \text{cm}^2$ 和 $1.47\text{ }\Omega \cdot \text{cm}^2$; 而纯 LNF 阴极的极化电阻在相应工作温度下分别为 $0.12\text{ }\Omega \cdot \text{cm}^2$ 、 $0.28\text{ }\Omega \cdot \text{cm}^2$ 、 $0.70\text{ }\Omega \cdot \text{cm}^2$ 、 $1.91\text{ }\Omega \cdot \text{cm}^2$ 和 $5.62\text{ }\Omega \cdot \text{cm}^2$. $750\text{ }^\circ\text{C}$ 时,GDC 包覆量为 21.3% 的 LNF-GDC 复合阴极的极化电阻($0.13\text{ }\Omega \cdot \text{cm}^2$)比纯 LNF 阴极的极化电阻($0.70\text{ }\Omega \cdot \text{cm}^2$)降低了大约 4 倍. 但当包覆量增至 26.9% ,其欧姆电阻急剧增加,阴极极化电阻亦

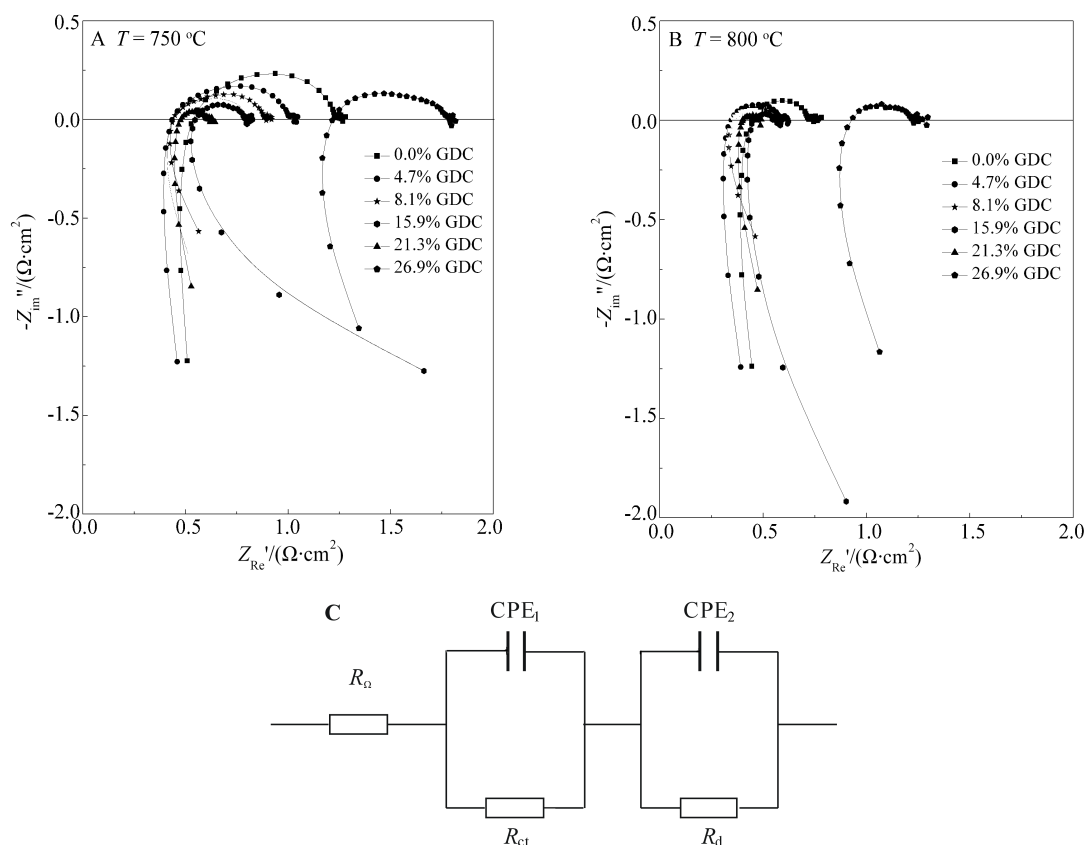


图2 不同 GDC 包覆量的 LNF-GDC 复合阴极在工作温度 750 °C(A)和 800 °C(B)的电化学阻抗谱图及等效电路图(C)

Fig. 2 Electrochemical impedance spectra of the LNF-GDC composite cathode with different contents of GDC measured at 750 °C(A), 800 °C (B) in air and the equivalent circuit for data fitting (C)

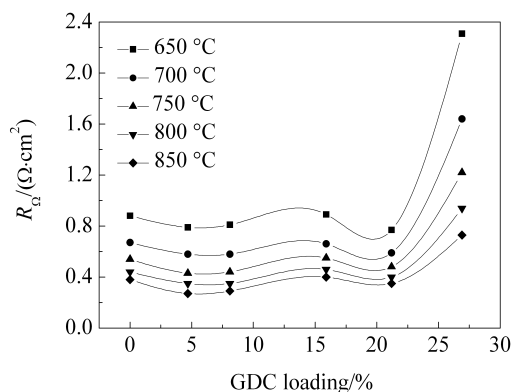


图3 不同温度下测得对称电池欧姆电阻与 GDC 包覆量变化的示意图

Fig. 3 Plots of ohmic resistance of symmetrical cell versus the contents of GDC obtained at different temperatures

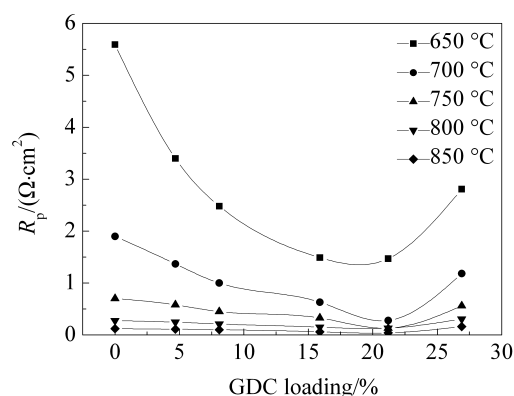


图4 不同温度下测得对称电池阴极极化电阻与 GDC 包覆量变化的示意图

Fig. 4 Plots of polarization resistance of LNF-GDC composite cathodes versus the contents of GDC obtained at different temperatures

增加. 这是因为, GDC 是氧离子导体, 26.9%GDC 包覆的 LNF-GDC 复合阴极的孔隙率接近于 0, 导致欧姆电阻增加、气相传输过程减弱和浓差极化增

加. GDC 包覆量 21.3%的 LNF-GDC 复合阴极有最低的阴极极化电阻值, 这源于离子导体和电子导体达到适当的比率.

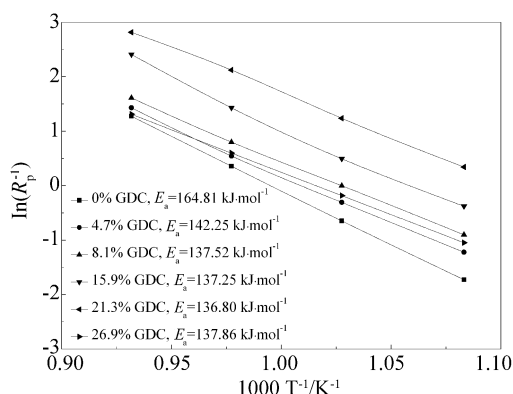


图 5 不同 GDC 包覆量 LNF-GDC 复合阴极的阿伦尼乌斯曲线

Fig. 5 Arrhenius curves of LNF-GDC composite cathodes with different contents of GDC

图 5 给出不同 GDC 包覆量的 LNF-GDC 复合阴极的阿伦尼乌斯曲线, 据式:

$$\frac{1}{R_p} = A \cdot \exp\left(-\frac{E_a}{kT}\right) \quad (1)$$

其中, A 为常数, k 为波尔兹曼常数, E_a 为活化能, R_p 为极化电阻。从阿伦尼乌斯曲线斜率可得活化能, 纯 LNF 阴极的活化能为 $164.81 \text{ kJ} \cdot \text{mol}^{-1}$, 4.7% GDC、8.1% GDC、15.9% GDC、21.3% GDC、26.9% GDC 包覆的 LNF 复合阴极的活化能分别为 $142.25 \text{ kJ} \cdot \text{mol}^{-1}$ 、 $137.52 \text{ kJ} \cdot \text{mol}^{-1}$ 、 $137.25 \text{ kJ} \cdot \text{mol}^{-1}$ 、 $136.80 \text{ kJ} \cdot \text{mol}^{-1}$ 和 $137.86 \text{ kJ} \cdot \text{mol}^{-1}$ 。其中 21.3% GDC 包覆的 LNF-GDC 复合阴极的活化能最小, 其性能最佳。

3 结 论

由燃烧合成法在已烧结的 LNF 阴极表面包覆 GDC 制成的 LNF-GDC 复合阴极, 随 GDC 包覆量 ($\leq 21.3\%$) 增加, 其欧姆电阻几乎不变, 极化电阻显著减小; 21.3% GDC 包覆其颗粒相互接触形成联通网络, 而 GDC 包覆量增至 26.9% 其颗粒堵塞阴极气孔, 欧姆电阻显著增加。21.3% GDC 包覆的 LNF-GDC 复合阴极性能最佳。在 850°C 、 800°C 、 750°C 、 700°C 和 650°C 工作温度其极化电阻分别为 $0.03 \Omega \cdot \text{cm}^2$ 、 $0.06 \Omega \cdot \text{cm}^2$ 、 $0.13 \Omega \cdot \text{cm}^2$ 、 $0.29 \Omega \cdot \text{cm}^2$ 和 $1.47 \Omega \cdot \text{cm}^2$, 纯 LNF 阴极的极化电阻分别为 $0.12 \Omega \cdot \text{cm}^2$ 、 $0.28 \Omega \cdot \text{cm}^2$ 、 $0.70 \Omega \cdot \text{cm}^2$ 、 $1.91 \Omega \cdot \text{cm}^2$ 和 $5.62 \Omega \cdot \text{cm}^2$ 。750 $^\circ\text{C}$ 工作温度时, 21.3% GDC 包覆量的 LNF-GDC 复合阴极极化电阻较纯 LNF 阴极的极化电阻降低约 4 倍, 其活化能为 136.80

$\text{kJ} \cdot \text{mol}^{-1}$, 阴极电化学性能最佳。

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Fabrication and Performance of $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ Cathode Modified by Coating with $\text{Gd}_{0.2}\text{Ce}_{0.8}\text{O}_2$ for Intermediate Temperature Solid Oxide Fuel Cell

REN Rui-xuan, HUANG Bo*, ZHU Xin-jian, HU Yi-xing, DING Xiao-yi,
LIU Zong-yao, LIU Ye-bin

(*Institute of Fuel Cell, School of Mechanical Engineering, Shanghai Jiaotong University,
Shanghai 200240, China*)

Abstract: The symmetric cell of $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}/\text{Sc}_{0.1}\text{Zr}_{0.9}\text{O}_{1.95}/\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ was fabricated with screen printing method. A $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ (LNF) cathode was modified by coating with nano-sized gadolinium-doped ceria (GDC, $\text{Gd}_{0.2}\text{Ce}_{0.8}\text{O}_2$) prepared using a simple combustion process within the pores of the cathode. According to the electrochemical impedance spectra (EIS), the polarization resistance of the pure LNF was $0.70 \Omega \cdot \text{cm}^2$ at 750°C , while $0.13 \Omega \cdot \text{cm}^2$ for the 21.3%GDC (by mass)-coated LNF cathode at the same temperature, which was only 1/5 of that of the pure LNF cathode. The activation energy of the 21.3% GDC (by mass)-coated LNF cathode ($136.80 \text{ kJ} \cdot \text{mol}^{-1}$) is the smallest among those of GDC-coated LNF cathodes with different contents of GDC. The 21.3% GDC (by mass)-coated LNF cathode showed the optimum performance. The results indicated that GDC coatings significantly affected electrocatalytic activity of the LNF cathodes towards O_2 reduction reaction. The improved performance of GDC-coated LNF cathode was attributed to the extended triple-phase boundary (TPB) and enhanced ion conductivity of oxide.

Key words: solid oxide fuel cell; $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ cathode; $\text{Gd}_{0.2}\text{Ce}_{0.8}\text{O}_2$ coating; polarization resistance; electrochemical impedance spectroscopy