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Fabrication and Impedance Performance of Gradient $LaNi_{0.6}Fe_{0.4}O_{3}$-î´-$Gd_{0.2}Ce_{0.8}O_{2}$ Composite Cathodes for Intermediate Temperature Solid Oxide Fuel Cell

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中温固体氧化物燃料电池$LaNi_{0.6}Fe_{0.4}O_{3-\delta}$-$Gd_{0.2}Ce_{0.8}O_2$梯度复合阴极制备及交流阻抗性能

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摘要: 应用丝网印刷和共烧结制备 $LaNi_{0.6}Fe_{0.4}O_{3-\delta}$(LNF)-$Gd_{0.2}Ce_{0.8}O_2$(GDC)梯度复合阴极/$Gd_{0.2}Ce_{0.8}O_2$/$Sc_{0.1}Zr_{0.9}O_{1.95}$(ScSZ)/$Gd_{0.2}Ce_{0.8}O_2$/$LaNi_{0.6}Fe_{0.4}O_{3-\delta}$(LNF)-$Gd_{0.2}Ce_{0.8}O_2$(GDC),组成梯度复合阴极对称电池. 实验表明,在 750 ℃ 工作温度下单层 70%LNF-30%GDC（文中均指质量百分比）复合阴极的极化电阻为 0.581 Ω·cm², 而三层 60%LNF-40%GDC/70%LNF-30%GDC/100%LNF 复合阴极的极化电阻最小（0.452 Ω·cm²). 由于阴极组成在 ScSZ 电解质和 LNF 阴极之间呈梯度变化,因此获得了最佳的阴极/电解质界面,大大加快了三相界面或气体/阴极/电解质三相接触点反应区的扩散,其电荷传递电阻 R_{ct} 和浓差极化电阻 R_d 均减小,因而具有最低的阴极极化电阻值.

关键词: 固体氧化物燃料电池; $LaNi_{0.6}Fe_{0.4}O_{3-\delta}$ 阴极; 梯度阴极; 极化电阻; 交流阻抗

中图分类号: O646　　**文献标识码:** A

中温（≤ 800 °C）固体氧化物燃料电池（Intermediate Temperature Solid Oxide Fuel Cell, IT-SOFC）使用廉价的金属合金作为电池的双极板材料,可使其稳定运行,提高电极的稳定性、减小热应力和延长电池寿命[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. 低导电性的 Cr_2O_3(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 CrO_3(g)和 Cr_2O_3(s)可与传统阴极材料如(La,Sr)MnO_3[10-11]、(La,Sr)(Co,Fe)O_3[12]反应生成绝缘的尖晶石相$(Cr,Mn)_3O_4$(s)、$(Sr,Cr)_3O_4$(s),即发生阴极“Cr 中毒”[12-13],使电池性能急剧下降. 据 Chiba 等[14]报道 $LaNi_{0.6}Fe_{0.4}O_{3-\delta}$(LNF)800 °C 电导率为 580 S·cm,为传统阴极 $La_{0.8}Sr_{0.2}MnO_3$(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 YSZ 电解质（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[15]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. 然而,在相同的烧结温度 LNF 较 LSM 更易与 ZrO_2 基电解质反应[16-17], 如高于 1000 ℃ 时 LNF 与 ZrO_2 基电解质反应生成绝缘的 $La_2Zr_2O_7$, 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之的氧空位浓度的下降[18],致使 LNF 阴极的初始性能下降. 因此, LNF 作为极具应用前景的中低温阴极材料, 其性能还需要进一步改善.

本文采用 LNF 构筑梯度结构设计, 将目前已知的催化性能优良、电子电导率高、物理化学稳定性优异的阴极材料 $LaNi_{0.6}Fe_{0.4}O_{3-\delta}$(LNF)和催化性能优良、具有突出的氧离子储存、释放和传输能力的氧化钆掺杂 CeO_2($Gd_{0.2}Ce_{0.8}O_2$、GDC)这两种材料组合起来. 使其对高性能 SOFC 阴极的多种要求由不同的组成梯度阴极层来承担, 并使各层间相互配合,利用协同作用达到优势互补的效果. 如此,可显著改善 LNF 组成梯度复合阴极的氧气还原反应动力学,从而达到提高 SOFC 性能的目的.

1 实　验

1.1 LNF 基梯度阴极的制备

按摩尔比 La:Ni:Fe = 1:0.6:0.4 称取一定量的 $La(NO_3)_3 \cdot 6H_2O$、$Ni(NO_3)_2$ 和 $Fe(NO_3)_3 \cdot 9H_2O$ 配成混合溶液,加入一定比例的柠檬酸(参见文献[19]),用

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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 $LaNi_{0.6}Fe_{0.4}O_{3-\delta}$(LNF)粉体材料. 详细的粉体制备方法参见文献[20].

GDC 溶液(见文献[21]):按摩尔比 Gd:Ce = 1:4 取一定量的 $Gd(NO_3)_3 \cdot 6H_2O$ 和 $Ce(NO_3)_3 \cdot 6H_2O$ 与一定化学计量比的柠檬酸溶于蒸馏水中(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. 将上述溶液置于烘箱 75 °C 烘干, 移入预热至 450 °C 的坩埚电炉内即升温至 600 °C, 保温 2 h 可得淡黄色的 $Gd_{0.2}Ce_{0.8}O_2$(GDC)粉体材料.

将 GDC 浆料通过丝网印刷覆盖于 ScSZ 片(厚约 200 μm) 两侧,1300 °C 煅烧 2 h 印制成 GDC/ScSZ/GDC,其中 GDC 层厚约 2 μm.

按质量比 LNF:GDC = 70:30 称取一定量的 LNF 和 GDC 粉体的混合物, 与松油醇混合成浆料,经丝网印刷覆盖于 GDC 层两侧并于 110 °C 干燥 0.5 h. 将得到的 70%LNF-30%GDC 复合阴极的坯体在 1050 °C 烧结 2 h, 即得到 70%LNF-30%GDC/GDC/ScSZ/GDC/70%LNF-30%GDC 对称电池,70%LNF-30%GDC 复合阴极记为 Cathode A, 厚度 30 μm.

按质量比 LNF:GDC = 60:40 称取一定量的 LNF 和 GDC 粉体的混合物, 与松油醇混合成浆料,经丝网印刷覆盖于 GDC 层两侧并于 110 °C 干燥 0.5 h. 然后将 LNF 粉体与松油醇混合成浆料, 经丝网印刷法沉积在 LNF-GDC 两侧并于 110 °C 干燥 0.5 h. 最后,将得到的 60%LNF-40%GDC/100%LNF梯度阴极的坯体在 1050 °C 烧结 2 h, 即得 100%LNF/60%LNF-40%GDC/GDC/ScSZ/GDC/60%LNF-40%GDC/100%LNF 对称电池,60%LNF-40%GDC/100%LNF 复合阴极标记为 Cathode B, 其中 60%LNF-40%GDC 层 10 μm,LNF 层 20 μm.

按质量比 LNF:GDC = 60:40 称取一定量的 LNF 和 GDC 粉体的混合物, 与松油醇混合成浆料,经丝网印刷覆盖于 GDC 层两侧并于 110 °C 干燥 0.5 h. 然后按质量比 LNF:GDC = 70:30 称取一定量的 LNF 和 GDC 粉体的混合物,与松油醇混合成浆料, 经丝网印刷覆盖于 60%LNF-40%GDC 层两侧并于 110 °C 干燥 0.5 h. 然后将 LNF 粉体与松油醇混合成浆料, 经丝网印刷法沉积在 70%LNF-30%GDC 两侧并于 110 °C 干燥 0.5 h. 最后, 将得到的 60% LNF-40%GDC/70%LNF-30%GDC/100%LNF 梯度阴极的坯体在 1050 ℃ 烧结 2 h, 即得 100%LNF/70%LNF-30%GDC/60%LNF-40%GDC/GDC/ScSZ/GDC/60%LNF-40%GDC/70%LNF-30%GDC/100%LNF 对称电池,60%LNF-40%GDC/70%LNF-30%GDC/100%LNF 复合阴极标记为 Cathode C, 其中 60%LNF-40%GDC 层 10 μm,70%LNF-30%GDC 层 10 μm,LNF 层 10 μm. 含有三种阴极 Cathode A、Cathode B 和 Cathode C 的对称电池的结构示意图如图 1 所示.

1.2 LNF 基梯度阴极的表征

使用 Solartron 1260 阻抗仪测试电池交流阻抗(开路),测试频率 10 mHz ~ 100 kHz,交流阻抗输入信号 20 mV, 工作温度为 650 °C、700 °C、750 °C、800 °C 和 850 °C. 使用扫描电子显微镜(SEM, S4800 型,Japan)观察材料微观形貌.

2 结果与讨论

图 2 给出 LNF 基梯度阴极 (Cathode A、B、C) 分别在 750 ℃(A)和 800 ℃(B)工作温度下的交流阻抗谱图和等效电路图(C). 为清晰地表示 LNF 基梯度阴极的阴极极化行为的差异,对称电池的欧姆

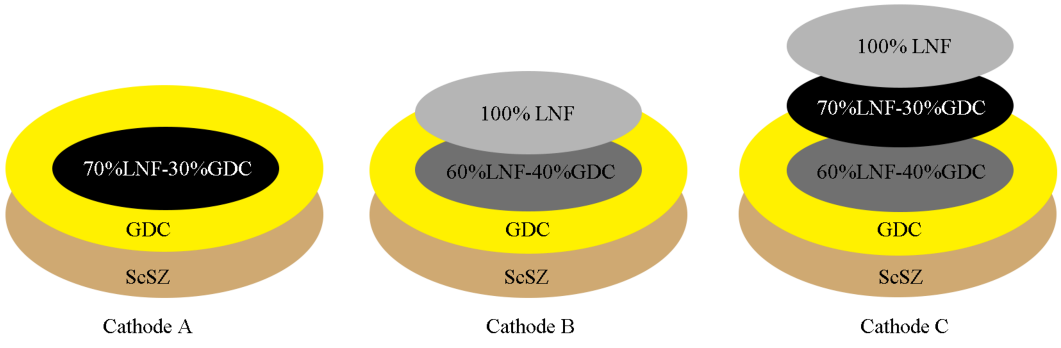

图 1 分别含有三种阴极 Cathode A、Cathode B 和 Cathode C 对称电池的结构示意图

Fig. 1 Schematic diagrams of one side of the symmetric cell structure for Cathode A, Cathode B, and Cathode C

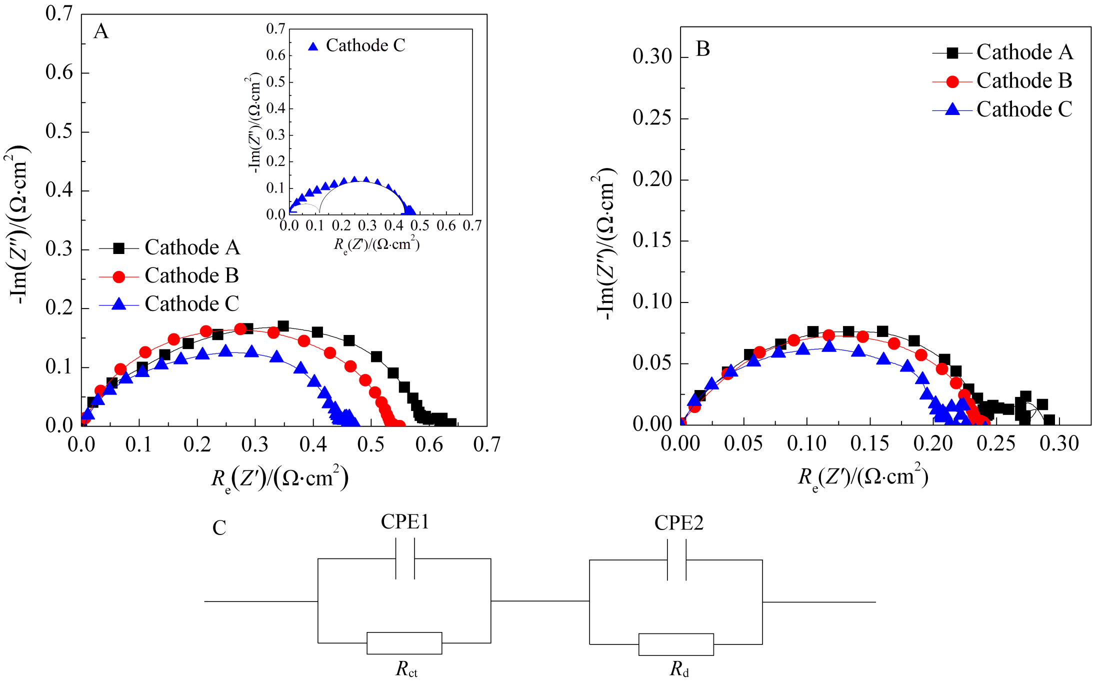

图 2 LNF 基阴极工作温度 750 °C(A)和 800 °C(B)的电化学阻抗谱图及等效电路图(C)
图 2A 插图显示 Cathode C 的交流阻抗的实测值和拟和值的比较

Fig. 2 Electrochemical impedance spectroscopic curves of LNF-based cathodes measured at 750 °C (A), 800 °C (B) in air at open circuit and the equivalent circuit for data fitting (C) The inset in Fig. 2A shows the measured impedance spectrum of Cathode C compared to that calculated using the equivalent circuit (C)

电阻 R_Ω 暂未标出. 由图 2 可知，该阻抗谱图是由高频弧和低频弧组成. 其高频弧相应阴极/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})，低频弧相应气体扩散过程(浓差极化电阻 R_d)，阴极极化电阻 $R_p = R_{ct} + R_d$[22-25]. 随阴极层数的增加，其阴极的电荷传递电阻 R_{ct} 和浓差极化电阻 R_d 皆渐减，相应地阴极极化电阻也皆渐减. 650 ℃、700 ℃ 以及 850 ℃ 工作温度下 LNF 梯度阴极的交流阻抗谱图也有同样的趋势.

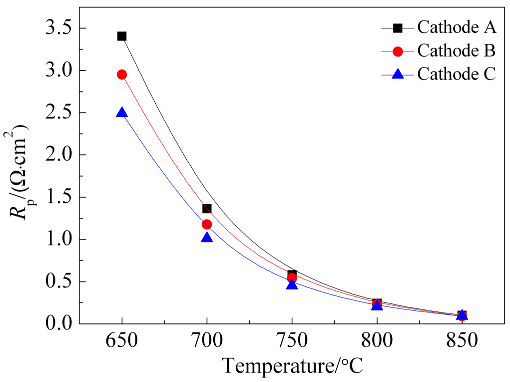

图 3 LNF 基阴极极化电阻-工作温度曲线

Fig. 3 Polarization resistance of LNF-based cathodes versus working temperature

图 3 和图 4 分别给出 LNF 基阴极(Cathode A、B、C)极化电阻、电荷传递电阻 R_{ct} 和浓差极化电阻 R_d-工作温度曲线. 从图中发现，相同工作温度下 LNF 基阴极层数由 1 层增至 3 层，其阴极极化电阻逐渐降低. 单层阴极 70%LNF-30% GDC (Cathode A)，其在 850 ℃、800 ℃、750 ℃、700 ℃ 和 650 ℃ 工作温度时的极化电阻分别为 0.101、0.242、0.581、1.363、3.405 Ω·cm². 两层阴极 60%LNF-40%GDC/100%LNF(Cathode B)的极化电阻在相应工作温度下分别减至 0.093、0.230、0.541、1.176、2.951 Ω·cm²，相对于 Cathode A 其极化电阻减小 5%～15%. 而三层阴极 60%LNF-40%GDC/70%LNF-30%GDC/100% LNF(Cathode C)的极化电阻在相应工作温度下又进一步分别降至 0.091、0.202、0.452、1.009 和 2.489 Ω·cm²，相对于 Cathode A 其极化电阻减小 10%～30%(图 3). LNF 基阴极的浓差极化电阻 R_d 皆远大于电荷传递电阻 R_{ct}，说明浓差极化电阻 R_d 在阴极极化电阻 R_p

中居于支配地位. 随阴极层数的增加，其阴极的电荷传递电阻 R_{ct} 和浓差极化电阻 R_d 皆渐减(图 4).

图 5 示出经 1050 °C 煅烧后含有三种阴极(Cathode A、B、C)的对称电池纵截面的 SEM 形貌照片. 可以看出，对于 70%LNF-30%GDC/GDC/ScSZ，阴极层与电解质层的界面结合不紧密; 随阴极层数的增加，对于 100%LNF/60%LNF-40%GDC/GDC/ScSZ，界面结合趋于紧密; 增至三层，对于 100% LNF/70%LNF-30%GDC/60%LNF-40%GDC/GDC/ScSZ，阴极层与电解质层的界面结合更加紧密. 显然，阴极层数的增加改善了阴极/电解质界面的微观结构，电荷传递电阻 R_{ct} 减小，也加速气体/阴极/电解质三相界面反应区的扩散过程，浓差极化电阻 R_d 同样减小. 因此，降低了阴极极化电阻，极大地改善阴极的电化学性能. 综上所述，三层阴极 Cathode C 中 ScSZ 电解质和 LNF 阴极间的组成呈梯度变化，可获得最佳的阴极/电解质界面，大大加快了三相界面或气体/阴极/电解质三相接触点反应区的扩散，电荷传递电阻 R_{ct} 和浓差极化电阻 R_d 均减小，故其阴极极化电阻值最低.

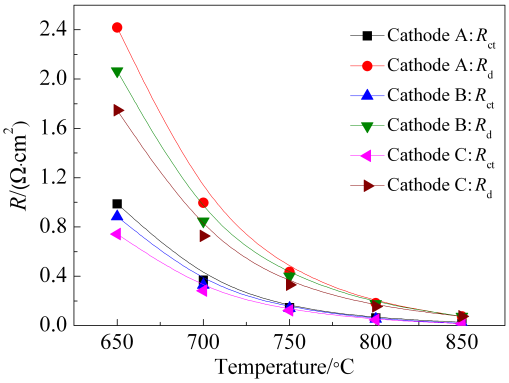

图 4 LNF 基阴极电荷传递电阻 R_{ct} 和浓差极化电阻 R_d-工作温度曲线

Fig. 4 Charge transfer resistance (R_{ct}) and gas phase diffusion resistance (R_d) of LNF-based cathodes versus working temperature

3 结 论

应用丝网印刷和共烧结制备 LNF-GDC 组成梯度复合阴极 /GDC/ScSZ/GDC/LNF-GDC 组成梯度复合阴极对称电池. 随梯度阴极层数的增加，其阴极的电荷传递电阻和浓差极化电阻皆减小，因而极化电阻也减小.

60%LNF-40%GDC/70%LNF-30%GDC/100% LNF 梯度复合三层阴极性能最佳. 在 850 ℃、800 ℃、750 ℃、700 ℃ 和 650 ℃ 工作温度其极化电阻分别为 0.091、0.202、0.452、1.009 和 2.489 Ω·cm²，小于 70%LNF-30%GDC 复合阴极的极化电阻，其在相应工作温度下分别为 0.101、0.242、0.581、1.363 和 3.405 Ω·cm². 梯度复合阴极层数增加改善了阴极/电解质界面的微观结构，加快了三相界面或气体/阴极/电解质三相接触点反应区的扩散，降低了阴极的极化电阻.

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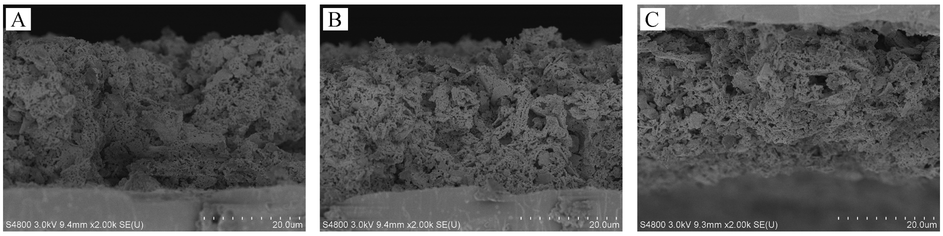

图 5 含有三种阴极 Cathode A、B 和 C 对称电池的纵截面 SEM 形貌照片

Fig. 5 SEM micrographs showing the interfaces of the symmetric cell structure for Cathode A, Cathode B, and

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Fabrication and Impedance Performance of Gradient $LaNi_{0.6}Fe_{0.4}O_{3-\delta}$-$Gd_{0.2}Ce_{0.8}O_2$ Composite Cathodes for Intermediate Temperature Solid Oxide Fuel Cell

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Abstract: A LNF-GDC composite cathode with a gradual change in the composition between ScSZ electrolyte and LNF cathode was fabricated to reduce the cathode polarization resistance (R_p). The gradual change in composition between ScSZ electrolyte and LNF cathode shows the decreases in the charge transfer resistance (R_{ct}) and gas phase diffusion resistance (R_d). The results revealed that the R_p value, measuring 0.452 Ω·cm^2 at 750 °C, was the lowest for LNF-GDC composite cathodes with three layers and gradient changes in composition between ScSZ and LNF (Cathode C), whereas the R_p value of 70%LNF-30%GDC composite cathodes with one layer (Cathode A) was 0.581 Ω·cm^2. The reduction in R_p for the LNF-GDC composite cathodes with three layers and gradient changes in composition between ScSZ and LNF may be related to the fact that the microstructure of the cathode/electrolyte interfaces is significantly improved, resulting in the increase in the area of triple phase boundaries (TPBs), which enhanced the surface exchange of oxygen. This implied that the gradient LNF-GDC composite cathodes showed excellent performance in terms of its electrochemical properties.