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Fabrication and Characterization of the Ni-ScSZ Composite Anodes with a Cu-LSCM-CeO₂ Catalyst Layer in the Thin Film SOFC

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固体氧化物燃料电池 Cu-LSCM-CeO₂/LSCM-YSZ/Ni-ScSZ 复合阳极制备及性能

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摘要: 采用流延法制得 LSCM-YSZ 阳极支撑层/Ni-ScSZ 阳极活性层/ScSZ 电解质层复合膜, 在 LSCM-YSZ 支撑层上印刷一层 Cu-LSCM-CeO₂ 阳极催化层, 即 Cu-LSCM-CeO₂/LSCM-YSZ/Ni-ScSZ 功能梯度层阳极. 研究表明, Cu/LSCM/CeO₂ 质量配比为 2:7:1 功能梯度阳极 (LSCM-YSZ2010) 有较好的性能, 单电池以氢气和乙醇为燃料 (750 °C) 最大功率密度分别为 511 和 390 mW·cm⁻². 单电池稳定性实验表明, LSCM-YSZ2010 阳极单电池以乙醇为燃料 750 °C 长时间运行 218 h, 性能稳定. X-射线能量散射分析表明该阳极具有较好的抗碳沉积性能.

关键词: 固体氧化物燃料电池; 催化层; 碳沉积; 阻抗谱; 电化学性能

中图分类号: TM911

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固体氧化物燃料电池 (SOFC) 可以将储存在燃料中的化学能直接转化为电能, 具有能量转化效率高、环境友好、燃料适应性强的优点^[1-3]. 传统的 Ni/YSZ 金属陶瓷阳极当直接使用碳氢化合物燃料时, 发生碳沉积, 降低电池性能^[4-7], 因此适宜的碳氢化合物燃料阳极材料一直备受关注. Cu-CeO₂-YSZ 系^[8-12]、氧化铈系^[13-17]、钙钛矿结构氧化物^[18-22]等阳极材料已被深入地研究. 氧化铈的中温电子导电率和电催化活性均不佳, 需掺入 Ni 金属. Cu-CeO₂-YSZ 系中 Cu 对碳氢化合物燃料的裂解反应活性不高, 虽可抑制碳沉积, 但 Cu 及其化合物的熔点低, 难与 CeO₂ 烧结, 故多采用反复浸渍, 该法耗时耗能, 且质量控制困难. 钙钛矿结构的陶瓷基阳极 (如掺杂的 LaCrO₃ 和 SrTiO₃) 抗碳沉积性较优良, 而其活性差. 因而研究目标更多转移至新型阳极结构. Barnett 等^[23-24]将 Ru-CeO₂ 催化层置于传统的 Ni-YSZ 阳极, 取得了较好的效果. 而该催化层 Ru 价格昂贵及在一定温度下还会生成 RuO₄^[25]. Wang 等^[26]采用 Ru-Al₂O₃ 催化层代替 Ru-CeO₂ 催化层, 该催化层具有极佳的稳定性以及与 Ni-YSZ 良好的兼容性, 且碳沉积量极少, 但 Ru 价格昂贵难以推广. Hornés 等^[27]采用 Cu-Ni-CeO₂ 阳极材料, 对甲烷有强电催化分解作用, 但仍有少量的碳沉

积. Yun 等^[28]将 SDC 包覆于 Ni/SDC 阳极且取得了极佳的效果, 该阳极运行 180 h 后性能无明显衰减, 但碳沉积问题仍未改善. Zhu 等^[29]将 Ni-La_xCe_{1-x}O₃ 催化层置于 Ni-SDC 阳极, 其电催化活性很好, 并在一定程度抑制了碳沉积. Jin 等^[30]将 Cu_{1.3}Mn_{1.7}O₄-SDC 催化层置于 Ni-SDC 阳极, 其电催化性能极佳, 然而催化层以及阳极仍有少量碳沉积.

钙钛矿结构 (ABO₃) 的陶瓷基阳极材料 (如掺杂的 LaCrO₃) 在碳氢化合物燃料中的电导率低, 但仍有一定的电催化活性, 能抗碳沉积, 且其氧化还原循环过程的体积变化很小. 本文采用复合流延共烧结和丝网印刷法制得 Cu-La_{0.75}Sr_{0.25}Cr_{0.5}Mn_{0.5}O₃ (LSCM)-CeO₂/LSCM-YSZ/Ni-ScSZ/ScSZ 复合膜, 采用功能梯度阳极相配方式, 以期降低碳氢化合物燃料发电时的碳沉积. 功能梯度阳极的 Cu-LSCM-CeO₂ 催化层, 实现碳氢化合物的阳极表面电催化氧化, 避免碳沉积; LSCM-YSZ 阳极支撑层, 支撑整个阳极复合膜; Ni-ScSZ 阳极活性层, 提高电催化活性. 以氢气和乙醇蒸汽为燃料, 氧气为氧化剂, 构筑以 Cu-LSCM-CeO₂/LSCM-YSZ/Ni-ScSZ/ScSZ 为阳极的 SOFC 单电池.

1 单电池与测试

阳极: 用乙醇和丁酮的共沸物作溶剂, 加入粉

料(ScSZ、NiO-ScSZ 及 LSCM-YSZ), 粘结剂(PVB: 10%~15%), 分散剂(三乙醇胺: 1%~3%), 增塑剂(聚乙二醇: 6%~9%) 和造孔剂(草酸氨: 10%~15%) 球磨 4 h 制浆, 3 次流延制得 LSCM-YSZ 阳极支撑层/Ni-ScSZ 阳极活性层/ScSZ 电解质复合膜, 于 1400 °C 烧结 3 h, 即得复合膜($\phi=14$ mm)。

阴极: (Pr_{0.7}Ca_{0.3})_{0.9}MnO₃ (PCM) 与松油醇按质量比 1:0.7 混合制浆, 经丝网印刷沉积至电解质 ScSZ 上, 1200 °C 烧结 2 h 即可, 组合构成 Ni-ScSZ/ScSZ/PCM 单体电池。低温燃烧法合成 La_{0.75}Sr_{0.25}Cr_{0.5}Mn_{0.5}O₃^[31] 和 Cu-CeO₂ 粉体^[32], 将粉体与松油醇混合成浆料, 经丝网印刷沉积到阳极支撑层 LSCM-YSZ 上, 于 1150 °C 烧结 2 h 得 CuO-LSCM-CeO₂/LSCM-YSZ/Ni-ScSZ 功能梯度层阳极, 燃料电池阳极 H₂ 气氛中还原成 Cu-LSCM-CeO₂/LSCM-YSZ/Ni-ScSZ。Cu/LSCM/CeO₂ 按质量配比制备不同的催化层, 其中, 15%Cu-70%LSCM-15%CeO₂、20%Cu-70%LSCM-10%CeO₂、22.5%Cu-70%LSCM-7.5%CeO₂ 分别标示为 LSCM-YSZ1515、LSCM-YSZ2010 和 LSCM-YSZ3010。

单电池: 将由阳极、催化层、电解质及阴极组成的单电池密封于 Al₂O₃ 管的一端, 管内通氧气, 管外通 H₂ 或者乙醇蒸汽。采用电化学工作站 (PARSTAT4000, America) 测试单电池的 *I-V* 曲线以及交流阻抗谱图。用扫描电子显微镜 (SEM, Sirion 200) 观察阳极形貌。

2 结果与讨论

图 1、2 给出 LSCM-YSZ1515、LSCM-YSZ2010

和 LSCM-YSZ3010 阳极单电池的氢气和乙醇燃料 (750 °C、800 °C) 的 *I-V* 曲线与 *I-P* 曲线。从图可见, LSCM-YSZ2010 阳极单电池在 750 °C 最大功率密度分别为 511 mW·cm⁻² (氢气) 和 390 mW·cm⁻² (乙醇), 而 LSCM-YSZ1515 阳极单电池, 最大功率密度分别是 228 mW·cm⁻² (氢气) 和 165 mW·cm⁻² (乙醇)。在 800 °C 下, LSCM-YSZ1515 为阳极单电池, 最大功率密度为 287 mW·cm⁻² (氢气) 和 210 mW·cm⁻² (乙醇), 而 LSCM-YSZ2010 阳极单电池, 最大功率密度分别达到 656 mW·cm⁻² (氢气) 和 492 mW·cm⁻² (乙醇)。然而, 随着 Cu 含量的增加, 750 °C 时 LSCM-YSZ3010 阳极单电池最大功率却只有 327 mW·cm⁻² (氢气) 和 294 mW·cm⁻² (乙醇)。800 °C 时, 其最大功率密度为 419 mW·cm⁻² (氢气) 和 371 mW·cm⁻² (乙醇)。随阳极催化层中 Cu 含量增加, 单电池性能先增加后反而下降。电化学交流阻抗谱图 (图 3、4) 也证实了随着阳极催化层 Cu 含量增加, 其电池的欧姆电阻和极化电阻也出现先减小后增加的趋势。同一单电池, 乙醇燃料极化电阻比氢气极化电阻稍有增加。Cu 颗粒不仅构成了阳极导电网络, 尤其连通了各个反应活性点位。因此, CeO₂ 含量达到一定值, 其量再增加对提高阳极性能并无太大裨益, 此时掺入更多 Cu, 不仅能提高阳极的电导率, 还能连接更多的反应活性点位, 增加阳极三相界面长度^[33-34]。乙醇的分子量比氢气的大得多, 二者的扩散速率也相差甚多, 另一种可能原因就是燃料自身的差异, 作为还原剂, 乙醇的活性显然比氢气的差得多, 这就导致更高的极化电阻

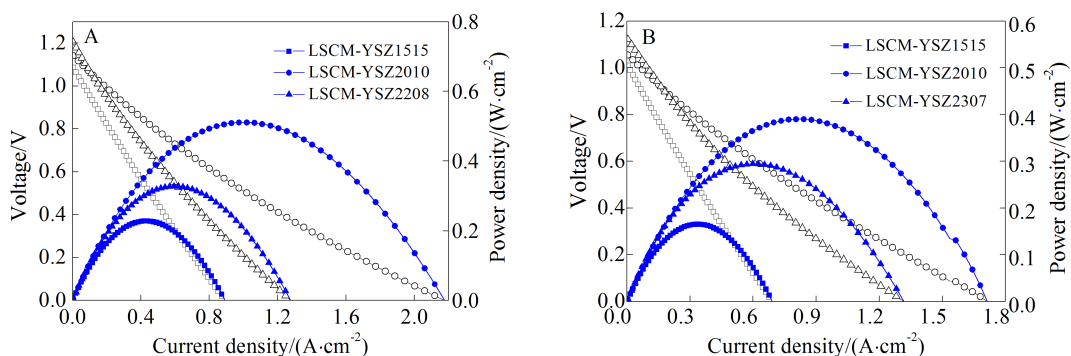


图 1 LSCM-YSZ1515、LSCM-YSZ2010 和 LSCM-YSZ3010 阳极单电池氢气(A)和乙醇蒸汽(B)燃料(750 °C)的 *I-V* 和 *I-P* 曲线

Fig. 1 Voltage and power density vs. current density curves for an SOFC with LSCM-YSZ1515, LSCM-YSZ2010 and LSCM-YSZ3010 anodes while running on hydrogen (A) and ethanol steam (B) at 750 °C

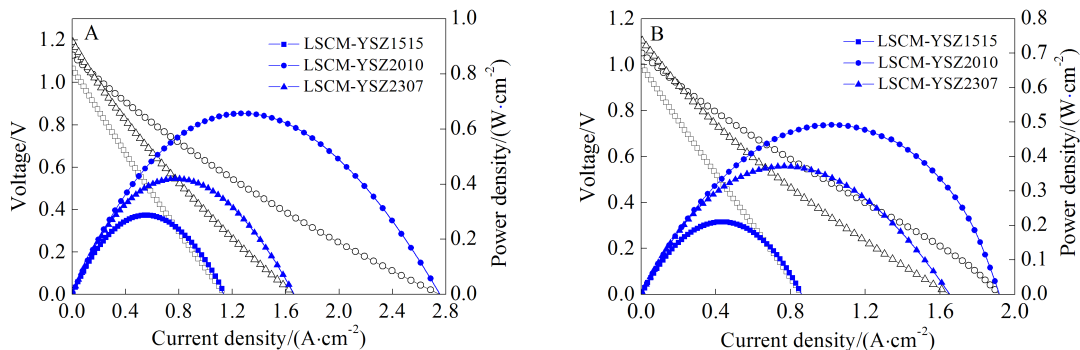


图 2 LSCM-YSZ1515、LSCM-YSZ2010 和 LSCM-YSZ3010 阳极单电池氢气(A)和乙醇蒸汽(B)燃料(800 °C)的 *I-V* 和 *I-P* 曲线

Fig. 2 Voltage and power density vs. current density curves for an SOFC with LSCM-YSZ1515, LSCM-YSZ2010 and LSCM-YSZ3010 anodes while running on hydrogen (A) and ethanol steam (B) at 800 °C

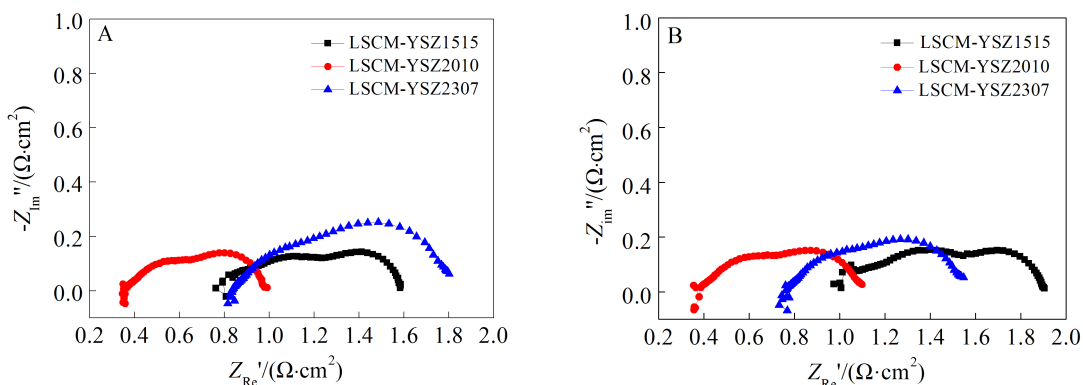


图 3 LSCM-YSZ1515、LSCM-YSZ2010 和 LSCM-YSZ3010 阳极单电池氢气(A)和乙醇蒸汽(B)燃料(750 °C)的阻抗谱图

Fig. 3 Electrochemical impedance spectra for an SOFC with LSCM-YSZ1515, LSCM-YSZ2010 and LSCM-YSZ3010 anodes while running on hydrogen (A) and ethanol steam (B) at 750 °C

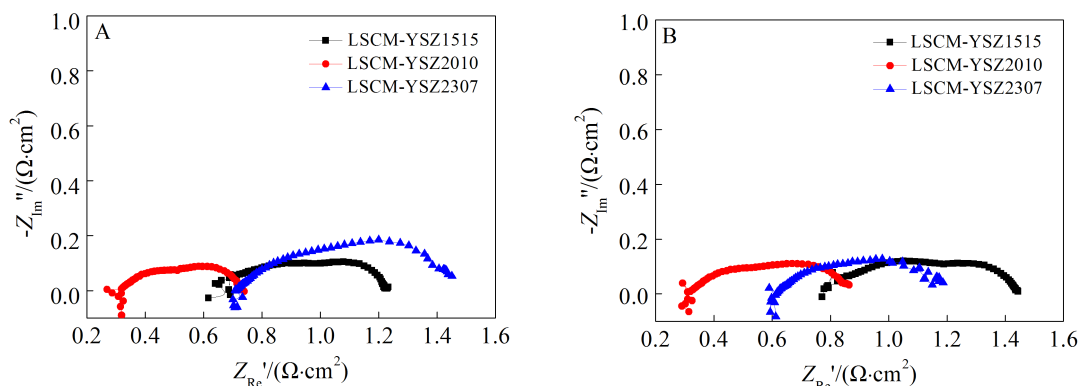


图 4 LSCM-YSZ1515、LSCM-YSZ2010 和 LSCM-YSZ3010 阳极单电池氢气(A)和乙醇蒸汽(B)燃料(800 °C)阻抗谱图

Fig. 4 Electrochemical impedance spectra for an SOFC with LSCM-YSZ1515, LSCM-YSZ2010 and LSCM-YSZ3010 anodes while running on hydrogen (A) and ethanol steam (B) at 800 °C

以及较慢的电化学氧化. 这些因素决定了乙醇燃料极化面电阻远大于其在氢气的数值, 其电化学

性能自然相对较差. LSCM-YSZ3010 阳极单电池欧姆电阻随 Cu 含量的增加反而增加, 可能是由于

高温操作时 Cu 颗粒分散不均匀, 以及 Cu 颗粒的烧结和质量转移导致阳极导电通路的减少, 从而降低了阳极的电导率. 同时, 过多的掺入 Cu 也降低了阳极的催化性能, 导致电池性能降低.

图 5 为三组单电池乙醇为燃料(750 °C, 0.6 V) $w-t$ 曲线. 从该图可发现, 三组电池在该条件下随时间变化功率基本不变, 且以 LSCM-YSZ2010 为阳极的单电池功率密度随时间变化有微小的波动, 可能是由于操作控制气流时导致乙醇蒸汽供应的不稳定而引起的, 运行 218 h, 其功率密度几乎维持不变(200 mW·cm⁻²), 说明该阳极单电池可较长时间稳定工作.

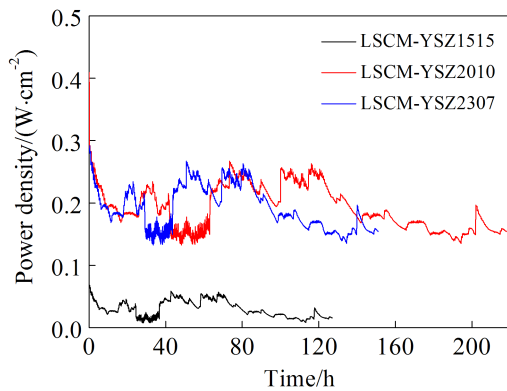


图 5 以 LSCM-YSZ1515、LSCM-YSZ2010 和 LSCM-YSZ3010 阳极单电池乙醇燃料(750 °C, 0.6 V) $w-t$ 曲线

Fig. 5 Power density vs. time curves of an SOFC with the LSCM-YSZ1515、LSCM-YSZ2010 and LSCM-YSZ3010 anodes using ethanol steam as fuel and oxygen as oxidant while discharging under 0.6 V at 750 °C during the aging process

3 扫描电镜分析

图 6 示出 Cu-LSCM-CeO₂/LSCM-YSZ/Ni-ScSZ/ScSZ/PCM 断面的 SEM 照片. 从照片可以看出, Cu-LSCM-CeO₂ 催化层、LSCM-YSZ 支撑层、Ni-ScSZ 活性层、ScSZ 电解质层和 PCM 阴极层. 其电化学反应发生于三相界面, 因而阳极的微观对电池的性能有很大的影响. 长时间运行的阳极支撑层与阳极催化层及其界面的断面 SEM 显微结构照片图 7 所示: LSCM-YSZ2010 阳极单电池两层间结合良好, 没有很明显的分层, 颗粒大小均匀, 仍有良好的多孔结构. 扫描电子显微镜

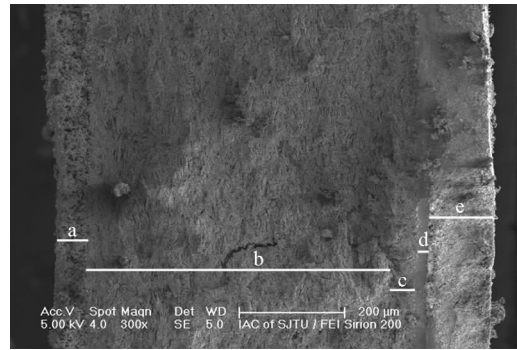


图 6 单电池 Cu-LSCM-CeO₂/LSCM-YSZ/Ni-ScSZ/ScSZ/PCM 断面的 SEM 照片

a. Cu-LSCM-CeO₂ 催化层; b. LSCM-YSZ 支撑层; c. Ni-ScSZ 活性层; d. ScSZ 电解质层; e. PCM 阴极层

Fig. 6 SEM micrograph of a fractured single cell showing five layers

a. porous Cu-LSCM-CeO₂ catalyst layer; b. porous LSCM-YSZ support layer; c. porous Ni-ScSZ active layer; d. dense ScSZ electrolyte layer; e. porous PCM cathode layer

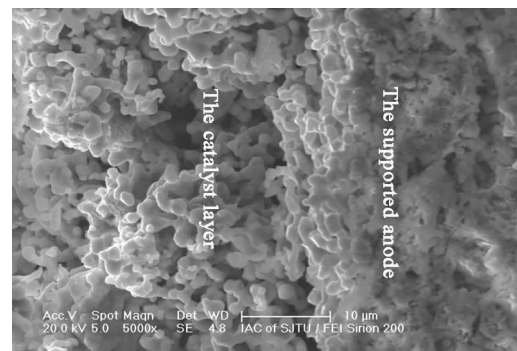


图 7 LSCM-YSZ2010 为阳极的单电池阳极 SEM 照片

Fig. 7 SEM cross-sectional micrograph of the LSCM-YSZ 2010 anode

和 X-射线能量散射分析谱 (EDAX) 显示 LSCM-YSZ2010 阳极仅有少量碳沉积(图 8), 有较好的抗碳沉积性能.

4 结论

三层流延法 LSCM-YSZ 支撑层 /Ni-ScSZ 活性层 /ScSZ 电解质层复合膜, 在烧结的 LSCM-YSZ 支撑层上丝网印刷一层 Cu-LSCM-CeO₂ 催化层, 并制得 Cu/LSCM/CeO₂ 不同配比功能梯度阳极的单电池. LSCM-YSZ2010 阳极单电池性氢气和乙醇蒸汽为燃料时(750 °C)

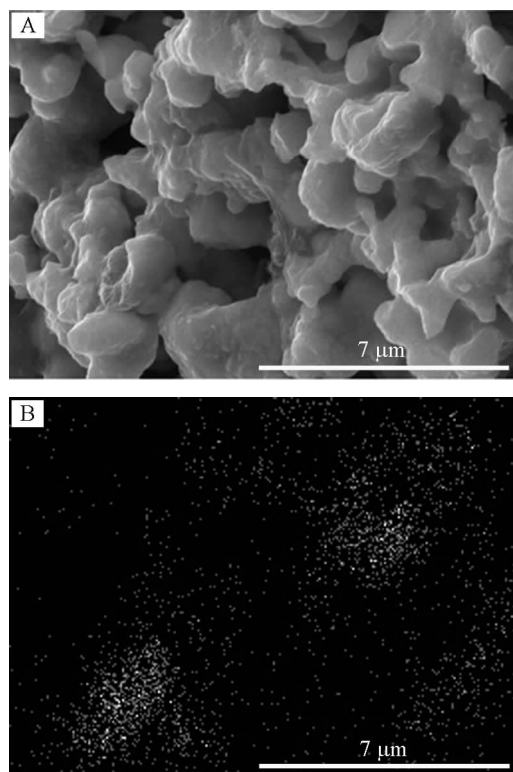


图 8 LSCM-YSZ2010 为阳极单电池以乙醇为燃料(750 °C)运行 218 h 的电子探针显微照片:A. 阳极自由面的 SEM 照片;B. 阳极自由面的碳沉积照片

Fig. 8 Electron probe microscopic analysis (EPMA) of the LSCM-YSZ2010 anode operated in ethanol fuel at 750 °C under an open circuit condition for 218 h: A. SEM image of the anode free surface; B. C mapping of the anode free surface

最大功率密度为 511 和 390 $\text{mW}\cdot\text{cm}^{-2}$ 。218 h 长时间运行后,其性能仅略有下降,有较好的抗碳沉积性。

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Fabrication and Characterization of the Ni-ScSZ Composite Anodes with a Cu-LSCM-CeO₂ Catalyst Layer in the Thin Film SOFC

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Abstract: Solid oxide fuel cell (SOFC) directly operating on hydrocarbon without external reforming has the potential of greatly speeding up the application of SOFCs for transportation. In this paper, a three-layer structure anode was fabricated by tape casting and screen printing method. The addition of Cu-LSCM-CeO₂ to the supported anode surface presented better performance running on H₂ and ethanol. The maximum power densities were 511 and 390 mW·cm⁻², respectively running on H₂ and ethanol at 750 °C. No significant degradation was observed on the anode. Consequently, the Cu-LSCM-CeO₂ catalyst layer on the surface of the LSCM-YSZ support layer makes it possible to have good stability for long-term operation in ethanol fuel due to free carbon deposition.

Key words: solid oxide fuel cell; catalyst layer; carbon deposit; impedance spectra; electrochemical performance