Supplementary Material (ESI) for Journal of Materials Chemistry

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Supporting Information for:

In Situ Neutron Diffraction Study of the High-Temperature Redox Chemistry of \( Ln_{3-x}Sr_{1+x}CrNiO_{8-\delta} \) (\( Ln = \text{La, Nd} \)) under Hydrogen

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Figure S1. Thermogravimetric analysis (heating rate of 2 °C min⁻¹) under 5 % H₂ in N₂ for Nd₂.25Sr₁.75CrNiO₈₋δ

Figure S2. XRD pattern fit at room temperature of La₂Sr₂CrNiO₆₋δ; (a) as-prepared, (b) after hydrogen reduction (in 5% H₂ at 700 °C, 8h)

Figure S3. XRD pattern fit at room temperature of Nd₂Sr₂CrNiO₆₋δ; (a) as-prepared, (b) after hydrogen reduction (in 5% H₂ at 700 °C, 8h)

Figure S4. XRD pattern fit at room temperature of Nd₂.25Sr₁.75CrNiO₈₋δ; (a) as-prepared, (b) after hydrogen reduction (in 5% H₂ at 700 °C 8h)

Figure S5. Neutron Powder Diffraction pattern fit of Nd₂Sr₂CrNiO₆₋δ; (a) at 20 °C before hydrogen reduction, (b) at 710 °C after reduction, (c) at 90 °C after reduction and cooling

Figure S6. Neutron Powder Diffraction pattern fit of Nd₂.25Sr₁.75CrNiO₈₋δ; (a) at 20 °C before hydrogen reduction, (b) at 450 °C after reduction, (c) at 80 °C after reduction

Figure S7. Occupancy factor of oxygen sites in (a) Nd₂Sr₂CrNiO₆₋δ and (b) Nd₂.25Sr₁.75CrNiO₈₋δ as a function of temperature under hydrogen flow. Green and black squares represent the occupancy of O1 (axial) and O2 (equatorial) sites during heating; blue and red triangles represent O1 and O2 on cooling. The blue line corresponds to the Ni (II) composition

Figure S8. Temperature dependence of the unit cell parameters of (a) Nd₂Sr₂CrNiO₆₋δ and (b) Nd₂.25Sr₁.75CrNiO₈₋δ under hydrogen flow. Squares (triangles) represent data collected on heating (cooling).

Figure S9. (a) Occupancy factor of oxygen sites in Nd₂Sr₂CrNiO₆₋δ as a function of temperature under oxidizing conditions after reduction. Green and black squares represent the occupancy of O1 (axial) and O2 (equatorial) sites during heating; blue and red triangles represent O1 and O2 during cooling;
(b) temperature dependence of the unit cell parameters of Nd$_2$Sr$_2$CrNiO$_{8-\delta}$ under oxidizing conditions. Squares (triangles) represent data collected on heating (cooling)

Figure S10. Rietveld refinements of the X-ray diffraction pattern of Nd$_2$Sr$_2$CrNiO$_{7.38}$ between $2\theta = 76.6^\circ$ and $81.1^\circ$ (a) with a single profile for all peaks, (b) with anisotropic peak-broadening taken into account.

Figure S11. Temperature dependence of the (a) Cr/Ni-O1, (b) Nd/Sr-O1 (along the c-axis) and (c) Nd/Sr-O2 bond lengths under hydrogen flow, in Nd$_2$Sr$_2$CrNiO$_{8-\delta}$. Squares (triangles) represent data collected on heating (cooling)

Figure S12. Temperature dependence of the (a) Cr/Ni-O1, (b) Nd/Sr-O1 (along the c-axis) and (c) Nd/Sr-O2 bond lengths under hydrogen flow, in Nd$_{2.25}$Sr$_{1.75}$CrNiO$_{8-\delta}$. Squares (triangles) represent data collected on heating (cooling)
Figure S1

Figure S2
Figure S3

Figure S4
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Figure S5

(a)                                           (b)                                           (c)

Figure S6

(a)                                           (b)                                           (c)
Figure S7

(a) 

(b) 

Figure S8

(a) 

(b)
Figure S9

Figure S10
Figure S11
Figure S12