Experimental report

06/03/2024

Proposal: **EASY-1130** **Council:** 10/2022

Title: New Ru-SrMo0.9O3-d perovskite with exsolved Ru nanoparticles as high-performance composite anodes for solid-oxide fuel cells

Research area: Materials

This proposal is a new proposal

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Experimental team:

Local contacts: Maria Teresa FERNANDEZ DIAZ

Samples: Ru-SrMo0.9O3-d

Instrument	Requested days	Allocated days	From	To
D2B	8	8	16/04/2023	17/04/2023

Abstract:

A new perovskite with metallic Ru in exsolution Ru-SrMo0.9O3-δ, has been prepared and tested as anode material in intermediate-temperature solid-oxide fuel cells (IT-SOFCs) generating a power of 600 mW/cm2 at 850 °C using pure H2 as fuel. It was obtained by reduction of a Ru-containing scheelite oxide, from which Ru is segregated to the surface as metal nanoparticles. The electrical conductivity gave a maximum value of 390 S cm-1 at 850 °C. A suitable thermal expansion coefficient, with values close to that of the electrolyte, showed no abrupt changes in its slope. All these features make this novel perovskite a perfect candidate as anode material in IT-SOFCs. In this experiment we aim at correlating the good performance of this material as anode with the structural features determined from NPD, as neutrons are very sensitive to the presence of oxygen vacancies, which are required for the MIEC properties of these materials. For this purpose we plan to study the crystallographic structures at RT for Ru-SrMo0.9O3-δ. We aim at gaining information about the actual crystal symmetry, the order-disorder of the oxygen vacancies and the thermal vibrations and oxygen contents.

A new perovskite with metallic Ru in exsolution Ru-$SrMo_{0.9}O_{3-\delta}$, has been prepared and tested as anode material in intermediate-temperature solid oxide fuel cells (IT-SOFCs) generating a power density of 600 mW/cm^2 at 850 °C using pure H_2 as fuel. It was obtained by reduction of a Ru-containing scheelite oxide, from which Ru is segregated to the surface as metal nanoparticles. The electrical conductivity gave a maximum value of 390 S cm^{-1} at 850 °C (Fig. 1). A suitable thermal expansion coefficient of the oxidized and reduced samples, with values close to that of the electrolyte, showed no abrupt changes in its slope (Fig. 2). All these features make this novel perovskite a perfect candidate as anode material in IT-SOFCs.

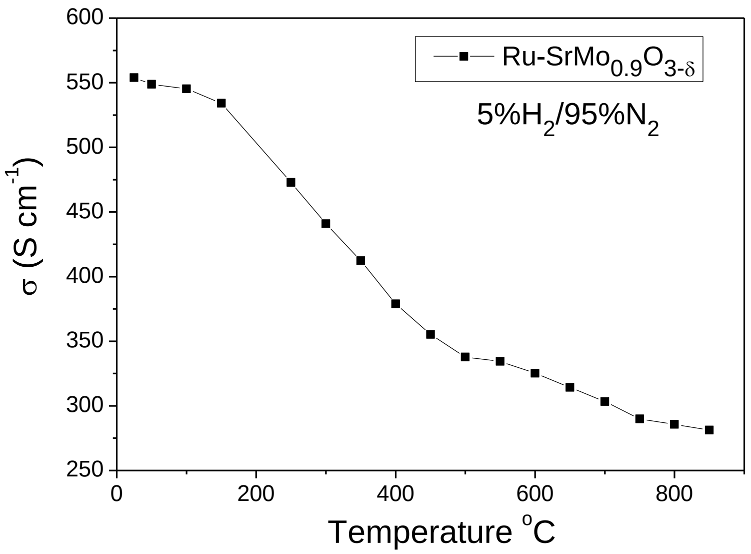

***Fig. 1**. DC-conductivity as a function of temperature for the Ru-$SrMo_{0.9}O_{3-\delta}$phase in reducing H_2/N_2 flow.*

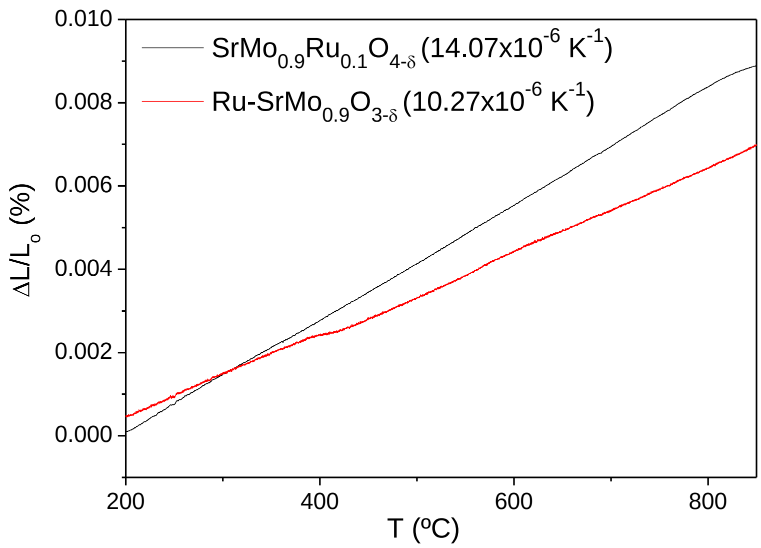

***Fig. 2.** Thermal expansion determined by dilatometry of the $SrMo_{0.9}Ru_{0.1}O_{4-\delta}$ and Ru-$SrMo_{0.9}O_{3-\delta}$phases in air or in reducing H_2/N_2 flow, respectively.*

In this experiment we have study the structure of Ru-$SrMo_{0.9}O_{3-\delta}$ sample to correlate the good performance of this material as anode with the structural features determined from NPD at 25 ºC, as neutrons are very sensitive to the presence of oxygen vacancies, which are required for the MIEC properties of these materials. Neutron powder diffraction (NPD) data were collected in the diffractometer D2B. The high intensity mode ($\Delta d/d \approx 5\cdot10^{-4}$) was selected, with a neutron wavelength λ= 1.594 Å within the angular 2θ range from 10° to 160°. About 2 g of the sample was contained in a vanadium can placed in the isothermal zone of a furnace with a vanadium resistor operating under vacuum ($P_{O2} \approx 10^{-6}$ Torr). The measurement was carried out at 25 ºC. The collection time was of 3 h.

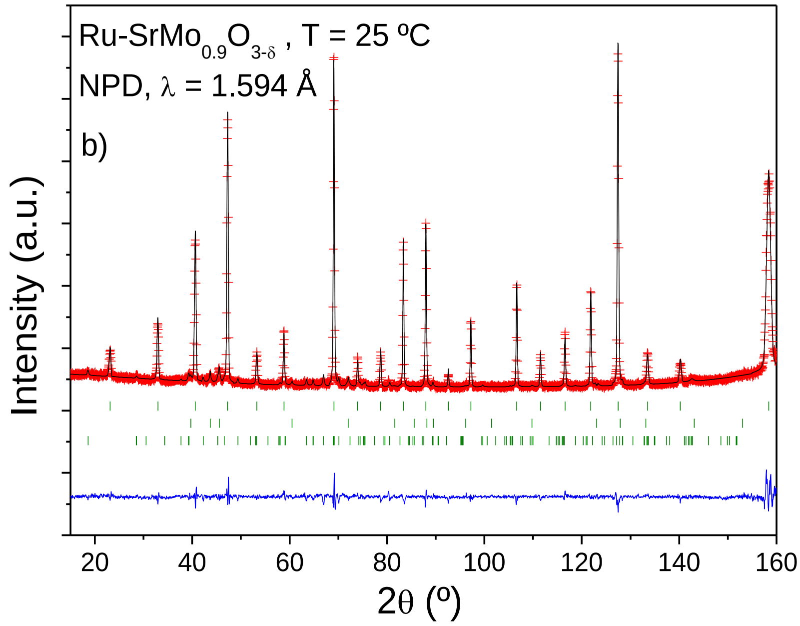

Fig. 3. *Observed (red crosses), calculated (black full line) and difference (blue line) NPD patterns for the Ru-$SrMo_{0.9}O_{3-\delta}$ sample at 25 ºC, refined in the Pm-3m space group. A second and third set of Bragg reflections correspond to the segregated metallic Ru (s.g. $P6_3/mmc$) and a minor amount of oxidized scheelite phase (s.g. $I4_1/a$) respectively.*

$SrMo_{0.9}O_{3-\delta}$ perovskite matrix can be Rietveld-refined in the cubic *Pm*-3*m* space group at room temperature (Fig.3.). Fig. 4 shows the structure of the cubic perovskite. The crystallographic formulae after refining and considering that all the Ru atoms are segregated to the surface of the perovskite, is denoted as Ru-$SrMo_{0.8720(7)}O_{2.736(2)}$. This huge amount of oxygen vacancies are an essential requirement in anodes to perform well in SOFCs.

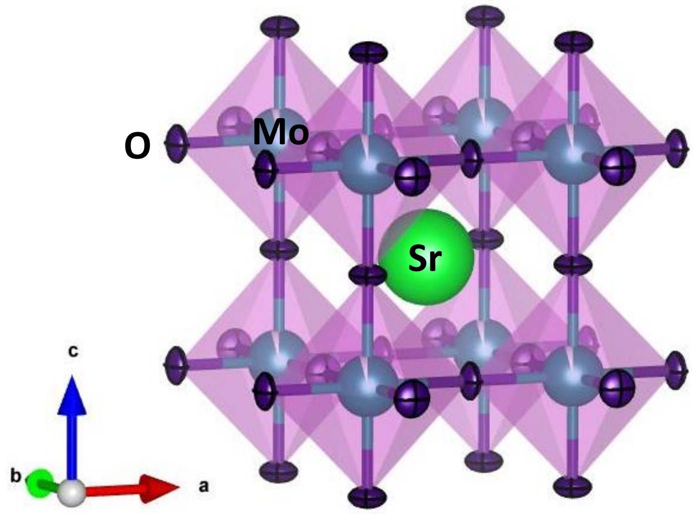

Fig. 4. *Crystal structure of $SrMo_{0.9}O_{3-\delta}$ perovskite matrix at 25 ℃.*