Electronic Supplementary Material (ESI) for Journal of Materials Chemistry A.

UNCERTAINTY OF OXYGEN CONTENT IN HIGHLY NONSTOICHIOMETRIC OXIDES FROM NEUTRON DIFFRACTION DATA: EXAMPLE OF PEROVSKITE-TYPE $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$

Aleksey A. Yaremchenko,*[a] Dmitry D. Khalyavin,[b] Mikhail V. Patrakeev [c]

[a] *CICECO – Aveiro Institute of Materials, Department of Materials and Ceramic Engineering, University of Aveiro, 3810-193 Aveiro, Portugal*

[b] *ISIS Facility, Rutherford Appleton Laboratory, Harwell Oxford, Didcot, OX11 0QX, United Kingdom*

[c] *Institute of Solid State Chemistry, UB RAS, 91 Pervomayskaya Str., 620990 Yekaterinburg, Russia*

** Corresponding author. Fax: +351-234-370204; Tel: +351-234-370235; e-mail: ayaremchenko@ua.pt*

Electronic supplementary information

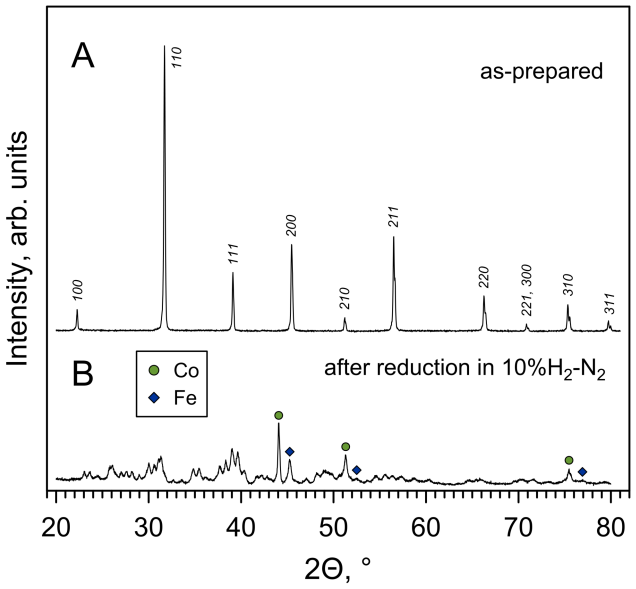

Fig.S1. XRD patterns of (A) as-prepared BSCF powder, and (B) product of reduction of BSCF powder in 10%H_2-N_2 flow at 1000-1100°C for 30 h. Unmarked peaks in the bottom pattern are assigned to a mixture of (Ba,Sr)O and (Ba,Sr)$(OH)_2(H_2O)_x$.

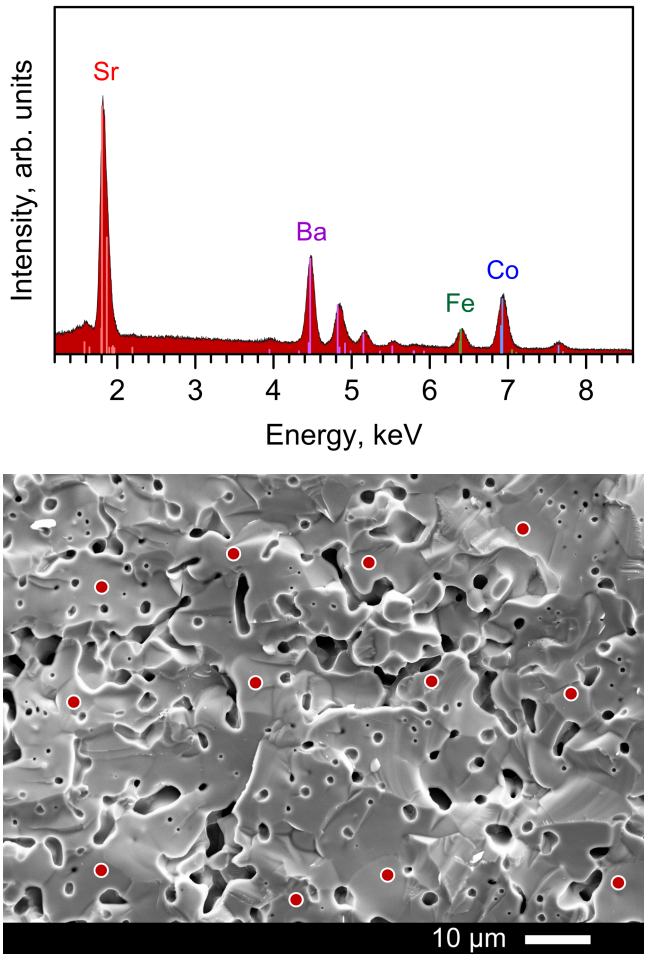

Fig. S2. Typical EDS spectrum (top, real time 200 s) obtained from the fractured surface of BSCF ceramics (bottom). Red points in the SEM micrograph indicate the places of spectra collection (electron interaction depth 1.3 μm, radius 0.7 μm).

Table S1. Atomic ratios of cations in A and B sublattices obtained by analysis of EDS spectra.[a]

Point	Ba : Sr [b]	Co : Fe [c]
1	0.471 : 0.529	0.806 : 0.194
2	0.482 : 0.518	0.795 : 0.205
3	0.509 : 0.491	0.809 : 0.191
4	0.527 : 0.473	0.808 : 0.192
5	0.475 : 0.525	0.801 : 0.199
6	0.479 : 0.521	0.799 : 0.201
7	0.482 : 0.514	0.809 : 0.191
8	0.514 : 0.486	0.802 : 0.198
9	0.527 : 0.473	0.805 : 0.195
10	0.516 : 0.484	0.799 : 0.201
11	0.465 : 0.535	0.802 : 0.198
12	0.527 : 0.473	0.794 : 0.206
Average	0.498 : 0.502	0.802 : 0.198

[a] Standardless ZAF method; [b] L series, estimated average error in element fractions is ±0.038 atoms; [c] K series, estimated average error in element fractions is ±0.024 atoms

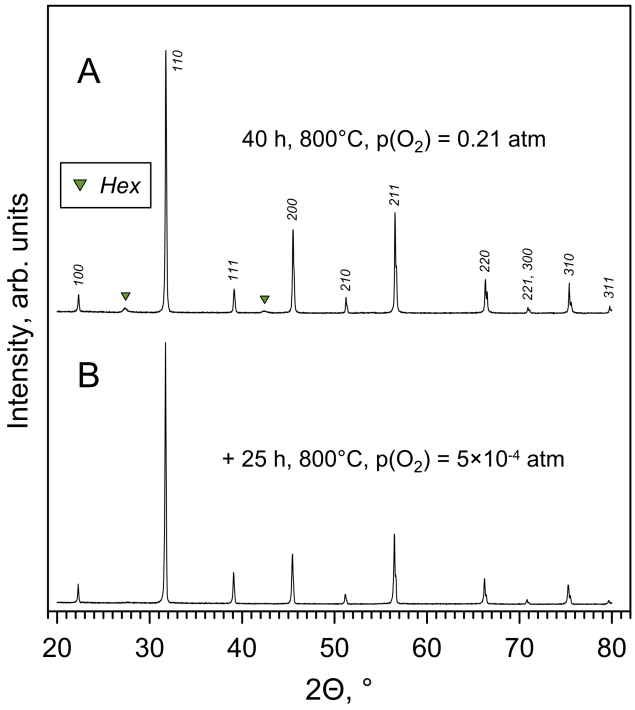

Fig.S3. XRD pattern of powdered BSCF calcined at 800°C for 40 h in air (log $p(O_2)$ = -0.68 (atm)) (**A**) and then for 25 h at intermediate oxygen partial pressure (log $p(O_2)$ = -3.3 (atm)) (**B**). Reflections of the hexagonal phase are indexed according to Ref.(64).