Electronic Supplementary Material (ESI) for Materials Advances.

Supporting Information for

Boosting methane partial oxidation on ceria through exsolution of robust Ru nanoparticles

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Ce3d XPS peak deconvolution

For the XPS deconvolution data for Ce3d core shell of the three tested materials some restrictions were followed. First, it was established a ratio between the intensity of the $3d_{5/2}$ and $3d_{3/2}$ components equal to 1.5, that is $I_{3d5/2}/ I_{3d3/2}=1.5$. Additionally, a spin-orbit position constrain of 18.6 eV was established between $3d_{5/2}$ and $3d_{3/2}$ components. For the background a Shirley function was used.

Table S1: XPS deconvolution data for Ce3d of exRuCeO$_2$

Name	Position	FWHM	Area	% Conc.
Ce 3d v′′′	898.08	2.851	7902.6	20.389
Ce 3d v	882.48	2.499	6701.2	17.289
Ce 3d u_0	899.22	2	765.2	1.974
Ce 3d u′′′	916.68	2.524	5254	13.555
Ce 3d v′	884.81	2.85	2019.5	5.21
Ce 3d u	901.08	2.332	4446.8	11.473
Ce 3d u′′	907.33	4.857	3661.9	9.448
Ce 3d u′	903.41	5	1340.5	3.458
Ce 3d v′′	888.73	3.666	5514.3	14.227
Ce 3d v_0	880.62	3	1153.4	2.976

Table S2: XPS deconvolution data for Ce3d Ru-CeO$_2$

Name	Position	FWHM	Area	% Conc.
Ce 3d v′′′	897.95	2.726	11809	20.842
Ce 3d v	882.47	2.62	10737.2	18.95
Ce 3d u_0	898.68	2	662.3	1.169
Ce 3d u′′′	916.55	2.508	7851	13.856
Ce 3d v′	885.18	2.708	1705.6	3.01
Ce 3d u	900.77	2.5	7125.5	12.576
Ce 3d u′′	907.34	4.5	5842	10.311
Ce 3d u′	903.78	4	1132.2	1.998
Ce 3d v′′	888.74	3.569	8797.1	15.526
Ce 3d v_0	880.08	3	998.4	1.762

Table S3: XPS deconvolution data for Ce3d CeO_2

Name	Position	FWHM	Area	% Conc.
Ce 3d v′′′	898.8	3.066	15974.5	24.225
Ce 3d v	883	3.259	16035.8	24.318
Ce 3d u′′′	917.04	3.018	10621.5	16.108
Ce 3d u	901.75	2.344	6335.7	9.608
Ce 3d u′′	907.7	5	6049.4	9.174
Ce 3d v′′	888.74	4.676	10924	16.566

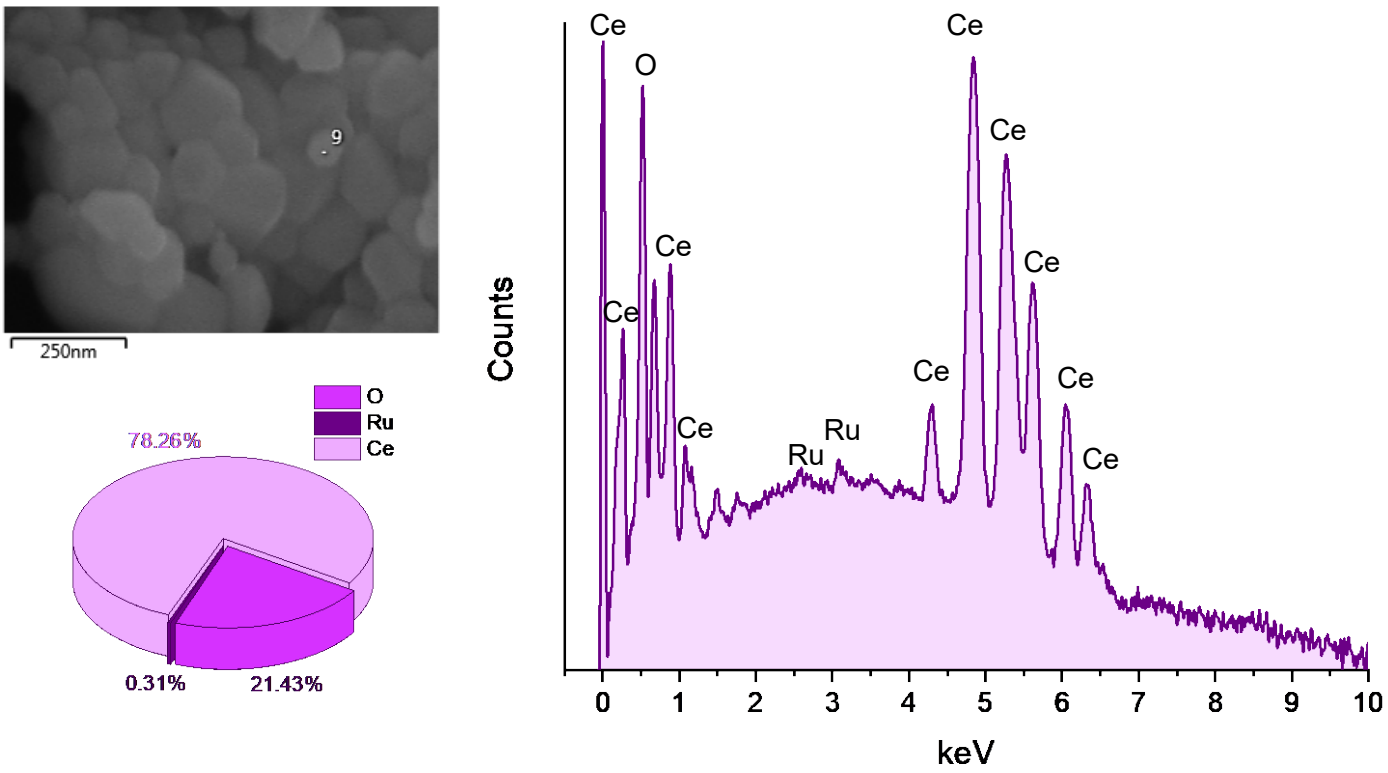

Figure S1. SEM-EDX analysis performed to the Ru-doped CeO_2 sample before exsolution. The pie chart shows the weight percentages for each element.

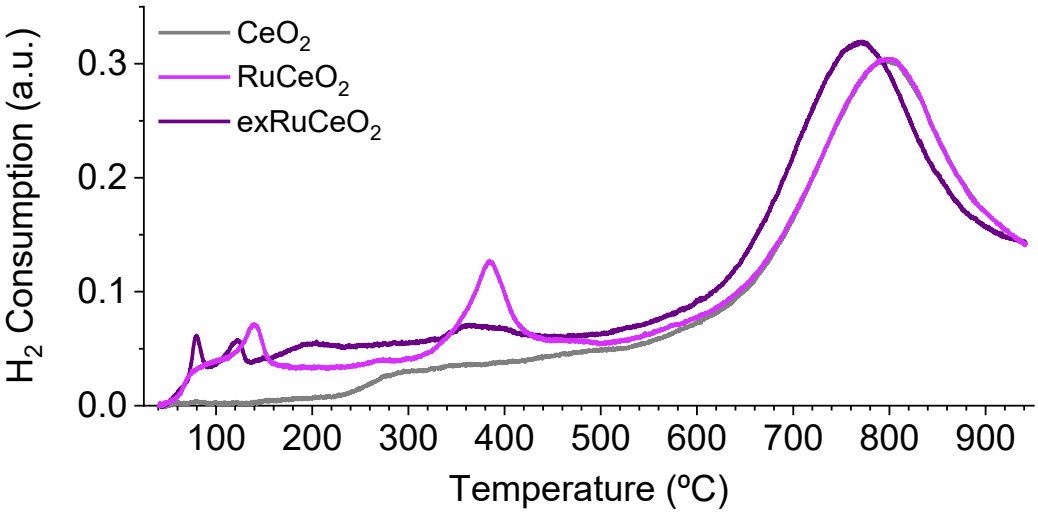

Figure S2. Temperature programmed reduction **(**TPR) analyses performed to CeO_2, Ru-doped CeO_2 before (Ru-CeO2) and after exsolution at 900 ºC and 2h under 5%H_2 ($exRuCeO_2$). The TPR measurements were conducted under a 10% H_2 atmosphere, and heating up to 950 ºC.

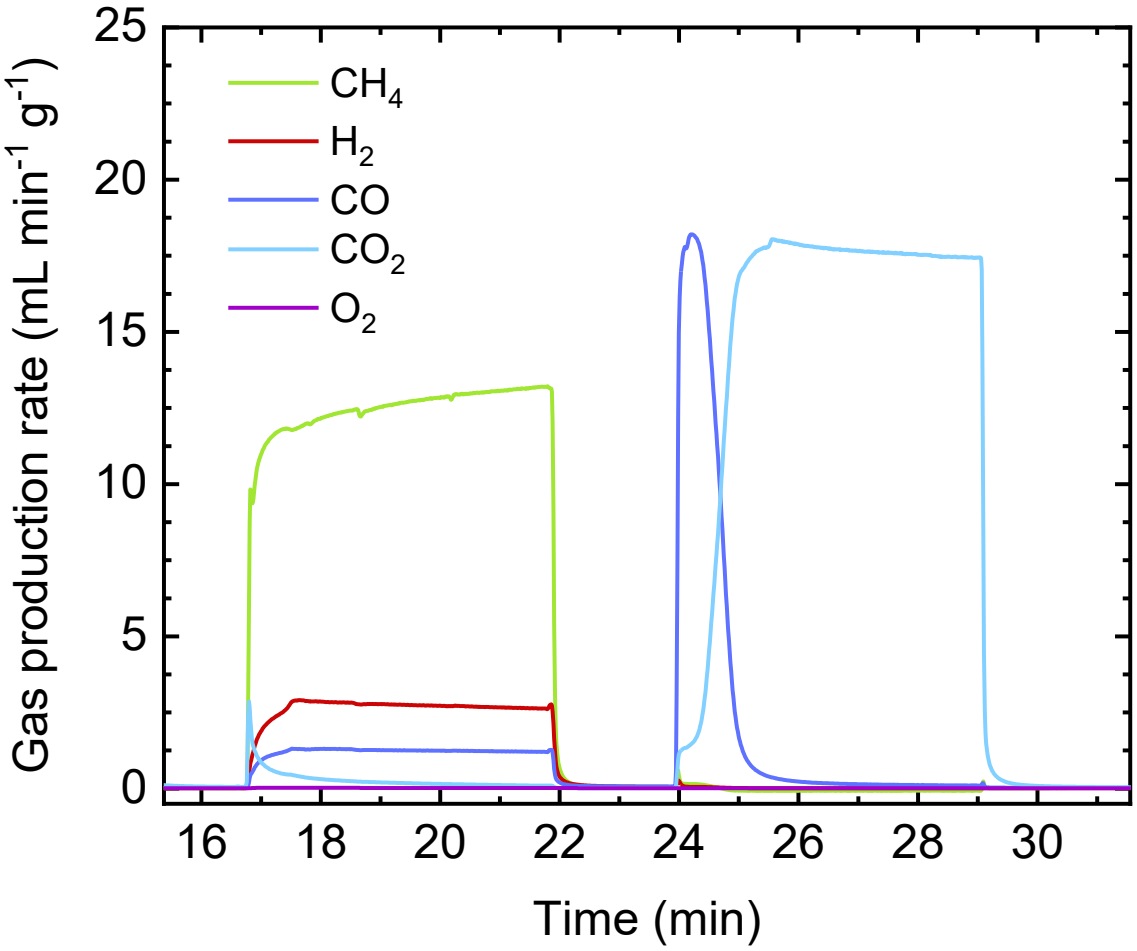

Figure S3. Gas production curves a 2-step chemical looping reforming of methane cycle at 900 ºC. The graph shows the methane consumption and the O_2 line, illustrating no residual oxygen in the reactor.

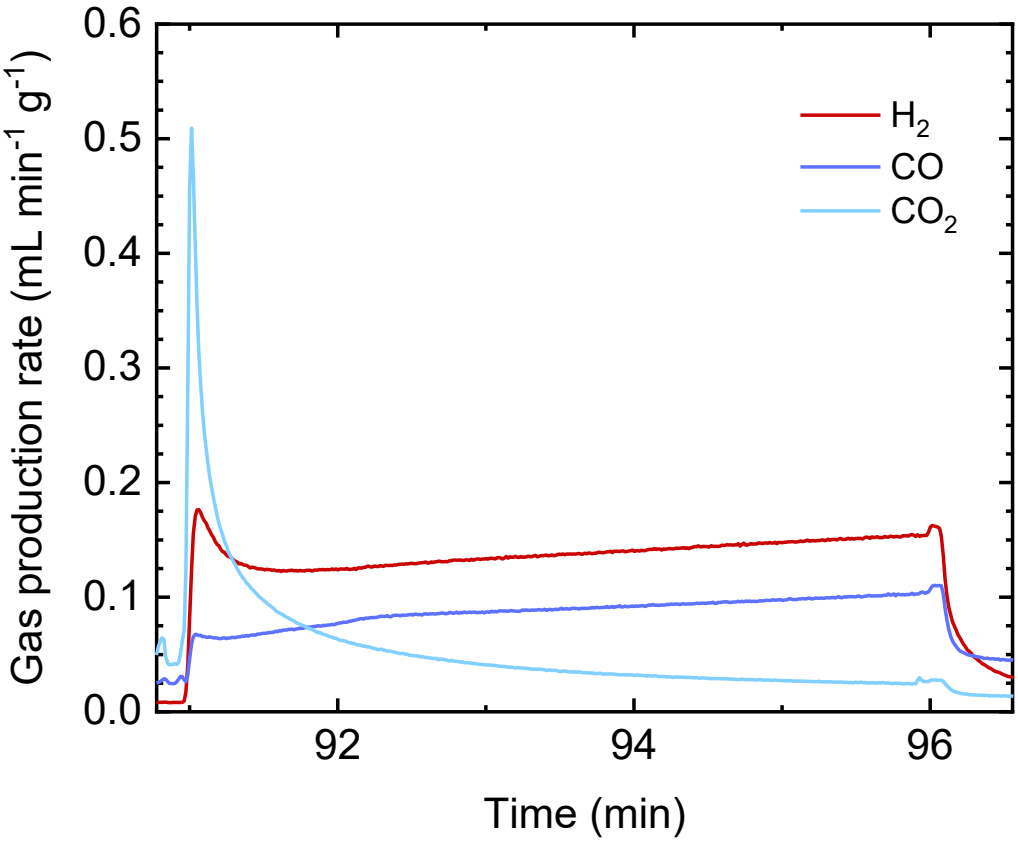

Figure S4. Gas production curves a 2-step chemical looping reforming of methane cycle at 700 ºC, zooming out Figure 6c.

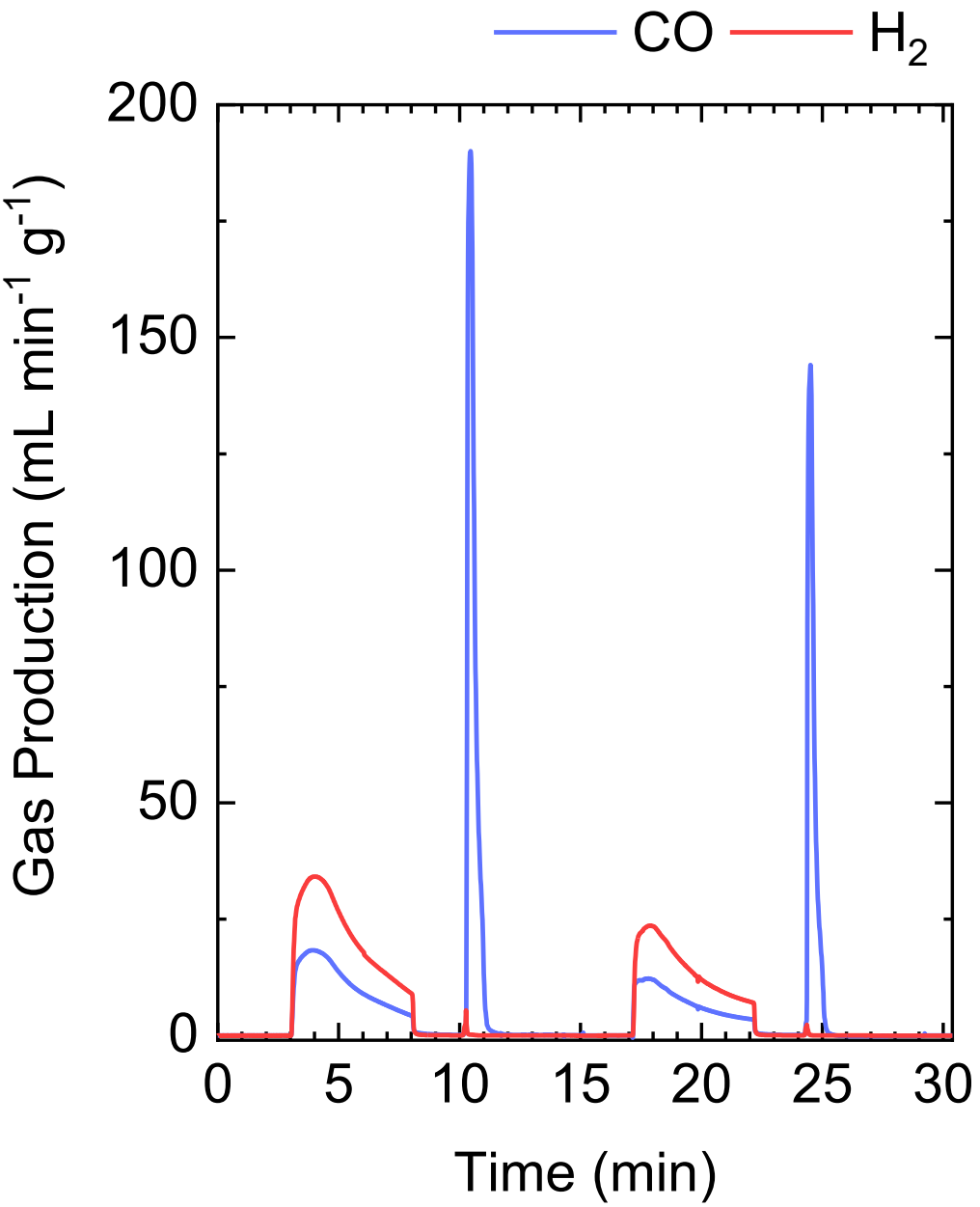

Figure S5. First and second cycle of the 20-cycle longevity test carried out to exRuCeO$_2$