

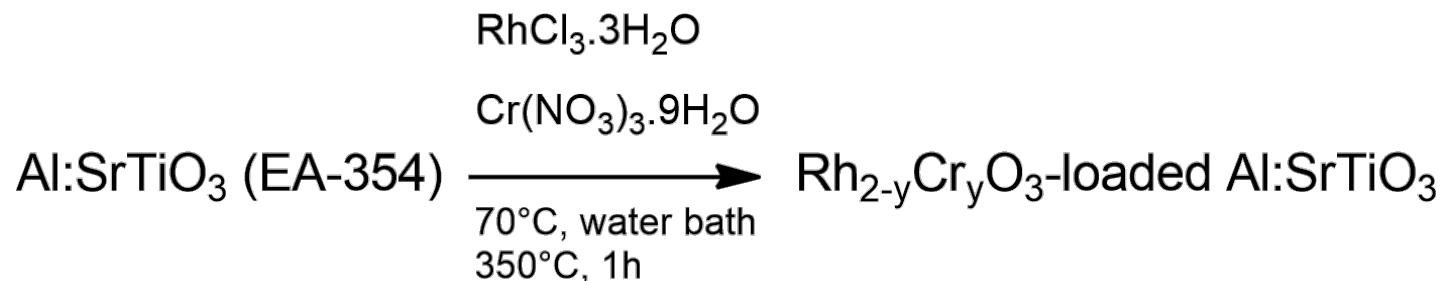
EA-358: Modification of Al:SrTiO₃ (EA-354) via deposition of Rh, Cr oxide co-catalyst, 350°C, 1h, Upscaling (3.33x)

Date: 2025-10-09
Tags: synthesis EA Furnace Muffle Furnace SrTiO₃ RhCl₃ Osterloh RhxCryO₃ Al:SrTiO₃ RhCrO₃:Al:SrTiO₃ Cr(NO₃)₃ Upscaling Category: SrTiO₃ Status: Done
Created by: Ebrahim Abedini

Objective

Modification of Al:SrTiO₃ (EA-354) by loading Rh,Cr oxide co-catalysts via the impregnation method and further annealing at 350°C,1h. 3.33 times upscaling.

Reaction scheme/sample structure



ChemDraw File (linked): [EA-358.cdx](#)

Literature/reference experiments

Literature	https://doi.org/10.1039/C9EE00310
Reproduction	/
Similar experiments	SrTiO ₃ - NB-289: Modification of NB-285 (SrTiO ₃ :Al (from self-made SrTiO ₃ , Osterloh, no Al ₂ O ₃ , PVDF filter) 1000 C, 10 h) with Rh, Cr oxide cocatalyst

Reagents

Name	CAS Number / Experiment Number	Inventory number	Amount [mmol]	Equivalents	Mass _{theo} [mg]	Mass _{exp} [mg]	Molar mass [g/mol]	Density (g/ml)	Volume [ml]	Concentration [mM]
Al:SrTiO ₃	SrTiO ₃ - EA-354: Preparation of Al:SrTiO ₃ (using EA-352-SrTiO ₃ -molten-salt and SrCl ₂ as flux) at 1000°C, 10 h, Osterloh route , upscaling x15, filtration with PVDF	/	2.72494	1	500	500.12	183.49	/	/	/

RhCl ₃ .3H ₂ O (9.874 mM in H ₂ O)	Prep work - EA-357: Preparation of RhCl ₃ and Cr(NO ₃) ₃ stock solutions 20251009_1850-RhCl ₃ & Cr(NO ₃) ₃ stock solutions.jpg	/	0.004858	0.00178	/	/	263.31 102.905 for Rh content	/	0.492	9.874
Cr(NO ₃) ₃ .9H ₂ O (28.809 mM in H ₂ O)	Prep work - EA-357: Preparation of RhCl ₃ and Cr(NO ₃) ₃ stock solutions 20251009_1850-RhCl ₃ & Cr(NO ₃) ₃ stock solutions.jpg	/	0.009616	0.00352	/	/	400.15 51.996 for Cr content	/	0.334	28.809
milli-Q water	/	/	0.694	0.2545	/	/	18	1	12.504	/

EA-358-calculations.xlsx

Furnace Parameters

Equipment - Muffelofen L3/11/P320, Nabertherm GmbH, Lab 106, CEEC I, (Matilda)

Protocol - Heat treatment using Muffelofen L3/11/P320, Nabertherm GmbH, Lab 106, CEEC I

Temperature/time parameters

Used zone or charge sensor	Zone
Used delayed start	/
Used automatic/manual/extended holdback	automatic
The temperature band entered for manual/extended holdback (°C)	/
End time [min], relative to start of program	182

Segments

Program 8	Target Temperature (°C)	Duration (h)	Rate (°C/h)	Temperature band (°C)	Description of the segment	Observations
First segment	350	00:33	636	/	Increase	Initial temperature: 23°C
Second segment	350	01:00	/	/	Holding	/
End segment	/	/	/	/	Natural cooling	End temperature (before opening): 177°C

Procedure/observations

Date	Time	Step	Observations	Pictures
08.10.20 25	17:18	500.12mg of the Al:SrTiO ₃ (EA-354) was weighed into a weighing paper.	Creamy/grey clumps	20251008_1718-weighing the Al:SrTiO ₃ .jpg
	17:37	The weighed sample was transferred into a 100ml borosilicate 3.3 beaker. Then 12504µl of milli-Q water was added to the beaker using a 100-1000 Eppendorf pipette. The stirring turned on at 300rpm.	Creamy/grey suspension	20251008_1737-addition of water to the beaker.jpg
	17:38	492µl of stock solution of 9.874 mM RhCl ₃ (Prep work - EA-MEJ-357: Preparation of RhCl₃ and Cr(NO₃)₃ stock solutions) was added to the beaker using 100-1000 Eppendorf pipette	Red solution	/
	17:38	334µl of stock solution of 28.809 mM Cr(NO ₃) ₃ (Prep work - EA-MEJ-357: Preparation of RhCl₃ and Cr(NO₃)₃ stock solutions) was added to the beaker using 100-1000 Eppendorf pipette	Blue solution	/
	17:41	The beaker was transferred into the water bath (Benmari-crystallizing dish 8cm filled with water). The heating was started at 70°C.	Creamy/grey suspension	20251008_1741-start heating.jpg
	19:27	After evaporation of water, stirring was stopped.	Light grey/pink solid Two colors Not homogeneous color	20251008_1927-sample after evaporation of water.jpg
	19:34	A crystallizing dish (8cm) was put on the beaker as a lid. The beaker with lid was transferred to E002 and put inside Equipment - Muffelofen L3/11/P320, Nabertherm GmbH, Lab 106, CEEC I, (Matilda) .	Beaker with dish on top as lid	20251008_1934-the beaker with lid inside the furnace.jpg
	19:36	Heating program was designed according to Protocol - Heat treatment using Muffelofen L3/11/P320, Nabertherm GmbH, Lab 106, CEEC I and saved on P=8. Then the heating program (EA-358-P=8) started.	Initial temperature: 23°C	20251008_1936-start of program EA-358-p=8.jpg
	22:38	After cooling down, the program ended and the furnace was turned off.	End temperature (before opening): 177°C	20251008_2238-end of the heating program.jpg
	22:40	The beaker was removed from the furnace, covered with Al foil and transferred to the fume hood.	Two colors Grey and Creamy pink Not homogenized solid	20251008_2240-sample after annealing-1.jpg 20251008_2240-sample after annealing-2.jpg
09.10.20 25	19:16	The annealed sample was weighed.	Grey and Creamy pink	20251009_1916-weighing the modified sample.jpg

	19:35	The weighed sample was transferred into a 10ml snap-cap vial, mixed thoroughly with smart spatula in the vial, and covered with Al foil. Named: EA-358-Rh,Cr-oxide loaded Al:SrTiO₃	Light grey/pink clumps EA-358-Rh,Cr-oxide loaded Al:SrTiO₃	20251009_1935-EA-358-Rh,Cr-oxide loaded Al:SrTiO ₃ .jpg
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Product characterization

Sample	Mass [mg]	Purity	Mass _{pure} [mg]	Amount [μmol]	Yield [%]	Description	Image	Storage location
EA-358-Rh,Cr-oxide loaded Al:SrTiO₃	495.98	/	/	/	99.17	Light grey/pink clumps	20251009_1935-EA-358-Rh,Cr-oxide loaded Al:SrTiO ₃ .jpg	Equipment - Safety storage cabinet - CEEC II Lab E004, shelve A, Ebi container

Results

3.33 times upscaling of modification of Al:SrTiO₃ (EA-354) by loading Rh,Cr oxide co-catalysts via the impregnation method was done. The color of obtained material after evaporation of water was not homogeneous; also, after calcination, the color of the sample was still not homogeneous. The sample after evaporation of water was annealed at 350°C for 1h. The yield was 99.17%.

Future recommendations

Old procedure	Problem	Suggested new procedure
Wet impregnation followed by calcination	Non-homogenized mixture of solid detected by the color after annealing	Higher rpm for stirring and longer time of annealing Addition of co-solvents with lower boiling point.

Linked experiment

SrTiO₃ - EA-354: Preparation of Al:SrTiO₃ (using EA-352-SrTiO₃-molten-salt and SrCl₂ as flux) at 1000°C, 10 h, Osterloh route , upscaling x15, filtration with PVDF

Linked resources

Equipment - [Muffelofen L3/11/P320, Nabertherm GmbH, Lab 106, CEEC I, \(Matilda\)](#)

Equipment - [Safety storage cabinet - CEEC II Lab E004](#)

Protocol - [Heat treatment using Muffelofen L3/11/P320, Nabertherm GmbH, Lab 106, CEEC I](#)

Attached files

EA-358-calculations.xlsx

sha256: 79eff6c44de033b9d1eb0772fe1dad6bf71716b5bee857ffcc11b6e27d48eadb

EA-358.cdx

sha256: 414891c976b9f4c114bb9a01b526576b8bb7e4c15ed8d34aa656033b3f5162a3

EA-358.png

sha256: 270344108c3a1435e549066e1bf0ce236ad72c05f4567a1759891c2b331d3f5a



20251008_1718-weighing the Al:SrTiO3.jpg

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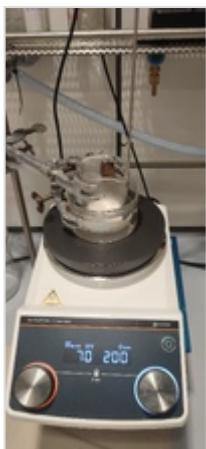
20251008_1927-sample after evaporation of water.jpg

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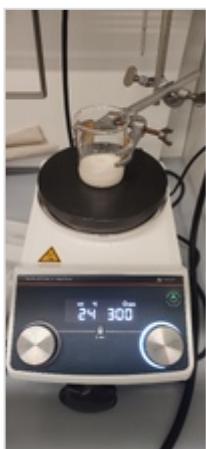
20251008_1741-start heating.jpg

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20251008_1737-addition of water to the beaker.jpg

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20251008_2240-sample after annealing-2.jpg

sha256: c6cee6c9c0b3985e3b162d2b7b0b7df4abf1459a72cf034a380c958034da9b60



20251008_2240-sample after annealing-1.jpg

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20251008_2238-end of the heating program.jpg

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20251008_1936-start of program EA-358-p=8.jpg

sha256: d5339497949801fdf290524fe7b1b00349f02bdb3ccf7066239c4731ab43cb2d



20251008_1934-the beaker with lid inside the furnace.jpg

sha256: 4cd7468f3420d357bf690c6f41732f912e882d1fde916556d046ba66189c5b15



20251009_1935-EA-358-Rh,Cr-oxide loaded Al:SrTiO₃.jpg

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20251009_1916-weighing the modified sample.jpg

sha256: dcdcc825c16967f27f23052c22f190d85b89f8257d556c03ab99ea847eee3bbf



20251009_1850-RhCl3 & Cr(NO3)3 stock solutions.jpg

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Comments

On 2025-11-23 16:54:45 Nadzeya Brezhneva wrote:

Sorry, I mistakenly cleaned experimental links in this protocol (the tab with EA-358 was opened instead of NB-352)

On 2025-11-25 01:16:47 Nadzeya Brezhneva wrote:

Reagent table: please correct the amount of Al:SrTiO₃ (the corrected amount should be equal to 2.7249 mmol , not 2724.94 mmol)



Unique eLabID: 20251009-faf7f0e426efe01a671b4b7f1c0e855ca9c6f186

Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3154>