

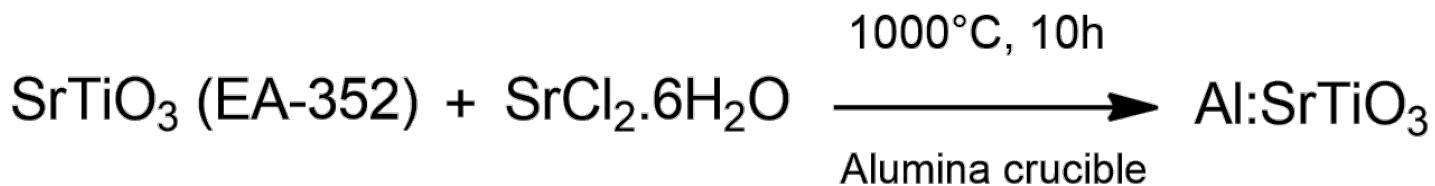
EA-354: Preparation of Al:SrTiO₃ (using EA-352-SrTiO₃-molten-salt and SrCl₂ as flux) at 1000°C, 10 h, Osterloh route , upscaling x15, filtration with PVDF

Date: 2025-10-09
Tags: EA SrTiO₃ Osterloh Al:SrTiO₃
Upscaling Filtration SrCl₂ molten salt
Category: SrTiO₃
Status: Done
Created by: Ebrahim Abedini

Objective

Doping of Al on the SrTiO₃ (EA-352) via the Osterloh route (no addition of Al₂O₃-getting the Al from the alumina crucible). 15 times upscaling.

Reaction scheme/sample structure



ChemDraw File (linked): [EA-354.cdx](#)

Literature/reference experiments

Literature	https://doi.org/10.1039/C9EE00310J
Reproduction	/
Similar experiments	SrTiO ₃ - NB-285: Preparation of SrTiO ₃ :Al (from self-made SrTiO ₃ , NB-283, batch V, Osterloh (no Al ₂ O ₃), upscaling x3), 1000 C, 10 h, filtration with PVDF

Reagents

Name	CAS Number / Experiment Number	Inventory number	Amount [mmol]	Equivalents	Mass _{theo} [g]	Mass _{exp} [g]	Molar mass [g/mol]	Density (g/ml)	Volume [ml]	Concentration [mM]
SrTiO ₃ -molten-salt (EA-352)	SrTiO ₃ - EA-352: Synthesis of SrTiO ₃ from SrCO ₃ and TiO ₂ via molten-salt approach, 1000°C, 10 h, Upscaling (2x)	/	30.246	1	15 × 0.37 = 5.55	15 × 0.37	183.49	/	/	/
SrCl ₂ *6H ₂ O, 99 % (ACS), Strem Chemicals	10025-70-4		299.864	10	15 × 5.33 = 79.95	15 × 5.33	266.62	/	/	/

Work-up and Analytical Reagents

Name	CAS Number / Experiment Number	Inventory number	Mass _{exp} [g]	Volume [ml]	Concentration [M]
milli-Q water	/	/	/	150ml for removing the sample from the crucible Washing the sample after thermal treatment to remove the SrCl ₂ flux ~ 10000ml 100ml for making the slurry	/
0.1 M AgNO ₃ solution	Prep work - ASD-040: Preparation of 0.1 M AgNO ₃ solution	/	/	ca. 0.1-0.2 (no precise values, just few drops for each Cl ⁻ test)	0.1

Furnace Parameters

Equipment - Muffle furnace Nabertherm LT 15/11/P330 (AWZ 304 lab)

Protocol - Muffle furnace Nabertherm GmbH LT 15/11/P330 (Lab AWZ 304)

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	2050

Segments

Program 7	Target Temperature (°C)	Duration (h)	Rate (°C/h)	Temperature band (°C)	Description of the segment	Observations
First segment	1000	01:38	600	/	Increase	Initial temperature: 79°C
Second segment	1000	10:00	/	/	Hold	/
End segment	/	/	/	/	Natural cooling	End temperature (before opening): 104°C

Procedure/observations

Date	Time	Step	Observations	Pictures
01.10.2025	00:54	SrCl ₂ ·6H ₂ O was weighed in a weighing bowl.	White crystals	20251001_0054-Weighing SrCl2.6H2O.jpg
	01:08	SrTiO ₃ (EA-352) was weighed in a weighing bowl.	Creamy white fine clumps	20251001_0108-weighing SrTiO3.jpg
	01:10	All of the weighed materials were transferred into an agate mortar (10cm).	Mixture of crystals and creamy white powders	20251001_0110-the components before mortaring.jpg
	01:24	The components were ground and mixed in the mortar.	Creamy white fine powder	20251001_0124-the components after mortaring.jpg
	01:28	The mortared powder was transferred into 3*150ml alumina crucibles.	Creamy white fine powder	20251001_0128-transferring the mortared powder into the alumina crucibles.jpg
	01:28	The mixture inside the crucible was pressed with the agate pestle (without applying force and just with help of weight of the pestle).	Pressed and evened white powder	20251001_0128-pressing the powder in the crucible.jpg
	01:32	The crucibles with lid were transferred to AWZ 304, CEEC II, Equipment - Muffle furnace Nabertherm LT 15/11/P330 (Lab AWZ 304 lab).	3 alumina crucibles	20251001_0132-the crucibles inside the furnace.jpg
	01:33	The heating program was designed according to Protocol - Muffle furnace Nabertherm GmbH LT 15/11/P330 (Lab AWZ 304) , and saved on P=7. The heating program was started.	Initial temperature: 79°C	20251001_0133-start of the heating program.jpg
	18:15	The temperature of the furnace was checked during the cooling phase.	T: 515°C	20251001_1815-checking the temperature of the furnace in cooling down phase.jpg
02.10.2025	11:43 - 11:44	After cooling down, the furnace was turned off and opened. The crucibles were removed from the furnace.	End temperature: 104°C	20251002_1143-end of the heating program.jpg 20251002_1144-the crucibles after heat treatment in the furnace.jpg
		Work-up procedure		

06.10.2025	12:17	The crucible was opened.	Hard, rock-shaped, grey/creamy solid Material was formed on the upper outer side of the crucibles	20251006_1217-the formed material after heat treatment.jpg 20251006_1217-the formation of the material on the upper-outer side of the crucibles.jpg
	12:19	25 mL H ₂ O was added to each crucible with the calcined sample using graduated cylinder. Using smart spatula, the suspension was mixed and the material was scratched to be dissolved. The suspension was left for 1 h for better dissolution of SrCl ₂ and its further transfer from the crucible.	Grey/creamy suspension	20251006_1219-addition of water to dissolve SrCl ₂ -before mixing.jpg 20251006_1219-addition of water to dissolve SrCl ₂ -after mixing.jpg
	13:08 - 13:11	The crucibles were transferred to the Equipment - Ultrasound Bath Fisherbrand select 300 (E004) . The crucibles were sonicated in the UltraSonication bath for 15 s ("Eco mode").	Grey/creamy suspension	20251006_1308-sonication of the crucibles-1.1.jpg 20251006_1311-spnication of the crucibles-1.2.jpg
	13:24	Transfer of the sonicated suspension from the crucible to a 600 mL glass beaker.	Grey/creamy suspension	20251006_1324-the suspensions were transferred to the beaker.jpg
	13:40	2nd round of addition of water/sonication was done.	Grey/creamy suspension	20251006_1340-2nd round of addition of water/sonication.jpg
	13:58	3rd round of addition of water/sonication was done.	Grey/creamy suspension	20251006_1358-3rd round of addition of water/sonication.jpg
	14:25	4th round of addition of water/sonication was done.	Grey/creamy suspension	20251006_1425-4th round of addition of water/sonication.jpg
	14:33	5th round of addition of water/sonication was done.	Grey/creamy suspension	20251006_1433-5th round of addition of water/sonication.jpg
	14:40	6th round of addition of water/sonication was done.	Grey/creamy suspension	20251006_1440-6th round of addition of water/sonication.jpg
	14:40	All of the suspension was collected in a 600ml beaker.	Grey/creamy suspension	20251006_1440-collecting the suspension in a beaker.jpg
	14:57	Set-up filtration with PVDF filter (0.22μm), solvent trap and pump.	/	20251006_1457-filtration setup.jpg
	15:32	Start of filtration, transfer of the suspension from the glass beaker.	/	0251006_1532-start filtration through PVDF filter.jpg 20251006_1533-start filtration.jpg
	15:37	1: Test of the presence of Cl- anions in the supernatant using 0.1 M AgNO₃. Ca. 1.5-2 mL of supernatant was transferred to a 5 mL vial. Several drops of 0.1 M AgNO ₃ solution were added to the vial using another glass pipette. The vial was swirled for several seconds	Turbid solution, positive result.	20251006_1537-Cl- test-1.jpg
	15:40 - 15:45	Pre heated warm water 70 °C was gradually added from the beaker to the filter (250 ml), while careful scratching of the solid with Smartspatula for better washing of the solid.	Grey/creamy suspension	20251006_1540-addition of warm water.jpg 20251006_1541-mixing the suspension with spatula on the filter.jpg 20251006_1545-after first round of washing.jpg
	15:46	2: Test of the presence of Cl- anions in the supernatant using 0.1 M AgNO₃. Ca. 1.5-2 mL of supernatant was transferred to a 5 mL vial. Several drops of 0.1 M AgNO ₃ solution were added to the vial using another glass pipette. The vial was swirled for several seconds	Turbid solution, positive result.	20251006_1546-Cl- test-2.jpg
07.10.2025	16:17 - 01:44	Pre heated warm water 70 °C was gradually added from the beaker to the filter (3x250ml), while careful scratching of the solid with Smartspatula for better washing of the solid. Then test of the presence of Cl- anions in the supernatant using 0.1 M AgNO₃ was done (Ca. 1.5-2 mL of supernatant was transferred to a 5 mL vial. Several drops of 0.1 M AgNO ₃ solution were added to the vial using another glass pipette. The vial was swirled for several seconds). This procedure was repeated till negative result of Cl-test.	Tests 3 - 14: Turbid solution, positive result. Test 15 - 16: Transparent solution, negative result. Total amount of warm water used for washing till this point: 10000ml	20251006_1617-Cl- test-3.jpg 20251006_1643-Cl- test-4.jpg 20251006_1728-Cl- test-5.jpg 20251006_1816-Cl- test-6.jpg 20251006_1844-Cl- test-7.jpg 20251006_1916-Cl- test-8.jpg 20251006_2000-Cl- test-9.jpg 20251006_2038-Cl- test-10.jpg 20251006_2112-Cl- test-11.jpg 20251006_2159-Cl- test-12.jpg 20251006_2245-Cl- test-13.jpg 20251006_2330-Cl- test-14.jpg 20251007_0019-Cl- test-15.jpg 20251007_0138-last round of washing.jpg 20251007_0144-Cl- test-16 next to water.jpg
	01:50	By addition of 100ml water, the slurry was made on the top of the filter.	Grey/creamy suspension	/

	01:59 - 02:03	The slurry was transferred into a petri dish (10cm). The dish was covered with Al foil with holes on it. The dish was transferred to the Equipment - Drying oven Binder FD 56 (E004, CEEC II) for further drying at 100°C, overnight.	Grey/creamy suspension	0251007_0159-transferring the slurry into the dish for drying.jpg 20251007_0203-covering the dish with Al foil.jpg
	14:18	The dried sample was removed from the oven.	Grey/creamy solid	20251007_1418-the dried sample.jpg
	14:20	The dried sample was collected from the dish with a smart spatula and weighed on the weing paper.	Grey/creamy solid	/
	14:32	The sample was transferred into a 10ml snap cap vial and covered with Al foil. Named: EA-354-Al:SrTiO3-Osterloh-15times-scaleup	Grey/creamy powder EA-354-Al:SrTiO3-Osterloh-15times-scaleup	20251007_1432-EA-354-Al:SrTiO3-Osterloh-15times-scaleup.jpg

Product characterization

Sample	Mass [mg]	Purity	Mass _{pure} [mg]	Amount [μmol]	Yield [%]	Description	Image	Storage location
EA-354-Al:SrTiO3-Osterloh-15times-scaleup	5222.61	/	/	/	94.10	Grey/creamy powder	20251007_1432-EA-354-Al:SrTiO3-Osterloh-15times-scaleup.jpg	Equipment - Safety storage cabinet - CEEC II Lab E004, Shelf A, Ebi container

Results

Al:SrTiO₃ sample using SrTiO₃-Osterloh-route without addition of Al₂O₃, annealed at 1000°C, 10 h, washed via filtration (PVDF) procedure was obtained. The yield of the final product was 94.10%.

Linked experiment

[SrTiO3 - EA-352: Synthesis of SrTiO3 from SrCO3 and TiO2, 1000°C, 10 h, Osterloh route, Upscaling \(2x\)](#)

Linked resources

[Equipment - Ultrasound Bath Fisherbrand select 300 \(E004\)](#)

[Equipment - Advanced irradiation chamber V1.0 I](#)

[Equipment - Advanced power measurement chamber V1.0 I](#)

[Equipment - Muffle furnace Nabertherm LT 15/11/P330 \(AWZ 304 lab\)](#)

[Equipment - Manual irradiation setup](#)

[Protocol - Muffle furnace Nabertherm GmbH LT 15/11/P330 \(Lab AWZ 304\)](#)

Attached files

EA-354.png

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EA-354.cdx

sha256: d072d0a147cb0e1589f747f95c6db9894d5285bd5621a629a519cb27dd0cba7d

20251001_0054-Weighing SrCl2.6H2O.jpg

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20251001_0108-weighing SrTiO3.jpg

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20251001_0110-the components before mortaring.jpg

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20251001_0124-the components after mortaring.jpg

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20251001_0128-transferring the mortared powder into the alumina crucibles.jpg

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20251001_0128-pressing the powder in the crucible.jpg

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20251001_0132-the crucibles inside the furnace.jpg

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20251001_0133-start of the heating program.jpg

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20251001_1815-checking the temperature of the furnace in cooling down phase.jpg

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20251006_1217-the formed material after heat treatment.jpg

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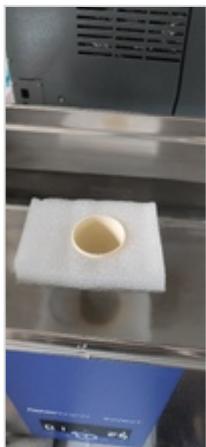
20251006_1308-sonication of the crucibles-1.1.jpg

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20251006_1311-spnciation of the crucibles-1.2.jpg

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20251006_1217-the formation of the material on the upper-outer side of the crucibles.jpg

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20251006_1219-addition of water to dissolve SrCl2-after mixing.jpg

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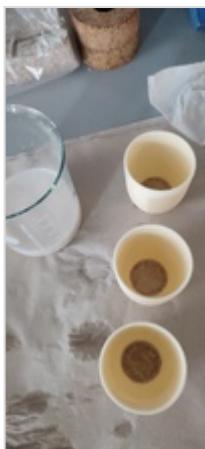


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20251006_1324-the suspensions were transferred to the beaker.jpg
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20251006_1340-2nd round of addition of water/sonication.jpg
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20251006_1533-start filtration.jpg

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20251006_1457-filtration setup.jpg

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20251006_1440-collecting the suspension in a beaker.jpg

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20251006_1440-6th round of addition of water/sonication.jpg

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20251006_1532-start filtration through PVDF filter.jpg

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20251006_1433-5th round of addition of water/sonication.jpg

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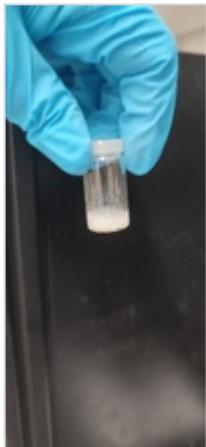
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20251006_1540-addition of warm water.jpg

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20251006_1541-mixing the suspension with spatula on the filter.jpg

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20251006_1545-after first round of washing.jpg

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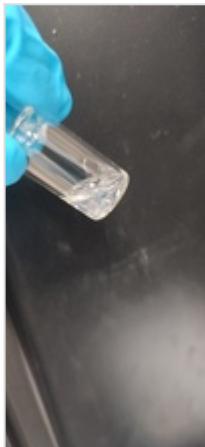
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20251006_1844-Cl- test-7.jpg

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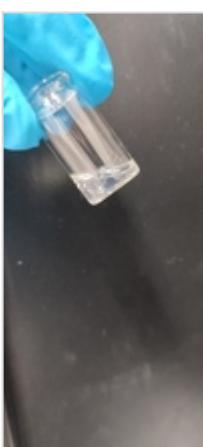
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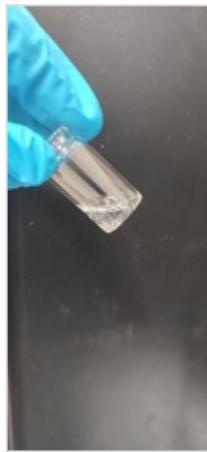
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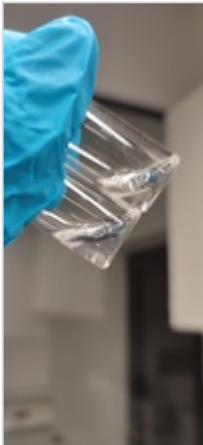
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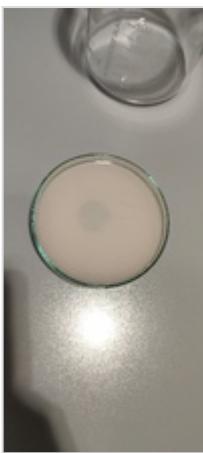


20251007_0144-Cl- test-16 next to water.jpg

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20251007_0203-covering the dish with Al foil.jpg
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20251007_1418-the dried sample.jpg
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20251002_1144-the crucibles after heat treatment in the furnace.jpg
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Comments

On 2025-11-25 13:33:04 Nadzeya Brezhneva wrote:

01.10.2025 01.33: Please adjust the link (protocol for using furnace) to a Nabertherm muffle furnace in AWZ 304, the samples were calcined there, not in 1400 °C Nabertherm furnace

On 2025-11-25 19:06:30 Ebrahim Abedini wrote:

Ebrahim: Done.



Unique eLabID: 20251009-b3dd99a4a87e97ad845f6f011e6dd41fc0b622a
Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3153>