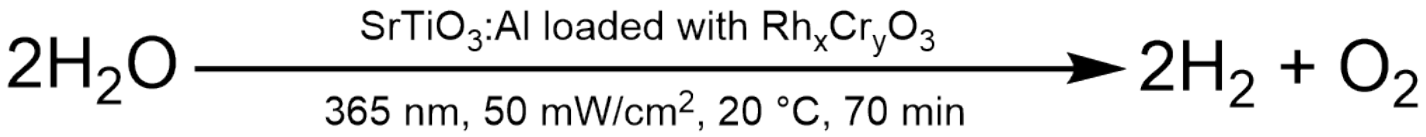
NB-363: Gas phase H2 and O2 measurements of Al:SrTiO3 RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C, 70 min, degassing (reproduction NB-362)

Date: 2025-11-26
Tags: O2 | Test | Calibration | NB | Firesting | Irradiation | O2 sensor | H2 | advanced irrad setup | Unisense | H2 Sensor | temperature | In situ | Trace range robust oxygen sensor | photocatalysis
Category: SrTiO3
Status: Done
Created by: Nadzeya Brezhneva

Objectives

Reproduction of NB-362: simultaneous detection of O_2 and H_2 evolution in gas phase for irradiated suspension of Rh,CrO_x:Al:$SrTiO_3$ suspension (EA-358, 0.5 mg/mL), 365 nm LED, 50 mW/cm^2, 20 °C during 70 min.

Reaction scheme



ChemDraw file linked: NB-362-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	/
Reproduction	SrTiO3 - NB-362: Gas phase H2 and O2 measurements of Al:SrTiO3 RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C, 70 min, degassing
Similar experiments	SrTiO3 - NB-361: Gas phase H2 and O2 measurements of Al:SrTiO3 RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C, 15 min, degassing

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]	pressure [bar]
milli-Q H_2O	/	/	/	/	/	/	/	/	25	/

Al:SrTiO3 RhCrOx (EA-358)	SrTiO3 - EA-358: Modification of Al:SrTiO3 (EA-354) via deposition of Rh, Cr oxide co-catalyst, 350°C, 1h, Upscaling (3.33x)	/	/	/	12.50	12.47	/	/	/	/

Irradiation Parameters

Power measurement was performed using Power Meter - 843-R-USB + 919P-020-12 in Equipment - Advanced power measurment setup V1.0 I

Power measurement was performed in experiment Prep work - NB-314: Measuring power output of UHP-365 nm #4 with 18A-4 in advanced irradiation setup

	Name
Used Set-up	Equipment - Advanced irradiation setup V1.0 I
Irradiation setup number	Equipment - Irradiation setup 4 (CEEC II, E002)

	Light Source Name	**Power Source Name**	**Wavelength [nm]**	**Power Setting [mW]**	**Analog Setting [0.00 - 10.00]**
First light source	Light Source - UHP LED 365 nm-4	Power Sources - BLS-18000-14	365	56	0.19

Used beam combiner [Name or None]	/
Irradiation distance [cm]	6.5
Thermostat temperature [°C]	20
Stirring speed [rpm]	500
Irradiation start: **1. Firesting [relative to start log]** **2. Unisense**	1. 610 s 2. 23:30:27

Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 4855 s 2. 0:41:12

O_2/H_2 sensor equipment

	Equipment	Used protocol
Used Firesting	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 2)	Protocol - Operation of Firesting Fiber-Optic Oxygen Meter 2 Channel Software
Used O_2 sensor	Equipment - Robust probe for liquid O2 measurment	Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor
Used H_2 sensor	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor

Procedure/observations

Date	Time	Step	Observations	Pictures/Files
25.11.2025		Calibration from experiment SrTiO3 - NB-361: Gas phase H2 and O2 measurements of Al:SrTiO3 RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C, 15 min, degassing was used.		
		Sample preparation		
	22:20	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	22:24	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	22:25-28	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	/
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	22:30	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	20251125_222810-suspension after vortex.jpg
	22:35	Assembling the setup.	/	/

	22:45	Start of O2 logging.	**NB-363-Ch2-1**	2025-11-25_224558_NB-363-Ch2-1.txt 2025-11-25_224558_NB-363-Ch2-1.png
	22:48	The degassing was started	/	20251125_225034-degassing of the suspension.jpg
	23:20	The degassing was stopped by removing the cannula and closing the valve.	/	/
	23:19	Stop of O2 logging.	/	/
	23:20	Start of O2 logging.	**NB-363-Ch2-2**	2025-11-25_232017_NB-363-Ch2-2.txt 2025-11-25_232017_NB-363-Ch2-2.png
	23:20	Start of H2 logging.	**NB-363-Logger1**	NB-363.ulog NB-363-Logger1.csv NB-363-Logger1.bmp
	23:20-30	Equilibration time.	/	/
	23:30	The irradiation was started	After 20:10 the O_2 and H_2 curves reached plateau	20251125_233106-after start of irradiation.jpg
26.11.2025	0:40	The irradiation was stopped.	/	/
	0:40-51	Equilibration time.	/	/
	0:51	Stop of O2 and H2 logging.	/	/
	ca. 1:00	Deassembling the setup, cleaning the reactor.	Tip: After preliminary cleaning with sticks, wipes, the residual particles attached to the walls of the reactor could be removed by sonication - fill the reactor with water and place it in ultrasonic bath for ca. 20 s (Eco mode).	/

Analysis

Used calibration for Firesting: 20250910-BOLA fitting-gas phase-4-neck photoreactor-trace oxygen robust probe-Ch2.ini

Used calibration for UniSense: NB-361-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
25.11.2025	23:20	NB-363-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-363.ulog NB-363-Logger1.csv	NB-363-O2 and H2 curve.py	NB-363-Logger1.bmp NB-363-O2 and H2 curves.png	SrTiO3 - NB-362: Gas phase H2 and O2 measurements of Al:SrTiO3 RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C, 70 min, degassing	H2 evolution during irradiation
	22:45	NB-363-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-25_224558_NB-363-Ch2-1.txt	/	2025-11-25_224558_NB-363-Ch2-1.png	/	Degassing of the suspension.
	23:20	NB-363-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-25_232017_NB-363-Ch2-2.txt	NB-363-O2 and H2 curve.py	2025-11-25_232017_NB-363-Ch2-2.png NB-363-O2 and H2 curves.png	SrTiO3 - NB-362: Gas phase H2 and O2 measurements of Al:SrTiO3 RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C, 70 min, degassing	O2 evolution during irradiation

Results

Reproduction of NB-362: simultaneous H_2 and O_2 measurements (gas phase) of irradiated suspension of EA-358 (0.5 mg/mL) in O_2/H_2 photoreactor under 365 nm irradiation (50 mW/cm^2, 20 °C**,** 70 min) were performed.

Linked experiments

SrTiO3 - NB-312: Gas phase H2 and O2 measurements with Unisense H2 sensor, Firesting O2 robust probe in irradiated Al:SrTiO3 RhCrOx (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing (reproduction NB-304)

SrTiO3 - NB-361: Gas phase H2 and O2 measurements of Al:SrTiO3 RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C, 15 min, degassing

SrTiO3 - NB-362: Gas phase H2 and O2 measurements of Al:SrTiO3 RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C, 70 min, degassing

Linked resources

- Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 2)
- Equipment - Robust probe for liquid O2 measurment
- Equipment - Advanced irradiation chamber V1.0 I
- Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle
- Equipment - Irradiation setup 4 (CEEC II, E002)
- Protocol - Operation of Firesting Fiber-Optic Oxygen Meter 2 Channel Software
- Protocol - Getting hydrogen from hydrogen bottle in CEEC II E014
- Protocol - Gas phase calibration of H2 UniAmp sensor
- Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor

Attached files

NB-363-O2 and H2 curve.py
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NB-363-O2 and H2 curves.png
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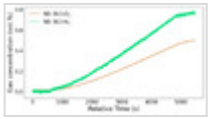

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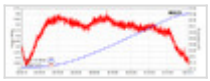

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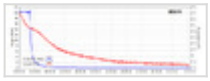

NB-363-Logger1.bmp
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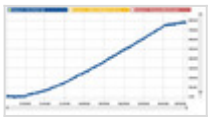

NB-363.ulog
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20251125_225034-degassing of the suspension.jpg
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20251125_222810-suspension after vortex.jpg
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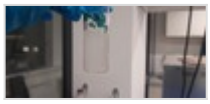

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20251126_005240-after irradiation.jpg
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Link: https://elab.water-splitting.org/experiments.php?mode=view&id=3558