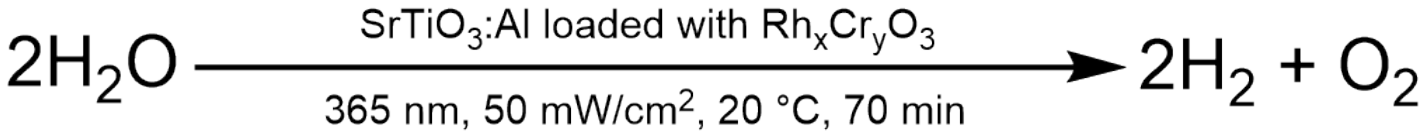
NB-362: Gas phase H2 and O2 measurements of Al:SrTiO3 RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C, 70 min, degassing

Date: 2025-11-26
Tags: O2 | Test | Calibration | Future | 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

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	/
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	12.61	/	/	/	/

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. 19:09:08
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 4830 s 2. 20:19:29

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		
	17:50	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	17:52	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	17:55-58	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251125_180013-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	18:00	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	18:05	Assembling the setup.	/	/
	18:16	Start of O2 logging.	**NB-362-Ch2-1**	2025-11-25_181629_NB-362-Ch2-1.txt 2025-11-25_181629_NB-362-Ch2-1.png
	18:17	The degassing was started	/	20251125_181835-degassing of the suspension.jpg

	18:54	The degassing was stopped by removing the cannula and closing the valve.	/	/
	18:55	Stop of O2 logging.	/	/
	18:58	Start of O2 logging.	**NB-362-Ch2-2**	2025-11-25_185858_NB-362-Ch2-2.txt 2025-11-25_185858_NB-362-Ch2-2.png
	18:58	Start of H2 logging.	**NB-362-Logger1**	NB-362.ulog NB-362-Logger1.csv NB-362-Logger1.bmp
	18:58-19:09	Equilibration time.	/	/
	19:09	The irradiation was started	After 20:10 the O_2 and H_2 curves reached plateau	20251125_191001-after start of irradiation.jpg
	20:19	The irradiation was stopped.	/	/
	20:19-30	Equilibration time.	/	/
	20:30	Stop of O2 and H2 logging.	/	/
	ca. 20:40	Deassembling the setup, cleaning the reactor.	When the front lid of the setup was opened, it was noticed that the stirring bar was not stirred properly. 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	18:58	NB-362-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-362.ulog NB-362-Logger1.csv	NB-362-O2 and H2 curve.py	NB-362-Logger1.bmp NB-362-O2 and H2 curves.png	/	H2 evolution during irradiation
	18:16	NB-362-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-25_181629_NB-362-Ch2-1.txt	/	2025-11-25_181629_NB-362-Ch2-1.png	/	Degassing of the suspension.
	18:58	NB-362-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-25_185858_NB-362-Ch2-2.txt	NB-362-O2 and H2 curve.py	2025-11-25_185858_NB-362-Ch2-2.png NB-362-O2 and H2 curves.png	/	O2 evolution during irradiation

Results

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. After 20:10 change in slope in both O2 and H2 values was observed.

After opening the lid, the problems with stirring were observed (most probably, it changed the slope in O2 and H2 curves during irradiation).

Futture recommendations

Old procedure	Problem	Suggested new procedure
/	Problems with stirring were noticed (the problem was found out after opening the lid of the setup).	Check the stirring bar or replace it with a new one if necessary.

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

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

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-362-O2 and H2 curve.py
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NB-362-O2 and H2 curves.png
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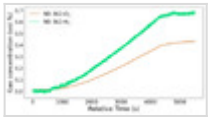

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20251125_181835-degassing of the suspension.jpg
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20251125_191001-after start of irradiation.jpg
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20251125_180013-suspension after vortex.jpg
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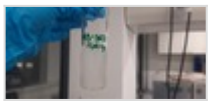

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NB-362-Logger1.bmp
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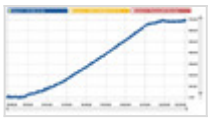

NB-362.ulog
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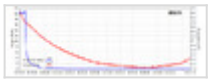

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