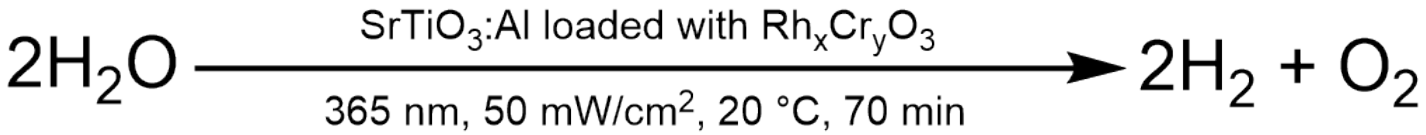
NB-364: Gas phase H2 and O2 measurements of Al:SrTiO3 RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C, 70 min, degassing (reproduction NB-363)

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-363: 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-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)
Similar experiments	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 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	/	/	/	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	1 balloon (ca. 1 L)	ca. 1

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. 615 s 2. 16:06:16
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 4830 s 2. 17:16:31

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		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor Important steps and deviations are listed below	/	/
		Important note: replacing 10 mm BOLA fitting to 2 mm BOLA fitting for H2 sensor.		
	11:39-13:06	Conditioning of H2 sensor	**NB-364-Logger1** offset - 2 mV	NB-364.ulog NB-364-Logger1-pre-polarization.csv NB-364-Logger1-pre-polarization.bmp
	13:15-20	Assembling the setup for calibration (25 mL of water was added using graduated cylinder), (LAUDA set to 20 °C) done according to Protocol - Liquid phase calibration of H2 UniAmp sensor with H2 bubbling.	/	

	13:24	Start of O2 logging.	**NB-364-Ch2-1**	2025-11-26_132450_NB-364-Ch2-1.txt 2025-11-26_132450_NB-364-Ch2-1.png
	13:25	Start of H2 logging.	**NB-364-Logger2** offset - 2 mV	NB-364.ulog NB-364-Logger2-calibration.csv NB-364-Logger2-calibration step.bmp
	13:27	Degassing was started.	/	20251126_132753-degassing of water.jpg
	14:11	0 ppm was taken.	/	/
	14:13	H2 bubbling of the reactor was started	/	20251126_141600-H2 bubbling.jpg
	14:18	1.000.000 ppm point was taken and calibration was used.	898 mV, slope 0.009, 97334 Pa	20251126_141816-H2 table.jpg
	14:22	Stop of H2 logging.	/	/
	14:22	Stop of O2 logging.	/	/
	14:30	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
		Sample preparation		
	14:50	Weighing **EA-358** photocatalyst in a 50 mL vial.	Creamy solid.	/
	14:55	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	14:59-15:02	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251126_150223-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	15:05	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	15:10	Assembling the setup.	/	/

	15:18	Start of O2 logging.	**NB-364-Ch2-2**	2025-11-26_151821_NB-364-Ch2-2.txt 2025-11-26_151821_NB-364-Ch2-2.png
	15:21	The degassing was started	/	20251126_152140-degassing of the suspension.jpg
	15:54	The degassing was stopped by removing the cannula and closing the valve.	/	/
	15:55	Stop of O2 logging.	/	
	15:56	Start of O2 logging.	**NB-364-Ch2-3**	2025-11-26_155601_NB-364-Ch2-3.txt 2025-11-26_155601_NB-364-Ch2-3.png
	15:56	Start of H2 logging.	**NB-364-Logger3**	NB-364.ulog NB-364-Logger3-during irradiation.csv NB-364-Logger3-during irradiation.bmp
	15:56-16:06	Equilibration time.	/	/
	16:06	The irradiation was started	/	20251126_160700-after start of irradiation.jpg
	17:16	The irradiation was stopped.	/	/
	17:16-26	Equilibration time.	/	/
	17:26	Stop of O2 and H2 logging.	/	/
	17:30	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).	20251126_175358-after irradiation.jpg

Analysis

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

Used calibration for UniSense: NB-364-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
25.11.2025	11:39	NB-364-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-364.ulog NB-364-Logger1-pre-polarization.csv	/	NB-364-Logger1-pre-polarization.bmp	/	Pre-polarization of the sensor.
	13:25	NB-364-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-364.ulog NB-364-Logger2-calibration.csv	/	NB-364-Logger2-2point calibration.bmp NB-364-Logger2-calibration step.bmp	/	Calibration of H2 sensor, 10^6 ppm corresponds to 898 mV
	15:56	NB-364-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-364.ulog NB-364-Logger3-during irradiation.csv	NB-364-O2 and H2 curve.py	NB-364-Logger3-during irradiation.bmp NB-364-O2 and H2 curves.png	SrTiO3 - 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)	H2 evolution during irradiation.
	13:24	NB-364-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-26_132450_NB-364-Ch2-1.txt	/	2025-11-26_132450_NB-364-Ch2-1.png	/	Degassing of water followed by calibration of H2 sensor.
	15:18	NB-364-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-26_151821_NB-364-Ch2-2.txt	/	2025-11-26_151821_NB-364-Ch2-2.png	/	Degassing of the suspension.
	15:56	NB-364-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-26_155601_NB-364-Ch2-3.txt	NB-364-O2 and H2 curve.py	2025-11-26_155601_NB-364-Ch2-3.png NB-364-O2 and H2 curves.png	SrTiO3 - 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)	O2 evolution during irradiation.

Results

Reproduction of NB-363: 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. Problems with stirring were eliminated.

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

SrTiO3 - 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)

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-364-O2 and H2 curves.png
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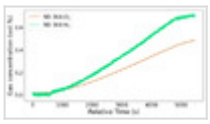

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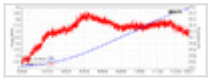

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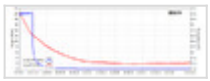

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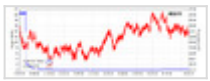

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NB-364-Logger2-2point calibration.bmp
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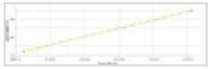

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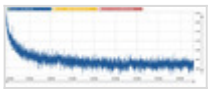

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NB-364-Logger3-during irradiation.csv
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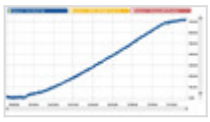

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