NB-351: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C

Date: 2025-11-18

Tags: O2 Test Calibration NB Firesting O2 sensor H2 SrTiO3 Unisense RhCrO3:Al:SrTiO3 H2 Sensor temperature In situ H2/O2 reactor Trace range robust oxygen sensor photocatalysis Unisense normal range
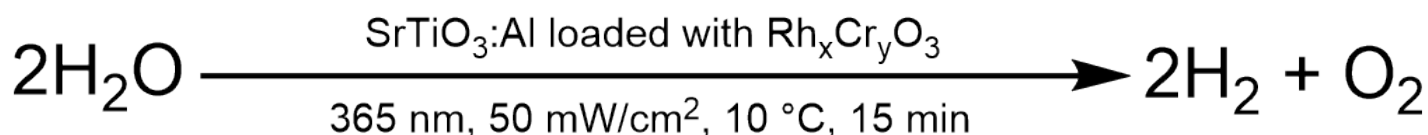
Category: SrTiO3
Status: Done
Created by: Nadzeya Brezhneva

Objectives

Simultaneous detection of H_2 and O_2 evolution in liquid 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, **10 °C**.

Reaction scheme

$$2H_2O \xrightarrow[\text{365 nm, 50 mW/cm}^2\text{, 10 °C, 15 min}]{\text{SrTiO}_3\text{:Al loaded with Rh}_x\text{Cr}_y\text{O}_3} 2H_2 + O_2$$

ChemDraw file linked: NB-351-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	/
Reproduction	/
Similar experiments	SrTiO3 - NB-316: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C

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]	Concentration [mM]
milli-Q H_2O	/	/	/	/	/	/	/	0.998	25.00 + 25 (for calibration)	/	/
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	12.67	/	/	/	/	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	2 balloons (ca. 2 L)	approx. 1	/

Excel sheet for reagent calculation

/

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]	10
Stirring speed [rpm]	500
Irradiation start: **1. Firesting [relative to start log]** **2. Unisense**	1. 603 s 2. 21:26:03
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 1518 s 2. 21:41:17

O₂/H₂ 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 O₂ sensor	Equipment - Robust probe for liquid O2 measurment	Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor
Used H₂ 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
18.11.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor Important steps and deviations are listed below	/	/
	9:13-10:28	Conditioning of H2 sensor	**NB-351-Logger1** 1.6 mV at the end of polarization procedure	NB-351.ulog NB-351-Logger1-pre-polarization.csv NB-351-Logger1-pre-polarization.bmp
	16:49	Assembling the setup for calibration (25 mL of water was added using graduated cylinder), (LAUDA set to 10 °C) done according to Protocol - Liquid phase calibration of H2 UniAmp sensor with H2 bubbling.	/	/
	16:50-17:03	Keeping the system under stirring to reach 10 °C (T control by PT1000).	/	/
	17:04	Start of O2 logging.	**NB-351-Ch2-2**	2025-11-18_170449_NB-351-Ch2-2txt 2025-11-18_170449_NB-351-Ch2-2.png
	17:06	Degassing was started.	/	20251118_170710-degassing of water.jpg

	17:07	Start of H2 logging.	**NB-351-Logger2** offset -2 mV	NB-351.ulog NB-351-Logger2-calibration.csv NB-351-2point calibration.bmp NB-351-Logger2-calibration step.bmp
	17:26	Moving cannula to gas phase above the liquid level.	/	/
	17:29	Introducing H2 sensor into the reactor under Ar flow.	/	/
	17:30	0 ppm was taken.	/	/
	17:37	Degassing was stopped.	/	/
	17:38	H2 bubbling of the reactor was started	/	/
	17:46	Introducing second H2 balloon.	Drop in H2 value after addition of the 2nd balloon, but afterwards increased and could reach steady state.	20251118_174602-after introducing 2nd H2 balloon.jpg
	17:55	1.000.000 ppm point was taken and calibration was saved	640 mV, slope: 0.750, 856 uM	/
	18:02	Stop of H2 logging.	/	/
	18:02	Stop of O2 logging.	/	/
	ca. 18:10	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
		Sample preparation		
	19:15	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	19:20	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/

	19:22-25	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251118_192609-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	ca. 19:35	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	19:40	Assembling the setup.	Currently, stopper instead of H_2 sensor, PT100, PT1000 and O_2 robust probe are inside the reactor immersed in the liquid phase	/
	19:40-50	Keeping suspension at 10 °C, T control by PT1000.	/	/
	19:50	Start of O2 logging.	**NB-351-Ch2-3**	2025-11-18_195017_NB-351-Ch2-3.txt 2025-11-18_195017_NB-351-Ch2-3.png
	19:52	The degassing was started	/	20251118_195316-degassing of the suspension.jpg
	20:30	Cannula was transferred to gas phase, above the suspension.	/	/
	20:34	H_2 sensor was added in Ar counterflow.	/	/
	20:36	The degassing was stopped by removing the cannula and closing the valve.	/	/
	20:38	Stop of O2 logging.	/	/
	20:38	Start of O2 logging.	**NB-351-Ch2-4**	2025-11-18_203830_NB-351-Ch2-4.txt 2025-11-18_203830_NB-351-Ch2-4.png
	20:38	Start of H2 logging.	**NB-351-Logger3** High H2 signal after introducing sensor to the reactor.	NB-351.ulog NB-351-Logger3.csv NB-351-Logger3.bmp

	Time	Action	Note	Files
	20:43	Stop of H2 and O2 loggings.	Removing the sensor from the reactor, introducing once again, checking cable management.	/
	20:48	Start degassing once again.	**NB-351-Ch2-5**	2025-11-18_204849_NB-351-Ch2-5.txt 2025-11-18_204849_NB-351-Ch2-5.png
	21:07	Moving cannula to gas phase above the liquid level.	/	/
	21:10	Introducing H2 sensor under Ar flow.	/	/
	21:13	Removing cannula, closing the valve.	/	/
	21:15	Stop of O2 logging.	/	/
	21:16	Start of O2 logging.	**NB-351-Ch2-6**	2025-11-18_211601_NB-351-Ch2-6.txt 2025-11-18_211601_NB-351-Ch2-6.png
	21:16	Start of H2 logging.	**NB-351-Logger4**	NB-351.ulog NB-351-Logger4-during irradiation.csv NB-351-Logger4-during irradiation.bmp
	21:16-26	Equilibration time.	/	/
	21:26	The irradiation was started	/	20251118_212721-after start of irradiation.jpg
	21:41	The irradiation was stopped.	/	/
	21:41-51	Equilibration time.	/	/
	21:51	Stop of O2 and H2 logging.	/	/

	ca. 22:00	Deassembling the setup, cleaning the reactor.	Tips of the sensors and reactor were covered with attached photocatalyst particles. 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).	20251118_215222-after irradiation.jpg

Analysis

Used calibration for Firesting: 20250611-BOLA-fitting-liquid-phase-trace-oxygen-sensor-H2-O2 reactor.ini

Used calibration for UniSense: NB-351-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
18.11.2025	9:13	NB-351-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-351.ulog NB-351-Logger1-pre-polarization.csv	/	NB-351-Logger1-pre-polarization.bmp	/	Pre-polarization of H2 sensor.
	17:07	NB-351-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-351.ulog NB-351-Logger2-calibration.csv	/	NB-351-2point calibration.bmp NB-351-Logger2-calibration step.bmp	/	Calibration of H2 sensor, at 10^6 ppm signal 640 mV, slope 0.750
	20:38	NB-351-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-351.ulog NB-351-Logger3.csv	/	NB-351-Logger3.bmp	/	High signal after introducing H2 snsor to the suspension --> needs to be eliminated.
	21:16	NB-351-Logger4	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-351.ulog NB-351-Logger4-during irradiation.csv	NB-351-O2 and H2 curve.py	NB-351-Logger4-during irradiation.bmp NB-351-O2 and H2 curves.png		H2 evolution during irradiation.
	17:04	NB-351-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-18_170449_NB-351-Ch2-2txt	/	2025-11-18_170449_NB-351-Ch2-2.png	/	Degassing of water followed by calibration of H2 sensor.
	19:50	NB-351-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-18_195017_NB-351-Ch2-3.txt	/	2025-11-18_195017_NB-351-Ch2-3.png	/	First degassing.

20:38	NB-351-Ch2-4	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-18_203830_NB-351-Ch2-4.txt	/	2025-11-18_203830_NB-351-Ch2-4.png	/	Check of O2 value during 1st logging - first attempt to start pre-reaction baseline.
20:48	NB-351-Ch2-5	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-18_204849_NB-351-Ch2-5.txt	/	2025-11-18_204849_NB-351-Ch2-5.png	/	Degassing after second introduction of H2 sensor in the suspension.
21:16	NB-351-Ch2-6	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-18_211601_NB-351-Ch2-6.txt	NB-351-O2 and H2 curve.py	2025-11-18_211601_NB-351-Ch2-6.png NB-351-O2 and H2 curves.png	/	O2 evolution during irradiation.

Results

Simultaneous H_2 and O_2 measurements of irradiated suspension of EA-358 (0.5 mg/mL) in O_2/H_2 photoreactor under 365 nm irradiation (50 mW/cm^2, 10 °C, 15 min) were performed.

Linked experiments

SrTiO3 - NB-316: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C

SrTiO3 - NB-318: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 100 mW/cm2, 20 °C

SrTiO3 - NB-322: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 100 mW/cm2, 20 °C (reproduction NB-318)

SrTiO3 - NB-325: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm2, 20 °C

SrTiO3 - NB-330: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 30 °C

SrTiO3 - NB-334: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 30 °C (reproduction NB-330)

Linked resources

Equipment - VWR® VV3, Vortex Mixer

Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 2)

Equipment - Advanced power measurment chamber V1.0 I

Equipment - Irradiation setup 4 (CEEC II, E002)
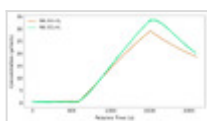
Light Source - UHP LED 365 nm-4

Power Sources - BLS-18000-1 4

Protocol - Getting hydrogen from hydrogen bottle in CEEC II E014

Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor

Attached files

NB-351-O2 and H2 curves.png
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NB-351-O2 and H2 curve.py
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NB-351-SrTiO3-photocatalytic H2O splitting.cdxml
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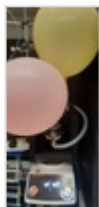

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20251118_174602-after introducing 2nd H2 balloon.jpg
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20251118_215222-after irradiation.jpg
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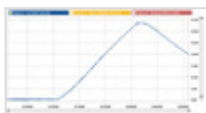sha256: 8726f844e90506532841db2a770e4e3ceb09ab2b71d9454be63dc0756c97a474

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NB-351-Logger4-during irradiation.csv
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NB-351-Logger4-during irradiation.bmp
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NB-351-Logger3.bmp
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NB-351-Logger3.csv
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NB-351-Logger2-calibration.csv
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NB-351-2point calibration.bmp
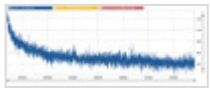sha256: 8485642c00c99066d352e9d32fb15e269125cf5a326845994cb80ceb3af5461e

NB-351-Logger2-calibration step.bmp
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NB-351-Logger1-pre-polarization.bmp
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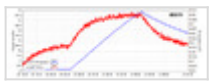

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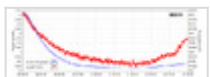sha256: f28085422f632509b32204e4ae86b476d5029949c1e563ae36842199202c75ed

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