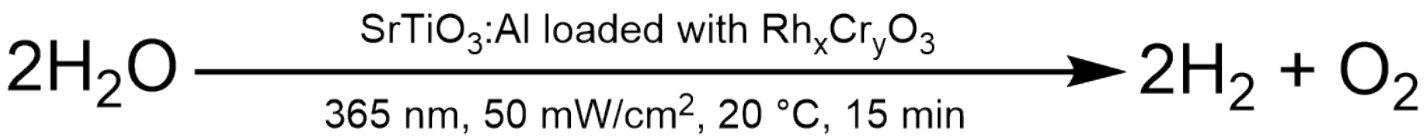
NB-356: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C IX (reproduction NB-316)

Date: 2025-11-20
Tags: O2 | Test | Calibration | Future | NB | Firesting | O2 sensor | H2 | SrTiO3 | troubleshooting | Unisense | RhCrO3:Al:SrTiO3 | H2 Sensor | temperature | In situ | Trace range robust oxygen sensor | photocatalysis | Unisense normal range
Category: SrTiO3
Status: Done
Created by: Nadzeya Brezhneva

Objectives

Reproduction of NB-316: simultaneous detection of H_2 and O_2 evolution in liquid phase for irradiated suspension of Rh,CrOx:Al:SrTiO3 suspension (EA-358 sample, 0.5 mg/mL), 365 nm, 50 mW/cm^2, 20 °C, 15 min (reference conditions).

Reaction scheme



ChemDraw file linked: NB-316-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	/
Reproduction	SrTiO3 - NB-320: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C (reprodution NB-316) I SrTiO3 - NB-329: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C (reprodution NB-316) I I SrTiO3 - NB-331: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C (reprodution NB-316) I I I SrTiO3 - NB-336: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C V (reproduction NB-316) SrTiO3 - NB-339: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C VI (reproduction NB-316) SrTiO3 - NB-348: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C VII (reproduction NB-316)
Similar experiments	SrTiO3 - NB-315: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-301, 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 + 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)	/	/	/	12.5	12.60	/	/	/	/	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	1 balloon (ca. 2 L)	ca. 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]	20
Stirring speed [rpm]	500
Irradiation start: **1. Firesting [relative to start log]** **2. Unisense**	1. 600 s 2. 23:10:16
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 1516 s 2. 23:25:28

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
20.11.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor Important steps and deviations are listed below	/	/

	10:22-11:36	Conditioning of H2 sensor	**NB-356-Logger1** 1.69 mV at the end of polarization procedure	NB-356.ulog NB-356-Logger1-pre-polarization.csv NB-356-Logger1-pre-polarization.bmp
	16:00-05	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.	/	/
	16:14	Start of O2 logging.	**NB-356-Ch2-1**	2025-11-20_161452_NB-356-Ch2-1.txt 2025-11-20_161452_NB-356-Ch2-1.png
	16:16	Start of H2 logging.	**NB-356-Logger2** offset -2 mV	NB-356.ulog NB-356-Logger2-calibration.csv NB-356-Logger2-2point calibration.bmp NB-356-Logger2-calibration step.bmp
	16:18	Degassing was started.	/	20251120_161902-degassing of water.jpg
	17:12	Moving cannula to gas phase.	/	/
	17:15	Introducing H2 sensor into the reactor under Ar flow.	/	/
	17:24	0 ppm was taken.	/	/
	17:26	H2 bubbling of the reactor was started	/	20251120_172742-H2 bubbling.jpg
	17:45	1.000.000 ppm point was taken and calibration was saved	847 mV, slope: 1.076, 787 uM	20251120_174523-H2 table.jpg
	17:46	Stop of H2 logging.	/	/
	17:46	Stop of O2 logging.	/	/
	ca. 18:00	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
		Sample preparation		
	21:40	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/

	21:44	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	21:46-49	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251120_215000-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	22:00	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	22:05	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	/
	22:13	Start of O2 logging.	**NB-356-Ch2-2**	2025-11-20_221307_NB-356-Ch2-2.txt 2025-11-20_221307_NB-356-Ch2-2.png
	22:15	The degassing was started	/	20251120_221526-degassing of the suspension.jpg
	22:50	Cannula was transferred to gas phase, above the suspension.	/	/
	22:54	H_2 sensor was added in Ar counterflow.	/	/
	22:58	The degassing was stopped by removing the cannula and closing the valve.	/	/
	22:59	Stop of O2 logging.	/	/
	23:00	Start of O2 logging.	**NB-356-Ch2-3**	2025-11-20_230014_NB-356-Ch2-3.txt 2025-11-20_230014_NB-356-Ch2-3.png
	23:00	Start of H2 logging.	**NB-356-Logger3**	NB-356.ulog NB-356-Logger3-during irradiation.csv NB-356-Logger3-during irradiation.bmp
	23:00-10	Equilibration time.	/	/
	23:10	The irradiation was started	/	20251120_231241-after start of irradiation.jpg

	23:25	The irradiation was stopped.	/	/
	23:25-35	Equilibration time.	/	/
	23:35	Stop of O2 and H2 logging.	/	/
	ca. 23:50	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).	20251121_000151-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-356-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
20.11.2025	10:22	NB-356-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-356.ulog NB-356-Logger1-pre-polarization.csv	/	NB-356-Logger1-pre-polarization.bmp	/	Pre-polarization of the sensor.
	16:16	NB-356-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-356.ulog NB-356-Logger2-calibration.csv	/	NB-356-Logger2-2point calibration.bmp NB-356-Logger2-calibration step.bmp	/	Calibration of H2 sensor, signal at 10^6 ppm - 847 mV, slope 1.076
	23:00	NB-356-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-356.ulog NB-356-Logger3-during irradiation.csv	NB-356-O2 and H2 curve.py	NB-356-Logger3-during irradiation.bmp NB-356-O2 and H2 curves.png	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	H2 evolution during irradiation.
	16:14	NB-356-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-20_161452_NB-356-Ch2-1.txt	/	2025-11-20_161452_NB-356-Ch2-1.png	/	Degassing of water followed by calibration of H2 sensor.

	22:13	NB-356-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-20_221307_NB-356-Ch2-2.txt	/	2025-11-20_221307_NB-356-Ch2-2.png	/	Degassing of the suspension.
	23:00	NB-356-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-20_230014_NB-356-Ch2-3.txt	NB-356-O2 and H2 curve.py	2025-11-20_230014_NB-356-Ch2-3.png NB-356-O2 and H2 curves.png	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	O2 evolution during irradiation.

Results

Reproduction of NB-316, 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, 20 °C**,** 15 min, reference conditions) 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-336: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C V (reproduction NB-316)

SrTiO3 - NB-339: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C VI (reproduction NB-316)

SrTiO3 - NB-348: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C VII (reproduction NB-316)

SrTiO3 - NB-353: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C VIII (reproduction NB-316)

Linked resources

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

Equipment - Irradiation setup 4 (CEEC II, E002)

Protocol - Getting hydrogen from hydrogen bottle in CEEC II E014

Protocol - Liquid phase calibration of H2 UniAmp sensor

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

Attached files

NB-356-O2 and H2 curves.png
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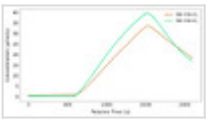

NB-356-O2 and H2 curve.py
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20251120_231241-after start of irradiation.jpg
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NB-356.ulog
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NB-356-Logger3-during irradiation.bmp
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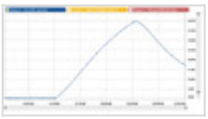

NB-356-Logger3-during irradiation.csv
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NB-356-Logger2-calibration.csv
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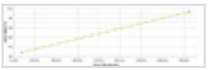

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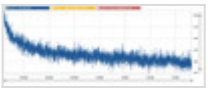

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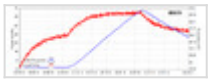

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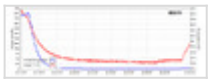

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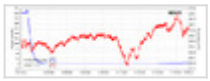

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