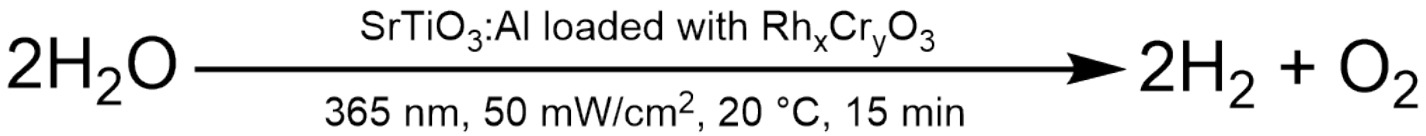
NB-353: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C VIII (reproduction NB-316)

Date: 2025-11-19
Tags: O2 | Test | Calibration | NB | Firesting | O2 sensor | H2 | SrTiO3 | 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-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-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 (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)	/	/	/	12.5	12.54	/	/	/	/	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	1 balloon (ca. 1 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. 604 s 2. 16:10:04
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1.1517 s 2. 16:25:18

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**
19.11.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor Important steps and deviations are listed below	/	/
	9:58-11:05	Conditioning of H2 sensor	**NB-353-Logger1** 1.76 mV at the end of polarization procedure	NB-353.ulog NB-353-Logger1-pre-polarization.bmp NB-353-Logger1-pre-polarization.csv

	11:15-30	Assembling the setup for calibration (25 mL of water was added using graduated cylinder), (LAUDA set to 0 °C) done according to Protocol - Liquid phase calibration of H2 UniAmp sensor with H2 bubbling.	/	/
	11:43	Start of O2 logging.	**NB-353-Ch2-1**	2025-11-19_114341_NB-353-Ch2-1.txt 2025-11-19_114341_NB-353-Ch2-1.png
	11:43	Start of H2 logging.	**NB-353-Logger2** offset -2 mV	NB-353.ulog NB-353-Logger2-calibration.csv NB-353-Logger2-calibration step.bmp NB-353-Logger2-2point calibration.bmp
	11:48	Degassing was started.	/	20251119_114802-degassing of water.jpg
	12:19	Moving cannula to gas phase above liquid.	/	/
	12:22	Introducing H2 sensor into the reactor under Ar flow.	/	/
	12:24	0 ppm was taken.	/	/
	12:25	H2 bubbling of the reactor was started	/	20251119_122724-H2 bubbling.jpg
	12:42	1.000.000 ppm point was taken	837 mV	/
	12:46	The previous point was deleted, new point was taken and calibration was saved	839 mV, slope: 1.058, 793 uM	20251119_124232-H2 table.jpg
	12:47	Stop of H2 logging.	/	/
	12:47	Stop of O2 logging.	/	/
	13:00	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
		Sample preparation		
	14:43	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	14:45	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	14:52-55	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251119_145537-suspension after vortex.jpg

		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	15:00-05	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	15:10	Assembling the setup.	Currently, stopper instead of H_2 sensor, PT100, PT1000 and O_2 robust probe are inside the reactor immersed in the liquid phase	/
	15:15	Start of O2 logging.	**NB-353-Ch2-2**	2025-11-19_151536_NB-353-Ch2-2.txt 2025-11-19_151536_NB-353-Ch2-2.png
	15:17	The degassing was started	/	20251119_151820-degassing of the suspension.jpg
	15:49	Cannula was transferred to gas phase, above the suspension.	/	/
	15:52	H_2 sensor was added in Ar counterflow.	/	/
	15:57	The degassing was stopped by removing the cannula and closing the valve.	/	/
	15:59	Stop of O2 logging.	/	/
	16:00	Start of O2 logging.	**NB-353-Ch2-3**	2025-11-19_160001_NB-353-Ch2-3.txt 2025-11-19_160001_NB-353-Ch2-3.png
	16:00	Start of H2 logging.	**NB-353-Logger3**	NB-353.ulog NB-353-Logger3-during irradiation.csv NB-353-Logger3-during irradiation.bmp
	16:00-10	Equilibration time.	/	/
	16:10	The irradiation was started	/	20251119_161145-start of irradiation.jpg
	16:25	The irradiation was stopped.	/	/
	16:25-35	Equilibration time.	/	/
	16:35	Stop of O2 and H2 logging.	/	/

	ca. 16:50	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).	20251119_164458-after irradiation.jpg 20251119_164405-after irradiation-1.jpg

Analysis

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

Used calibration for UniSense: NB-353-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
19.11.2025	9:58	NB-353-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-353.ulog NB-353-Logger1-pre-polarization.csv	/	NB-353-Logger1-pre-polarization.bmp	/	Pre-polarization of H2 sensor.
	11:43	NB-353-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-353.ulog NB-353-Logger2-calibration.csv	/	NB-353-Logger2-calibration step.bmp NB-353-Logger2-2point calibration.bmp	/	2 point calibration, signal at 10^6 ppm - 839 mV
	16:00	NB-353-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-353.ulog NB-353-Logger3-during irradiation.csv	NB-353-O2 and H2 curve.py	NB-353-Logger3-during irradiation.bmp NB-353-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.
	11:43	NB-353-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-19_114341_NB-353-Ch2-1.txt	/	2025-11-19_114341_NB-353-Ch2-1.png	/	Degassing, followed by calibration of H2 sensor.
	15:15	NB-353-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-19_151536_NB-353-Ch2-2.txt	/	2025-11-19_151536_NB-353-Ch2-2.png	/	Degassing of the photocatalyst suspension.
	16:00	NB-353-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-19_160001_NB-353-Ch2-3.txt	NB-353-O2 and H2 curve.py	2025-11-19_160001_NB-353-Ch2-3.png NB-353-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 of the suspension.

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-320: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C (reproduction 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 (reproduction 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-345: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm2, 20 °C (reproduction NB-325) I I

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)

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-353-O2 and H2 curve.py
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NB-353-O2 and H2 curves.png
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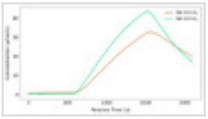

NB-353.ulog
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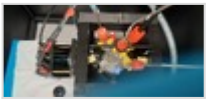

20251119_164458-after irradiation.jpg
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20251119_161145-start of irradiation.jpg
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20251119_151820-degassing of the suspension.jpg
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20251119_145537-suspension after vortex.jpg
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20251119_124232-H2 table.jpg
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20251119_122724-H2 bubbling.jpg
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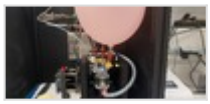

NB-353-Logger3-during irradiation.bmp
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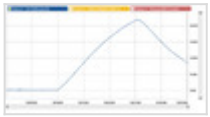

NB-353-Logger3-during irradiation.csv
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NB-353-Logger2-2point calibration.bmp
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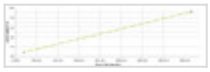

NB-353-Logger2-calibration step.bmp
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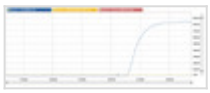

NB-353-Logger2-calibration.csv
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NB-353-Logger1-pre-polarization.bmp
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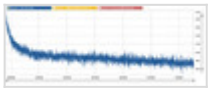

NB-353-Logger1-pre-polarization.csv

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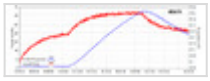

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