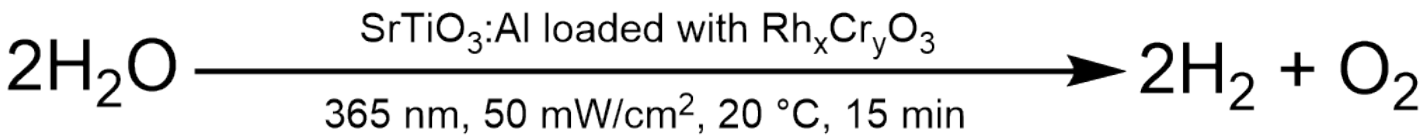
NB-316: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C

Date: 2025-10-20
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

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	/
Similar experiments	SrTiO3 - NB-313: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-301, 0.5 mg/mL), 365 nm, 50 mW 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]
milli-Q H_2O	/	/	/	/	/	/	/	0.998	25.00

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	12.86	/	/	/

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. 605 s 2. 21:15:23
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 1524 s 2. 21:30:42

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
20.10.2025		Calibration from 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 was used		
		Sample preparation		
	19:05	Weighing **EA-358** photocatalyst in a 50 mL vial.	Creamy solid	20251020_190334-weighed solid.jpg
	ca. 19:08	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	19:10-13	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251020_191321-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	19:30	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/

	19:35	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	20251020_193516-before degassing and irradiation.jpg
	19:39	Start of O2 logging.	**NB-316-Ch2-1**	2025-10-20_193930_NB-316-Ch2-1.txt 2025-10-20_193930_NB-316-Ch2-1.png
	19:48	The degassing was started	/	/
	ca. 20:20	Cannula was transferred to gas phase, above the suspension.	/	/
	20:21	H_2 sensor was added in Ar counterflow.	/	20251020_202318-introducing H2 sensor.jpg
	20:26	The degassing was stopped by removing the cannula and closing the valve.	/	/
	20:29	Stop of O2 logging.	/	/
	20:30	Start of H2 logging.	**NB-316-Logger1**	NB-316.ulog NB-316-Logger1.bmp
	20:31	Start of O2 logging.	**NB-316-Ch2-2**	2025-10-20_203104_NB-316-Ch2-2.txt 2025-10-20_203104_NB-316-Ch2-2.png
	20:35	Stop O2 logging.	Too high leakage rate - 20:36 - 4.58 uM O_2	/
	20:42	Stop H2 logging.	Problem that caused leakage - BOLA fitting for PT100 was not tight, Deassembling the setup, eliminating problem --> solved	/
	20:46	Restart of degassing.	For degassing procedure H_2 sensor was removed from the reactor, it was replaced with NS14 stopper	/
	20:59	Introducing H2 sensor under Ar flow (cannula was in gas phase above the liquid).	/	/
	ca. 21:03	Removing cannula, closing the valve.	/	/
	21:05	Start of O2 logging.	**NB-316-Ch2-3**	2025-10-20_210518_NB-316-Ch2-3.txt 2025-10-20_210518_NB-316-Ch2-3.png
	21:05	Start of H2 logging.	**NB-316-Logger2**	NB-316.ulog NB-316-Logger2-during irradiation.csv NB-316-Logger2-during irradiation.bmp

	21:05-15	Equilibration time.	/	/
	21:15	The irradiation was started	/	20251020_211617-after start of irradiation.jpg
	21:30	The irradiation was stopped.	/	/
	21:30-41	Equilibration time.	/	/
	21:41	Stop of O2 and H2 logging.	/	/
	ca. 21:45	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).	20251020_214330-after irradiation.jpg 20251020_214344-after irradiation-closer view.jpg

Analysis

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

Used calibration for UniSense: NB-315-Logger3

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
20.10.2025	20:30	NB-316-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-316.ulog	/	NB-316-Logger1.bmp	/	High O2 leakage rate was found - logging was stopped, needs to be redone.
	21:05	NB-316-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-316.ulog NB-316-Logger2-during irradiation.csv	NB-316-O2 and H2.py	NB-316-Logger2-during irradiation.bmp NB-316-H2 and O2 curves.png	/	Clean H2 response, H2 value ca. 52 uM at the end of irradiation.
	19:39	NB-316-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-10-20_193930_NB-316-Ch2-1.txt	/	2025-10-20_193930_NB-316-Ch2-1.png	/	Degassing of the suspension.

	20:31	NB-316-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-10-20_203104_NB-316-Ch2-2.txt	/	2025-10-20_203104_NB-316-Ch2-2.png	/	High O2 leakage rate.
	21:05	NB-316-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-10-20_210518_NB-316-Ch2-3.txt	NB-316-O2 and H2.py	2025-10-20_210518_NB-316-Ch2-3.png NB-316-H2 and O2 curves.png	/	Change in O2 value during irradiation, O2 value ca. 40 uM at the end of 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, 20 °C**,** 15 min, reference conditions) were performed.

H_2 level at the end of irradiation - 52 umol/L, O_2 level - 40 umol/L.

Future recommendations

Old procedure	Problem	Suggested new procedure
/	High O2 leakage rate after degassing.	Check BOLA fitting to each sensor before experiment, should fit tightly.

Linked experiments

SrTiO3 - NB-313: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-301, 0.5 mg/mL), 365 nm, 50 mW

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

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-316-O2 and H2.py
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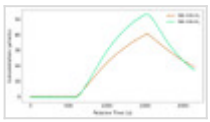
NB-316-H2 and O2 curves.png
sha256: c5925f567c35b5673158cdfe50114a41f6e58254e1472bc684d484cb89a13bfe



Unisense-NB-316-Screenshot 2025-11-07 083140.png
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NB-316-SrTiO3-photocatalytic H2O splitting.png
sha256: f9541119d3b1697940988dad6b96e3acc271559fdf00a3ad36913aa8e3309d61

NB-316-SrTiO3-photocatalytic H2O splitting.cdxml
sha256: 492252c43cd5886d865bfe0134680d27dce9e3bbfbf36d2890b07a732289313c

NB-316-Logger2-during irradiation.csv
sha256: 584b70d220a548571176fcc50ec042c82db9c41b01271091b8b6a3a4e73dc52f

NB-316-Logger2-during irradiation.bmp
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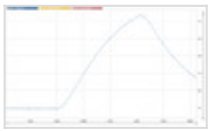

20251020_214344-after irradiation-closer view.jpg
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20251020_214330-after irradiation.jpg
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20251020_202318-introducing H2 sensor.jpg
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20251020_211617-after start of irradiation.jpg
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20251020_193516-before degassing and irradiation.jpg
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20251020_190334-weighed solid.jpg
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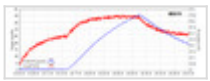

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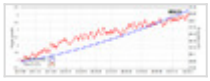

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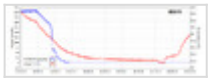

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NB-316.ulog
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