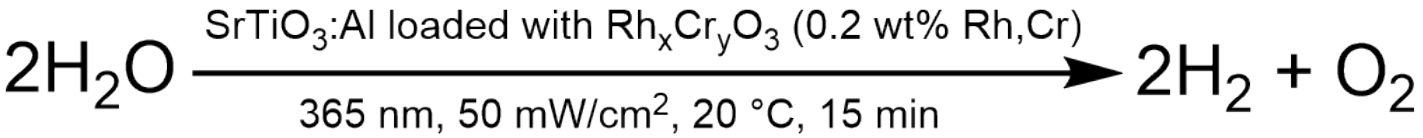
NB-333: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-323, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C (reproduction NB-328)

Date: 2025-11-03
Tags: O2 | Test | Calibration | Future | 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-328: simultaneous detection of H_2 and O_2 evolution in liquid phase for irradiated suspension of Rh,CrO_x:Al:$SrTiO_3$ suspension (NB-323, **0.2 wt% Rh,Cr**, 0.5 mg/mL), 365 nm LED, 50 mW/cm^2, 20 °C.

Reaction scheme



ChemDraw file linked: NB-328-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	/
Reproduction	SrTiO3 - NB-328: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-323, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C
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 SrTiO3 - NB-327: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-321, 0.05 wt% Rh, Cr, 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]	Concentration [mM]
milli-Q H_2O	/	/	/	/	/	/	/	0.998	25	/
Al:SrTiO3 RhCrOx (NB-323)	SrTiO3 - NB-323: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.2 wt%)	/	/	/	12.50	12.68	/	/	/	/

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. 1:37:41
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 1525 s 2. 1:53:01

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
		Calibration from 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 was used.	/	/
04.11.2025		**Sample preparation**		
	ca. 0:10	Weighing photocatalyst in a 50 mL vial (the vial was covered with Al foil before further use) (preliminary, the lumps of the solid were broken with Smartspatula inside the vial).	Slightly grey solid	/
	0:20	Addition of 25 mL H_2O to the vial via graduated cylinder.	/	/
	0:22-25	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	Dispersion seems to look better than in the previous experiment SrTiO3 - NB-328: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-323, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C	20251104_002643-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	0:30-35	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/

	ca. 0:37	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	20251104_003621-before degassing and irradiation.jpg
	0:39	Start of O2 logging.	**NB-333-Ch2-1**	2025-11-04_003927_NB-333-Ch2-1.txt 2025-11-04_003927_NB-333-Ch2-1.png
	0:41	The degassing was started	/	
	1:19	Cannula was transferred to gas phase, above the suspension.	/	
	1:21	H_2 sensor was added in Ar counterflow.	/	
	1:25	The degassing was stopped by removing the cannula and closing the valve.	/	
	1:27	Stop of O2 logging.	/	
	1:27	Start of O2 logging.	**NB-333-Ch2-2**	2025-11-04_012735_NB-333-Ch2-2.txt 2025-11-04_012735_NB-333-Ch2-2.png
	1:27	Start of H2 logging.	**NB-333-Logger1**	NB-333.ulog NB-333-Logger1.csv NB-333-Logger1.bmp
	1:27-37	Equilibration time.	/	/
	1:37	The irradiation was started	/	20251104_013911-after start of irradiation.jpg
	1:53	The irradiation was stopped.	/	/
	1:53-2:03	Equilibration time.	/	/
	2:03	Stop of O2 and H2 logging.	/	/

	2:10	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).	20251104_021611-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-331-Logger3

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
04.11.2025	1:27	NB-333-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-333.ulog NB-333-Logger1.csv	NB-333-O2 and H2 curve.py	NB-333-Logger1.bmp NB-333-O2 and H2 curve.png	SrTiO3 - NB-328: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-323, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C	Clean H2 response signal, H2 value ca. 29 uM
	0:39	NB-333-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-04_003927_NB-333-Ch2-1.txt	/	2025-11-04_003927_NB-333-Ch2-1.png	/	Degassing of the suspension.
	1:27	NB-333-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-04_012735_NB-333-Ch2-2.txt	NB-333-O2 and H2 curve.py	2025-11-04_012735_NB-333-Ch2-2.png NB-333-O2 and H2 curve.png	SrTiO3 - NB-328: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-323, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C	Sharp increase/decrease in O2 value after start and stop of irradiation. O2 value ca. 29 uM.

Results

Reproduction of NB-328, simultaneous H_2 and O_2 measurements of irradiated suspension of NB-323 (0.5 mg/mL) in O_2/H_2 photoreactor under 365 nm irradiation (50 mW/cm^2, 20 °C**,** 15 min) were performed.

H_2 level at the end of irradiation - 29 umol/L, O_2 level - 29 umol/L (but sharp change in signal after start and finish of irradaition in O2 curve - needs to be eliminated).

Future recommendations

Old procedure	Problem	Suggested new procedure
/	Sharp increases in O2 signal - maybe related either with the position of the sensors in the reactor.	Adjust the position of the sensors relatively to the light beam.

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-323: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.2 wt%)

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-326: 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)

SrTiO3 - NB-327: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-321, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C

SrTiO3 - NB-328: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-323, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C

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

Linked resources

Equipment - VWR® VV3, Vortex Mixer

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

Equipment - Irradiation setup 4 (CEEC II, E002)

Protocol - Hydrogen Measurement Using GC

Protocol - Getting hydrogen from hydrogen bottle in CEEC II E014

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

Attached files

NB-333-O2 and H2 curve.py
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NB-333-O2 and H2 curve.png
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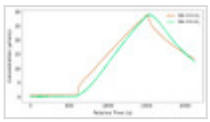

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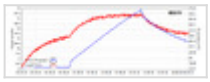

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NB-333-Logger1.csv
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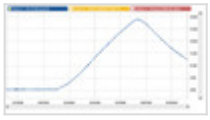

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Link: https://elab.water-splitting.org/experiments.php?mode=view&id=3325