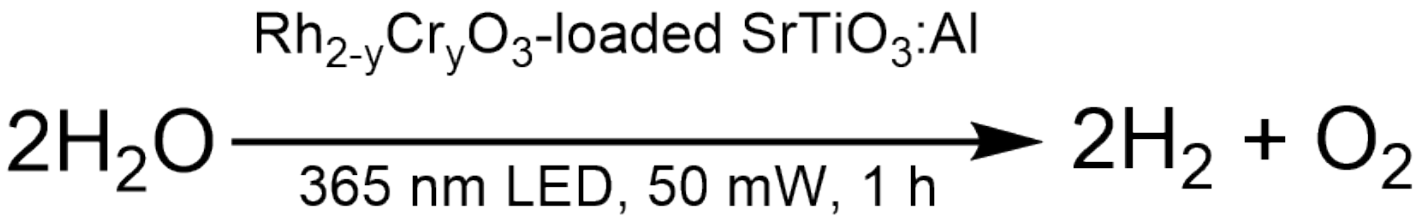
NB-312: Gas phase H2 and O2 measurements with Unisense H2 sensor, Firesting O2 robust probe in irradiated Al:SrTiO3 RhCrOx (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing

Date: 2025-10-08
Tags: O2 | Test | Calibration | NB | Firesting | Irradiation | O2 sensor | H2 | advanced irrad setup | Unisense | H2 Sensor | temperature | In situ | Trace range robust oxygen sensor | photocatalysis
Category: SrTiO3
Status: Done
Created by: Nadzeya Brezhneva

Objectives

Simultaneous gas phase H2 and O2 measurements in irradiated Al:SrTiO3 RhCrOx (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing.

Reaction scheme



ChemDraw file linked: NB-304-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	https://doi.org/10.1039/C9EE00310J
Reproduction	SrTiO3 - NB-304: Gas phase H2 and O2 measurements with Unisense H2 sensor, Firesting O2 robust probe in irradiated Al:SrTiO3 RhCrOx (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing
Similar experiments	SrTiO3 - NB-231: Gas phase H2 and O2 measurements with Unisense H2 sensor, Firesting O2 cap in irradiated Al:SrTiO3 RhCrOx (NB-162-4, 1 mg/mL), 365 nm, 50 mW, 1 h, degassing

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]
milli-Q H_2O	/	/	/	/	/	/	/	/	25	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	1 ballon (approx. 2 L)	approx. 1

Al:SrTiO3 RhCrOx (**NB-289**)	SrTiO3 - NB-289: Modification of NB-285 (SrTiO3:Al (from self-made SrTiO3, Osterloh, no Al2O3, PVDF filter) 1000 C, 10 h) with Rh, Cr oxide cocatalyst	/	/	/	12.50	12.62	/	/	/	/

Irradiation Parameters

Power measurement was performed in experiment Prep work - NB-310: Measuring power output of UHP-365 nm #1 with 18A-2 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	**Used power source**	**Wavelength [nm]**	**Power Setting [mW]**	**Analogue settings [-]**
First light source	Light Source - UHP LED 365 nm-1	Power Sources - BLS-18000-1 2	365	50	0.32

Used beam combiner [Name or None]	None
Irradiation distance [cm]	6.5
Thermostat temperature [°C]	25
Stirring speed [1/min]	500
Start time [s], relative to start of log	930 s - Firesting 21:49:41 (not relative time) - Unisense

End time [s], relative to start of log	4650 s - Firesting 22:51:41 - Unisense

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 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
09.10.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor Important steps and deviations are listed below	/	/
		The PT100 was not placed inside the reactor, but outside the reactor insde the advanced irradiaiton setup, close to the double walled beaker.	/	/
	15:44-16:43	Conditioning of H2 sensor	**NB-312-Logger-1** 1 s interval, 1.39 mV at the end of pre-polarization	NB-312-pre-measurements.bmp
	ca. 17:50	Assembling the set-up for calibration.	/	/
	ca. 18:00	Addition of 25 mL milli-Q H_2O to the photoreactor via funnel.	/	/
	ca. 18:10	Assembling sensors with appropriate BOLA fitting and GL14/NS14 adapters.	PT1000 - 4 mm BOLA fitting Robust oxygen probe - 3 mm BOLA fitting Unisense sensor - 2 mm fitting (used from the previous measurement, only removed from the protective holder)	/

		Calibration was done first according to Protocol - Liquid phase calibration of H2 UniAmp sensor with H2 bubbling	/	/
	18:19	The O2 log was started	**NB-312-Ch2-1**	2025-10-09_181947_NB-312-Ch2-1.txt 2025-10-09_181947_NB-312-Ch2-1.png
	18:19	The H2 log was started	**NB-312-Logger-2** offset -1 mV	NB-312-calibration.bmp NB-312-calibration-1.bmp
	18:22	0 ppm point taken	0.51 mV	/
	18:23	Degassing was started by immersing cannula (from Ar supply setup) in water.	/	20251009_182518-calibration-during degassing.jpg
	18:41	Removing cannula from H_2O closing the valve.	/	/
	18:42	H2 bubbling of the reactor was started.	/	/
	18:50	1.000.000 ppm point was taken and calibration was saved	22.76 °C 1004.5 mbar 1.000.000 ppm 97674.034 Pa Signal of the sensor 1017 mV Slope: 0.010, intercept: 0.491	/
	18:51	The H2 logging was stopped.	/	/
	18:51	The O2 logging was stopped.	/	/
	18:55-20:00	Deassembling the setup. degreasing and drying the reactor at 120 °C.	/	/
		Sample preparation		
	20:10	Weighing 12.57 mg of **NB-289** sample in a 50 mL vial.	Creamy solid.	20251009_201235-weighed photocatalyst.jpg
	20:17	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/

	20:19-22	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	Milky white suspension.	20251009_202215-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6	/	/
	ca. 20:40-50	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	20251009_205632-before irradiation.jpg 20251009_205642-positions of the sensors inside the reactor.jpg
	21:00	The O2 log was started	**NB-312-Ch2-2**	2025-10-09_210005_NB-312-Ch2-2.txt 2025-10-09_210005_NB-312-Ch2-2.png
	21:11	The degassing was started	/	/
	21:31	The degassing was stopped	No leakage	/
	21:33	The O2 log was stopped	Adjusting stirring, there were some problems with stirring of the suspension, but were eliminated.	/
	21:34	The O2 log was started	**NB-312-Ch2-3**	2025-10-09_213411_NB-312-Ch2-3.txt 2025-10-09_213411_NB-312-Ch2-3.png
	21:34	The H2 log was started	**NB-312-Logger-3**	NB-312-during irradiation-H2 evolution.bmp
	21:35-49	Equilibration time.	21:35-38 - making sure that the stirring was good	/
	21:49	The irradiation was started	0 h : 15 min : 30 s - Firesting 21:49:41 - Unisense (there is no lid on the setup, covered only by the side wall)	20251009_215035-after start of irradiation.jpg
	22:51	The irradiation was stopped	1 h : 17 min : 30 s - Firesting 22:51:41 - Unisense 0.75 vol.% O_2 at the end of irradiation 1558 Pa H_2 at the end of irradiation	/

	23:06	The H2 on O2 log were stopped	/	/
	23:10	Deassembling the setup, cleaning the reactor.	Adhesion of photocatalyst to the walls of the reactor.	20251009_230913-after irradiation.jpg

Analysis

Used calibration for Firesting: Prep work - NB-280: Gas phase calibration of O2 robust oxygen sensor (Firesting) in a 4 neck H2/O2 photoreactor

Used calibration for Unisense: NB-312-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
09.10.2025	15:44	NB-312-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor	H2O	NB-313-Logger.ulog NB-312-Logger1-Data (1 H2 UniAmp (503157)).csv	/	NB-312-pre-measurements.bmp	/	Initial slight decrease in the signal, afterwards stabilizing (1.39 mV at the end of pre-polarization).
	18:19	NB-312-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor	H2O	NB-313-Logger.ulog NB-312-Logger2-Data (1 H2 UniAmp (503157)).csv	/	NB-312-calibration.bmp NB-312-calibration-1.bmp	SrTiO3 - NB-304: Gas phase H2 and O2 measurements with Unisense H2 sensor, Firesting O2 robust probe in irradiated Al:SrTiO3 RhCrOx (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing	two point calibration (0 and 1.000.000 ppm), Slope = 0.010, intercept = 0.451, R^2 = 1
	21:34	NB-312-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor	H2O	NB-313-Logger.ulog NB-312-Logger3-Data (1 H2 UniAmp (503157)).csv	NB-312-with O2 and H2 axes.py NB-312-with O2, H2 and T axes.py	NB-312-during irradiation-H2 evolution.bmp NB-312-H2 and O2.jpeg NB-312-H2, O2 and T.jpeg	/	Gradual increase in H2 value (Up to ca. 1558 Pa at the end of irradiation).
	18:19	NB-312-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 2)	H2O	2025-10-09_181947_NB-312-Ch2-1.txt	/	2025-10-09_181947_NB-312-Ch2-1.png	/	Degassing (during calibration) worked, till 0.00 vol.%
	21:00	NB-312-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 2)	H2O	2025-10-09_210005_NB-312-Ch2-2.txt	/	2025-10-09_210005_NB-312-Ch2-2.png	/	Degassing worked, no leakage after finishing degassing step in the configuration of the assembled setup for irradiation.
	21:34	NB-312-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 2)	H2O	2025-10-09_213411_NB-312-Ch2-3.txt	NB-312-with O2 and H2 axes.py NB-312-with O2, H2 and T axes.py	2025-10-09_213411_NB-312-Ch2-3.png NB-312-H2 and O2.jpeg NB-312-H2, O2 and T.jpeg	/	Increase in O2 value during irradiation (up to 0.75 vol. % after 1 h irradiation).

Results

Gas phase measurements of O_2 and H_2 were performed during photocatalytic test of Al:$SrTiO_3$ suspension (reproduction of NB-304, sample **NB-289**, 0.5 mg/L, 365 nm, 50 mW, 1h) under degassed conditions were performed. Increase in O_2 and H_2 values during irradiation was observed (up to 0.75 vol.% O_2 and 1558 Pa

H_2 at the end of irradiation.

for comparison: SrTiO3 - NB-304: Gas phase H2 and O2 measurements with Unisense H2 sensor, Firesting O2 robust probe in irradiated Al:SrTiO3 RhCrOx (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing

O_2: 0.71 vol.% at the end of irradiation,

H_2: 1370 Pa at the end of irradiation.

Linked experiments

SrTiO3 - NB-289: Modification of NB-285 (SrTiO3:Al (from self-made SrTiO3, Osterloh, no Al2O3, PVDF filter) 1000 C, 10 h) with Rh, Cr oxide cocatalyst

SrTiO3 - NB-304: Gas phase H2 and O2 measurements with Unisense H2 sensor, Firesting O2 robust probe in irradiated Al:SrTiO3 RhCrOx (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing

Linked resources

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

Equipment - Robust probe for liquid O2 measurment

Equipment - Advanced irradiation chamber V1.0 I

Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle

Equipment - Irradiation setup 4 (CEEC II, E002)

Protocol - Operation of Firesting Fiber-Optic Oxygen Meter 2 Channel Software

Protocol - Getting hydrogen from hydrogen bottle in CEEC II E014

Protocol - Gas phase calibration of H2 UniAmp sensor

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

Attached files

NB-312-H2, O2 and T.jpeg
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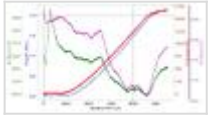

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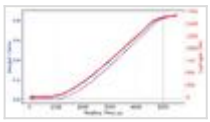

NB-312-with O2 and H2 axes.py
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NB-312-with O2, H2 and T axes.py
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20251009_205632-before irradiation.jpg
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20251009_211211-degassing of the suspension.jpg
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20251009_205642-positions of the sensors inside the reactor.jpg
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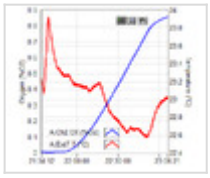

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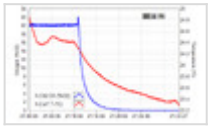

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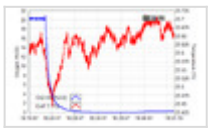

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NB-312-Logger2-Data (1 H2 UniAmp (503157)).csv
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NB-312-Logger1-Data (1 H2 UniAmp (503157)).csv
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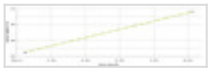

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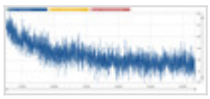

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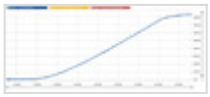

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