Free Hydro Cells

Funded by the European Union

Title **Enhanced photoelectrochemical water splitting with doped transition metal dichalcogenide nanofilms**

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Event/Date MRS Meeting, Boston, USA, 26 November - 1 December 2023

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Talk Outline

FreeHydroCells

- Introduction
- Materials' growth and formation
- Structural and physical characterisation
- Hall-effect analysis
- Photoelectric and Photoelectrochemical characterisation
- Conclusions, Future Work and Acknowledgements

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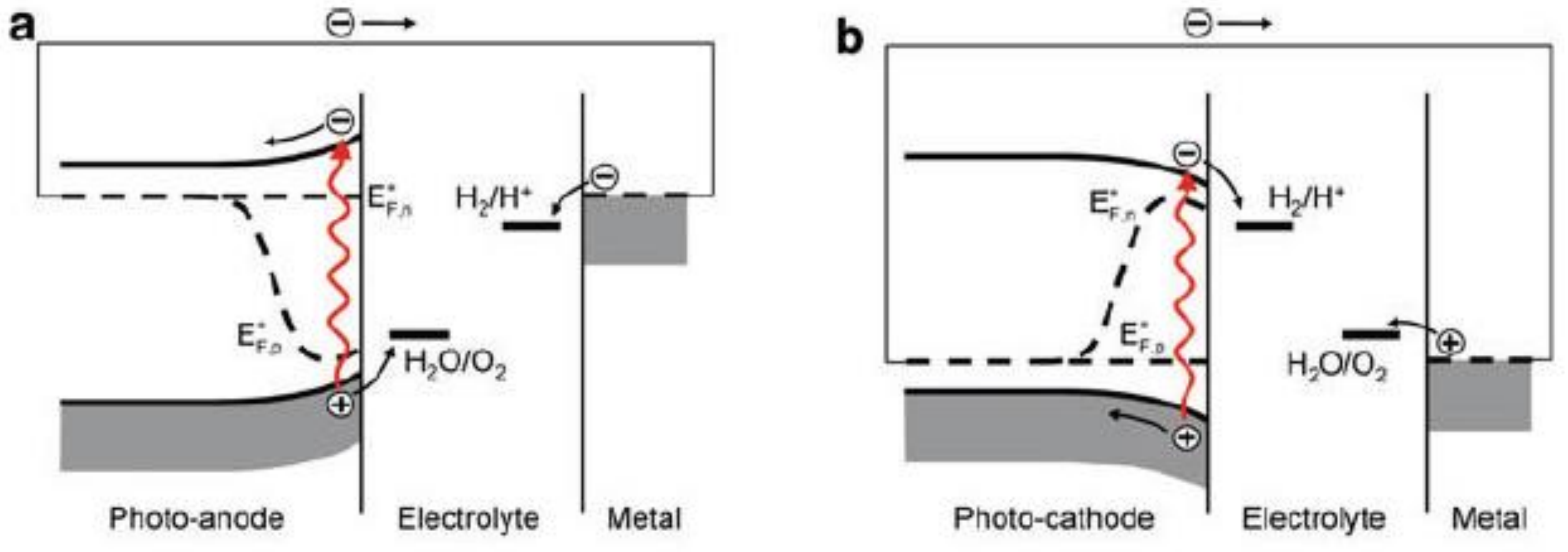
Photoelectrochemical Water Splitting

FreeHydroCells

Single Layer Photoelectrochemical devices



Standard single semiconductor devices based on a (a) Photo-anode or (b) Photo-cathode.

R. van de Krol , Photoelectrochemical Hydrogen Production (2012)

- Our work focuses on creation and characterising of novel and earth abundant electrodes for photo-anode [n-type] and photo-cathode [p-type] semiconductors to produce hydrogen.

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Introduction to TMDs

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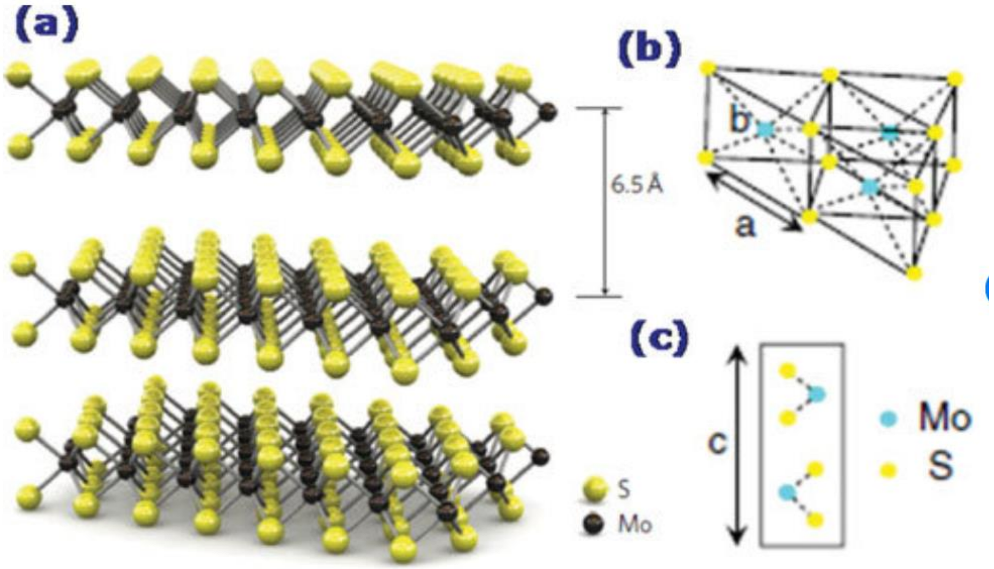
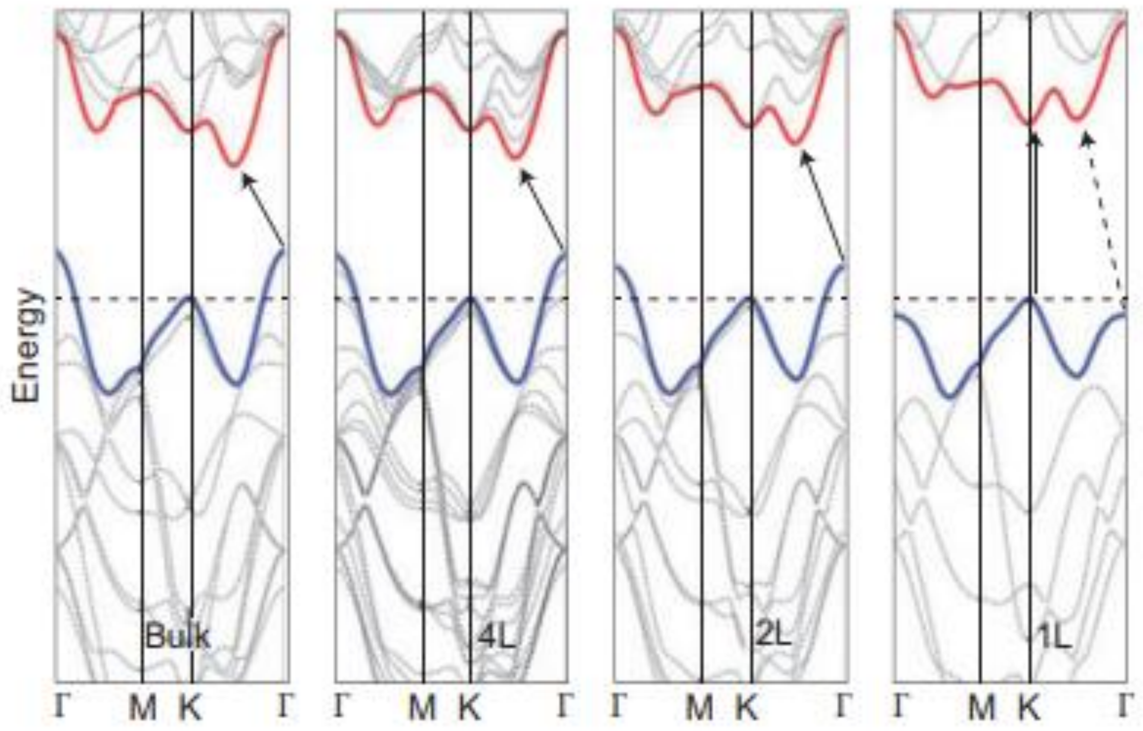


MX_2 (M=Mo, W X=S, Se)
- Earth abundant composition
- 1.1-1.6 eV bulk E_g
- High absorption coefficient
- Structure stability under illumination

Chhowalla *et al*,. Nature Chemistry vol.5, p.263 (2013)



Energy dispersion in bulk MoS_2

Splendiani *et al*,. Nano Lett vol.10, p.1271 (2010)

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Transition Metal Dichalcogenide Film Growth

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Magnetron Sputtering [Co-sputtering]

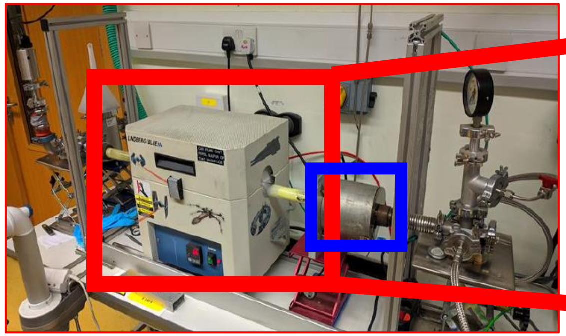
Powers *et al*,. Journal of Materials Research vol 35, p.1531 (2020)

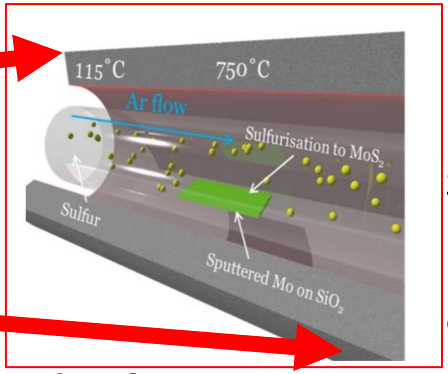
1

2

3

Thermally Assisted Conversion (TAC)





Sulphur Furnace

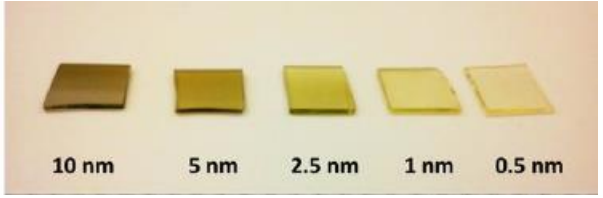
Growth Furnace

Two zone quartz tube furnace

Gatensby *et al*,. Applied Surface Science vol.297, p.139 (2014)

PVD system for TiO_2 capping layer deposition

Wang *et al*,. Chemical Engineering Journal vol.381, 122605 (2020)



Different thicknesses of films on quartz

Hallam *et al*,. Appl. Phys. Lett. 111, 203101 (2017)

This work includes doping of TMD

42 Mo Molybdenum 95.95 + 75 Re Rhenium 186.21

74 W Tungsten 183.84 + 41 Nb Niobium 92.906

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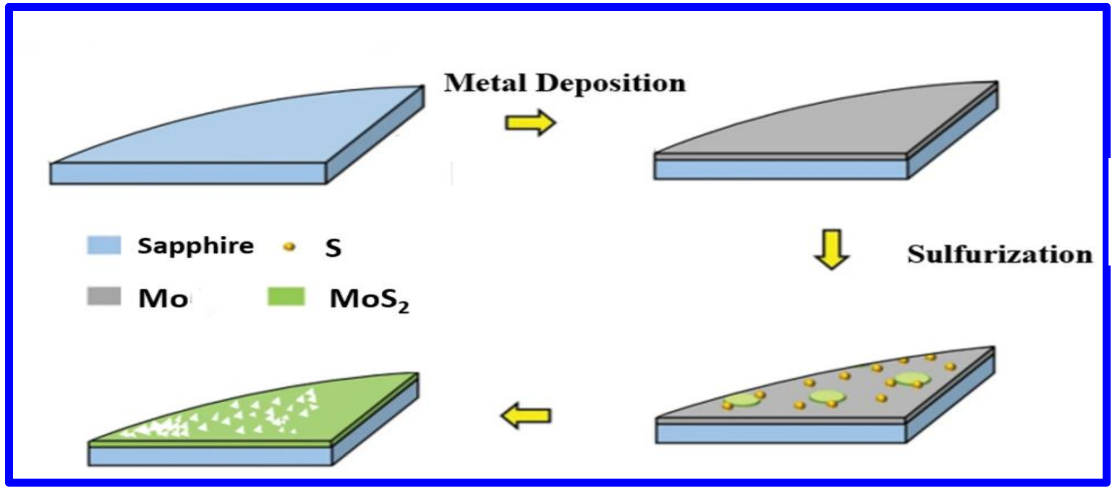
Structural and Physical Characterisation I

FreeHydroCells

- The methods used to assess TMDs in the presentation

1 X-Ray Reflectivity (Before and after Conversion)



Furnace Process [Conversion]

2 Raman Spectroscopy

3 X-Ray Diffraction

Structural and Physical Characterisation I

XRR : Thickness Analysis

The basic principle is that when X-rays strike a surface, they are partially reflected, and the amount of reflection depends on the angle of incidence and the properties of the material.

WS_2 Samples (p –type targeted)

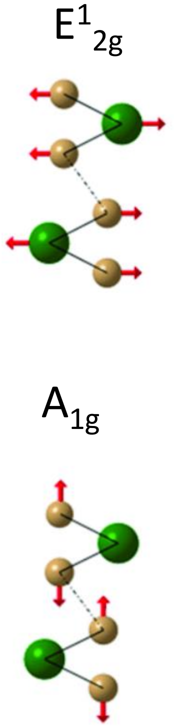
Samples	Before conversion	After conversion	TiO_2 Capping layer
0% Nb doped WS_2	**9-13 nm**	**21-25.5 nm**	**7 – 11 nm**
2% Nb doped WS_2	**9-13 nm**	**21-25.5 nm**	**7 – 11 nm**
4% Nb doped WS_2	**9-13 nm**	**21-25.5 nm**	**7 – 11 nm**

MoS_2 Samples (n-type targeted)

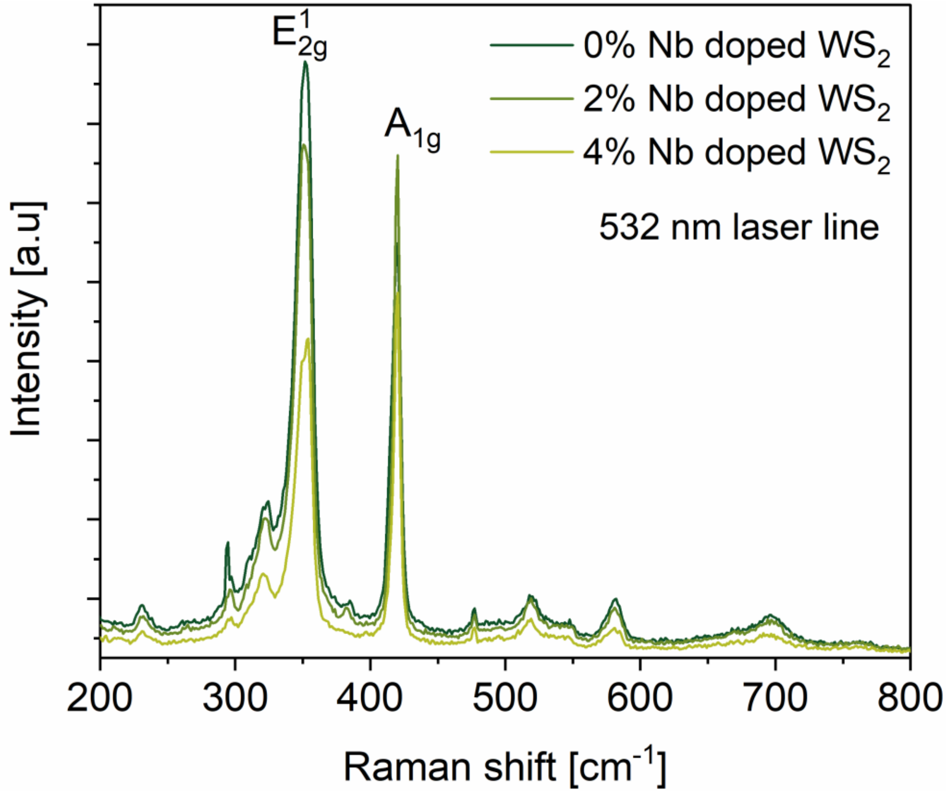
Samples	Before conversion	After conversion	TiO_2 Capping layer
0% Re doped MoS_2	**15-20 nm**	**44-48 nm**	**7 – 11 nm**
5% Re doped MoS_2	**15-20 nm**	**40-48 nm**	**7 – 11 nm**
9% Re doped MoS_2	15-20 nm	30-40 nm	**7 – 11 nm**
18% Re doped MoS_2	**13-17 nm**	**26-35 nm**	**7 – 11 nm**

Structural and Physical Characterisation II

Raman Spectroscopy



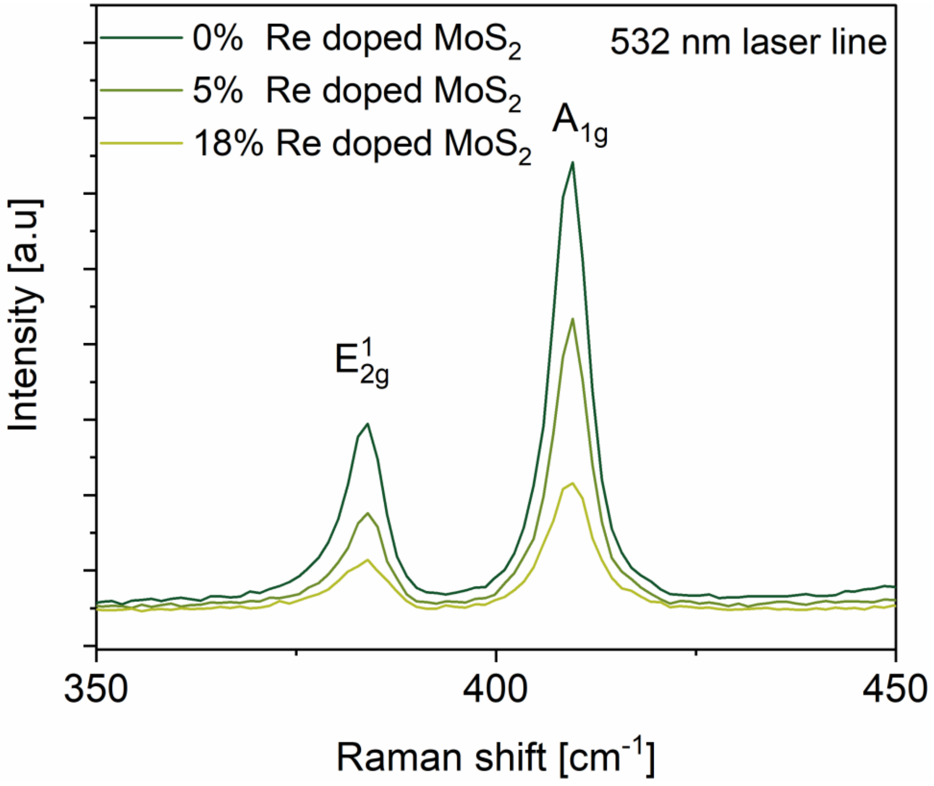
WS_2 Samples



Igbal *et al*,. Microelectronic Engineering. 219, 111152 (2020)

Yang *et al*,. Research. 9862483, 111152 (2021)

MoS_2 Samples

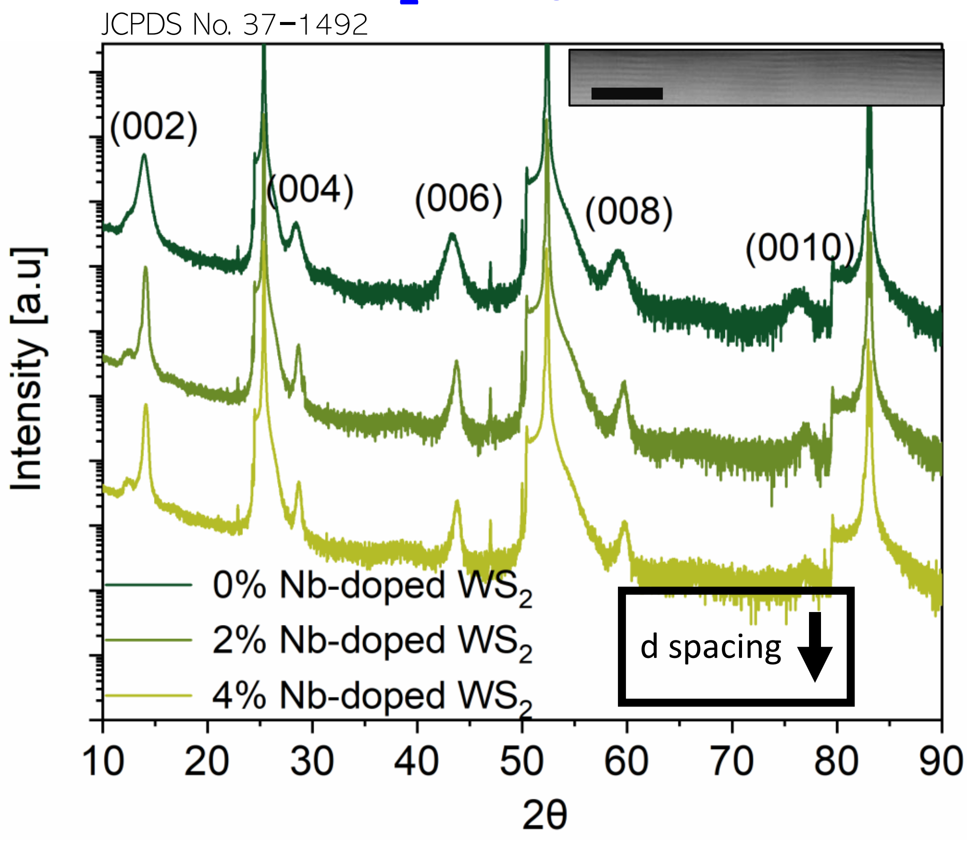


Hallam *et al*,. Appl. Phys. Lett. 111, 203101 (2017)

Structural and Physical Characterisation III

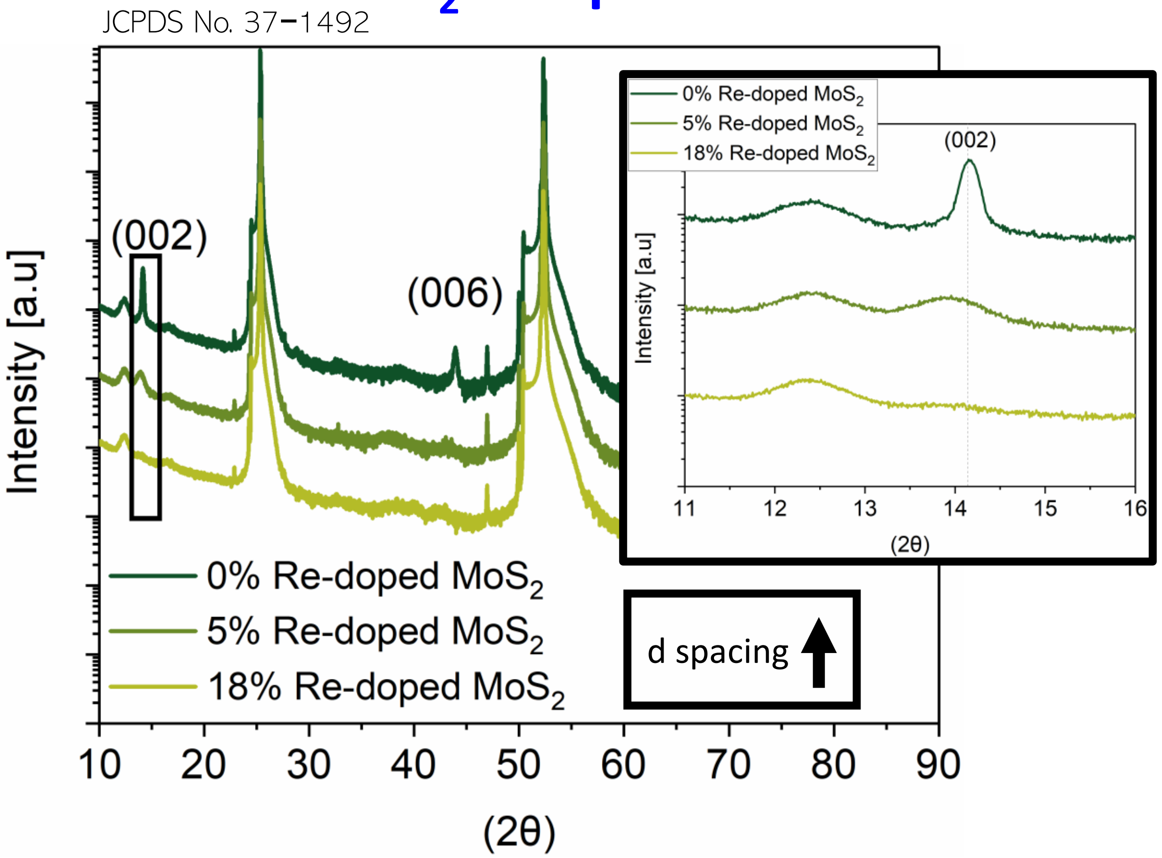
X-Ray Diffraction

WS_2 Samples



Pu *et al*,. Flat Chem. 29, 100278 (2021)

MoS_2 Samples



Qin *et al*,. Scientific Reports. 4, 7582 (2014)

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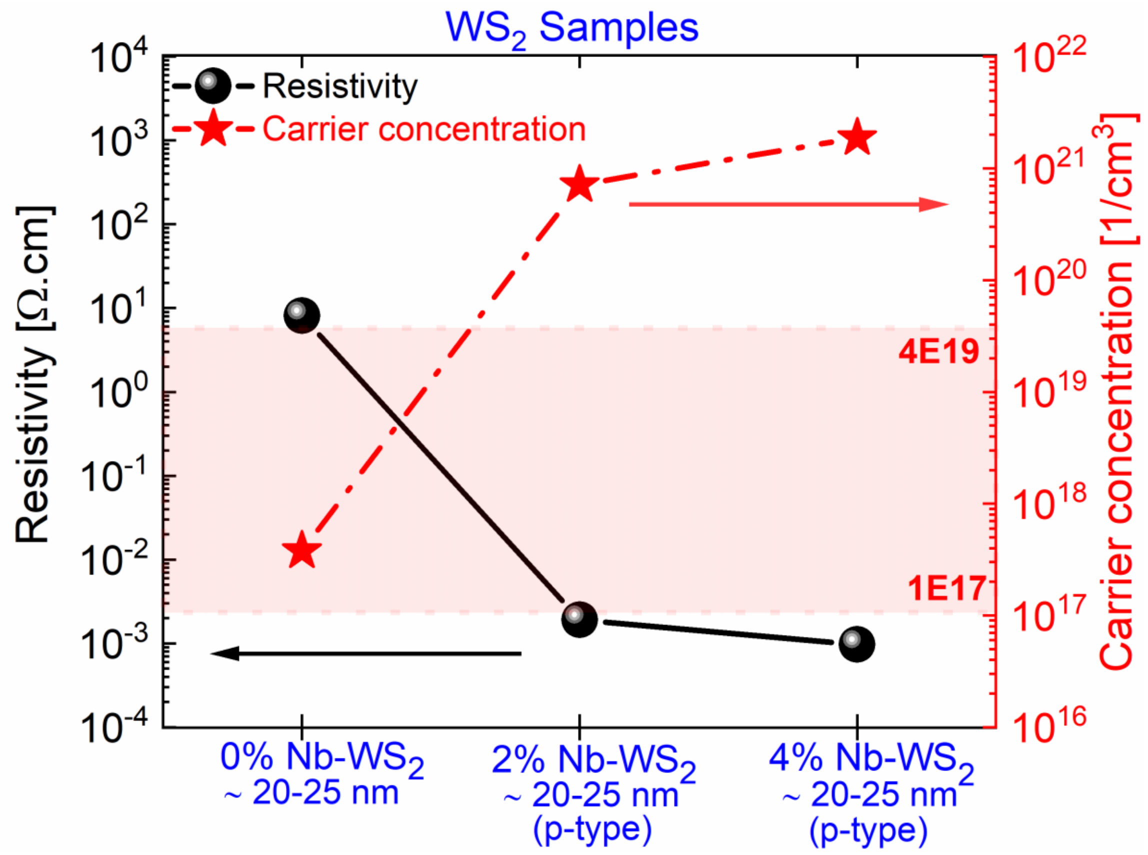
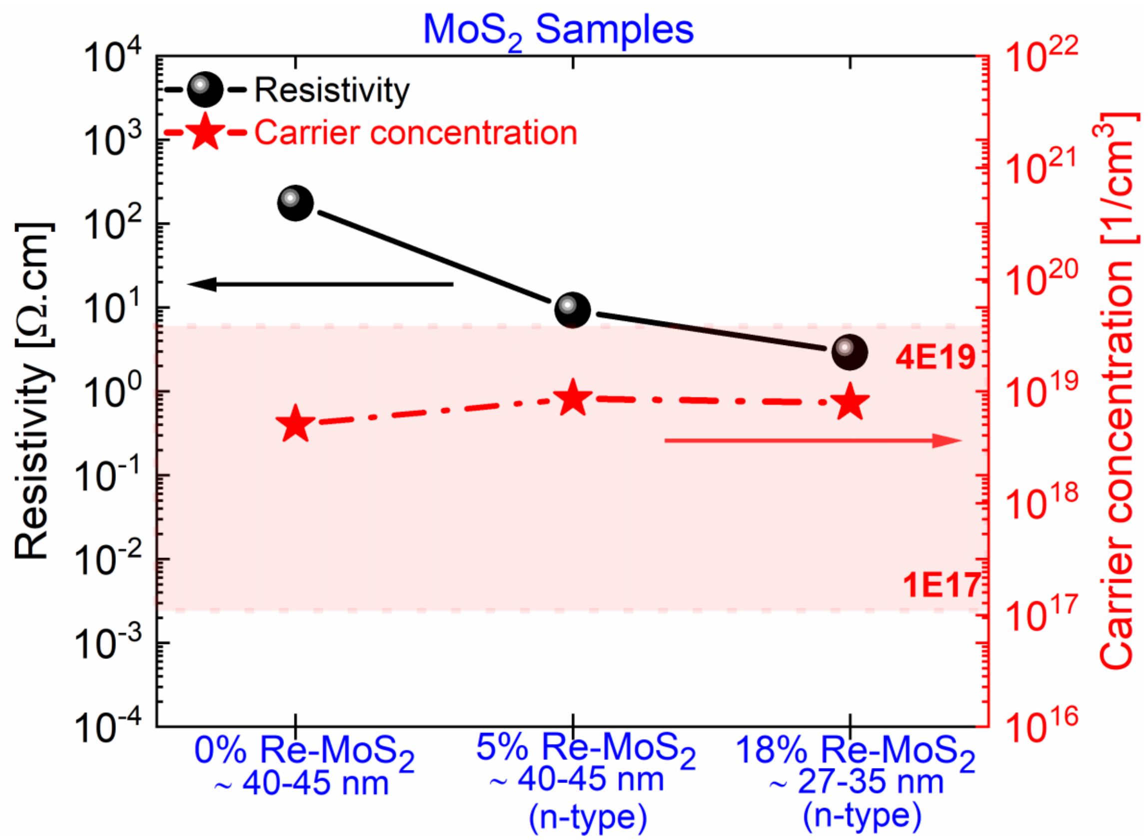
Hall Effect Analysis

Doping Study





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Photoelectric and photoelectrochemical analysis

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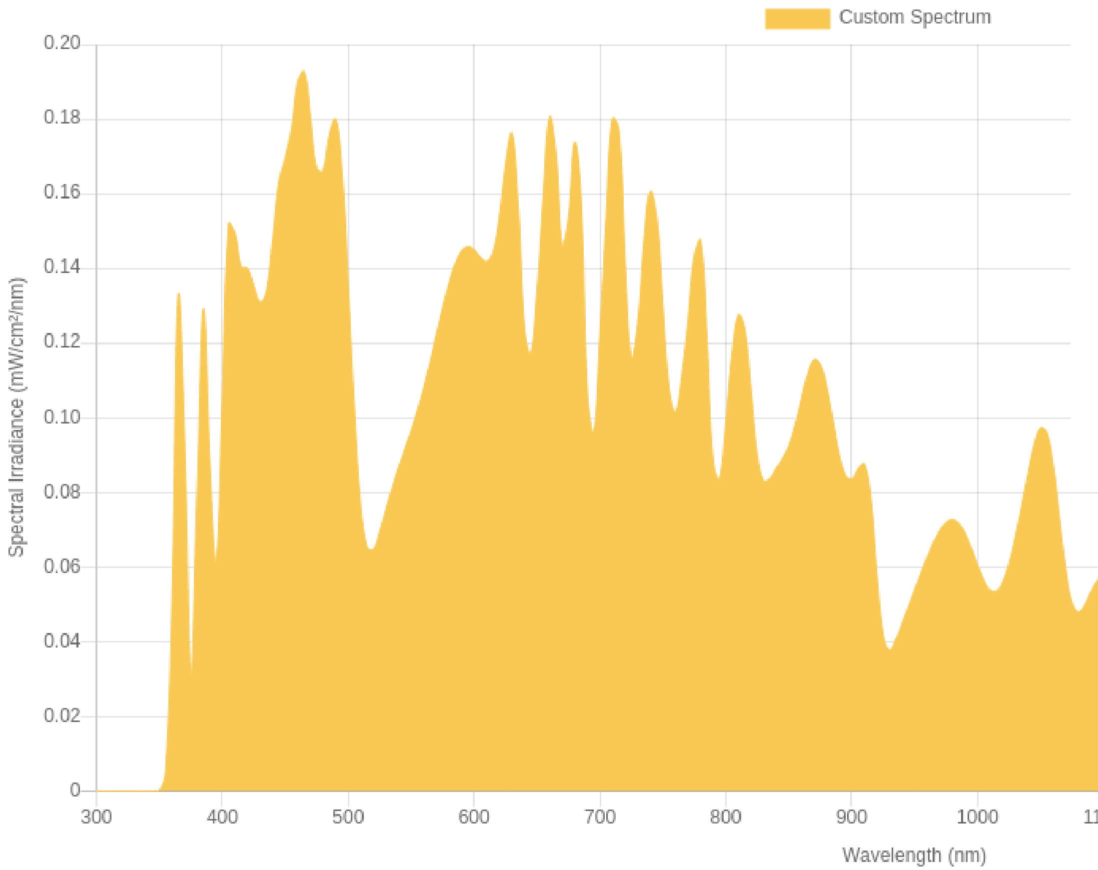
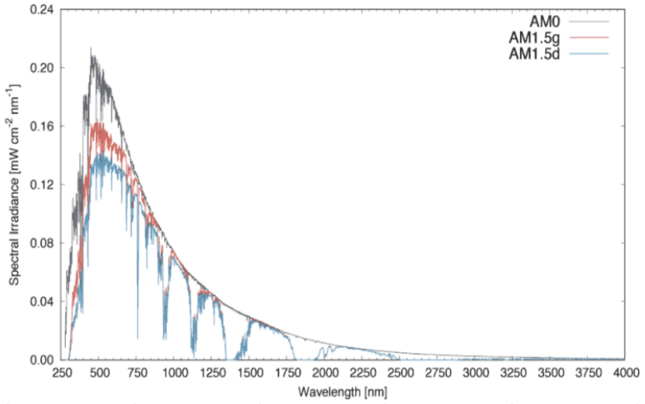


Standard spectrum of AM1.5G, 1.1 suns

www.nrel.gov/grid/solar-resource/spectra-am1.5



The spectrum of AM1.5G, 1.1 suns used in this work

www.g2voptics.com/solar-simulation

- Light on:

32 LEDs

Total radiance is 88.3mW/cm^2

Spectra: 361 - 1556 nm

AM1.5G, 1.1 Suns
(Effectively 0.9 x 1.1 Suns = 0.99 Suns)

- Light off:

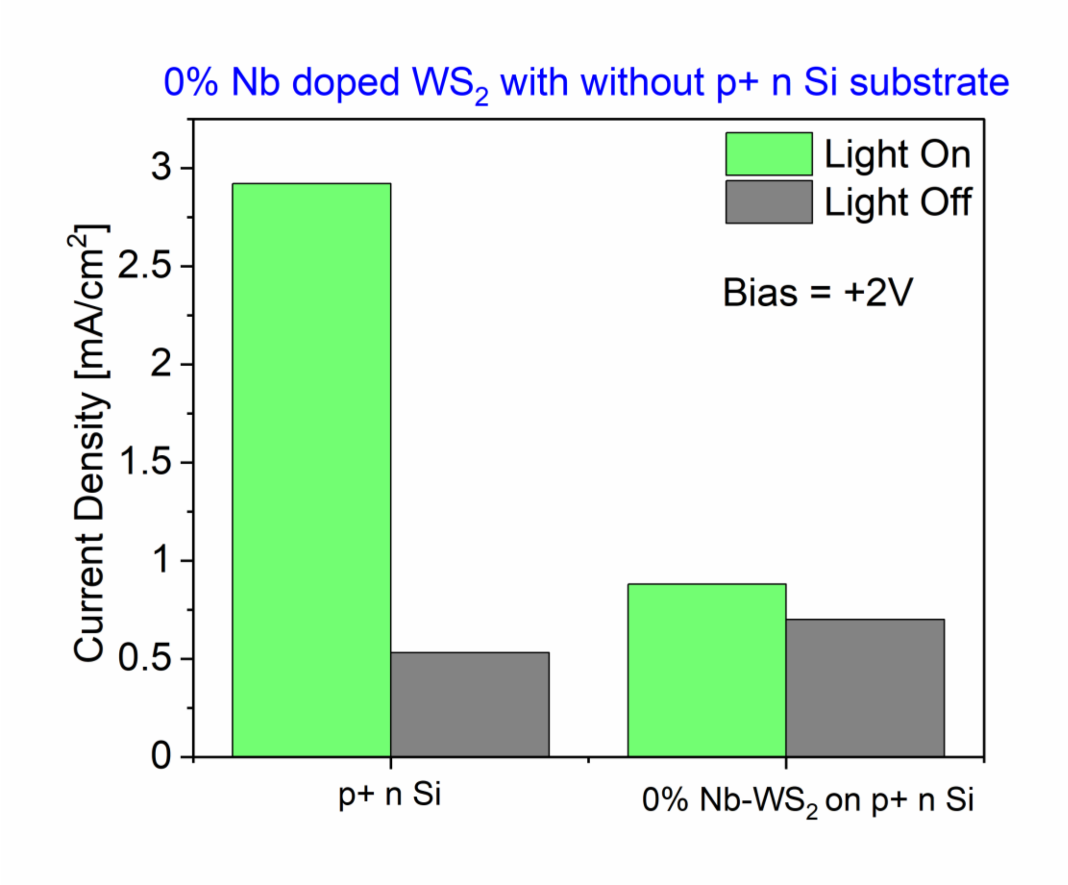
Dark chamber.

- Temperature in both case:

20.4^{o}C to 23.5^{o}C

Photo response assessment of TMD films on Si substrate

0% Nb-WS_2



18% Re-MoS_2

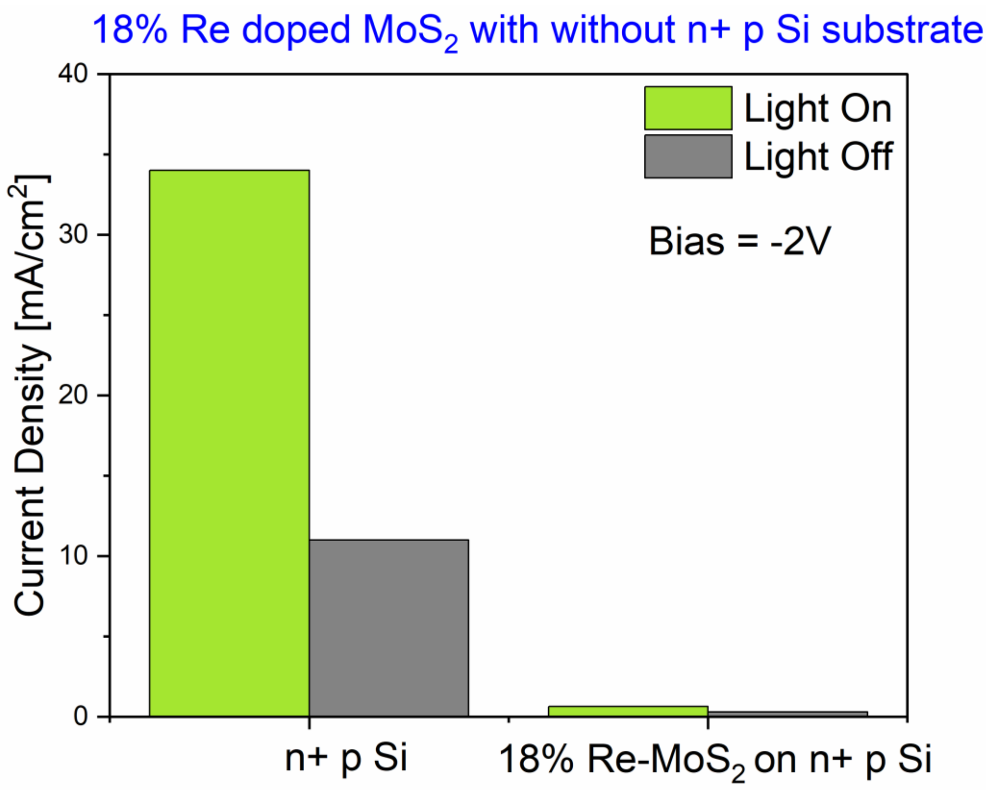
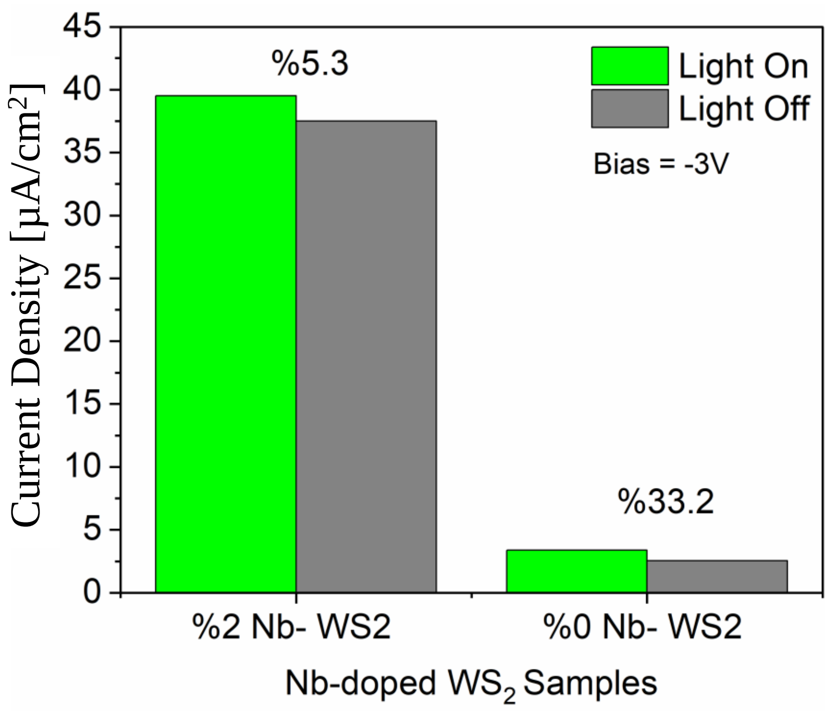


Photo response assessment of TMD films on Al_2O_3 substrate

With 7nm of TiO_2 Capping layer Nb-Doped WS_2

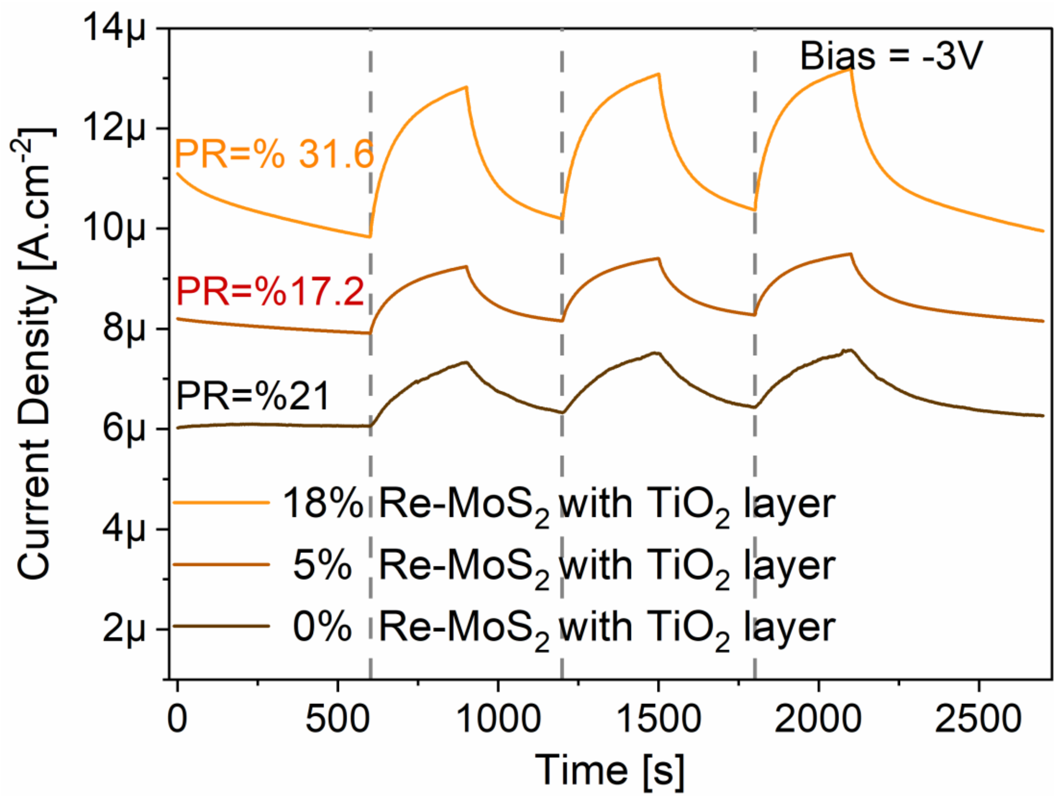
0% Nb-WS_2 , 2% Nb-WS_2



$$Photoresponse\ \%\ [PR] = \frac{I\ on - I\ off}{I\ off}\ x\ 100$$

With 7nm of TiO_2 Capping layer Re-Doped MoS_2

0% Re-MoS_2 , 5% Re-MoS_2 , 18% Re-MoS_2



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Photoelectrochemical Analysis of doped TMD films

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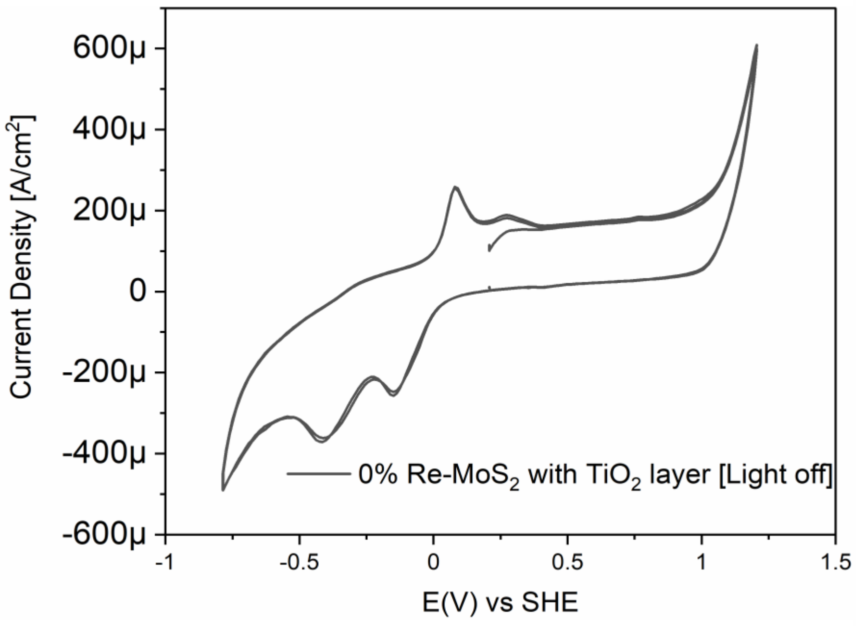
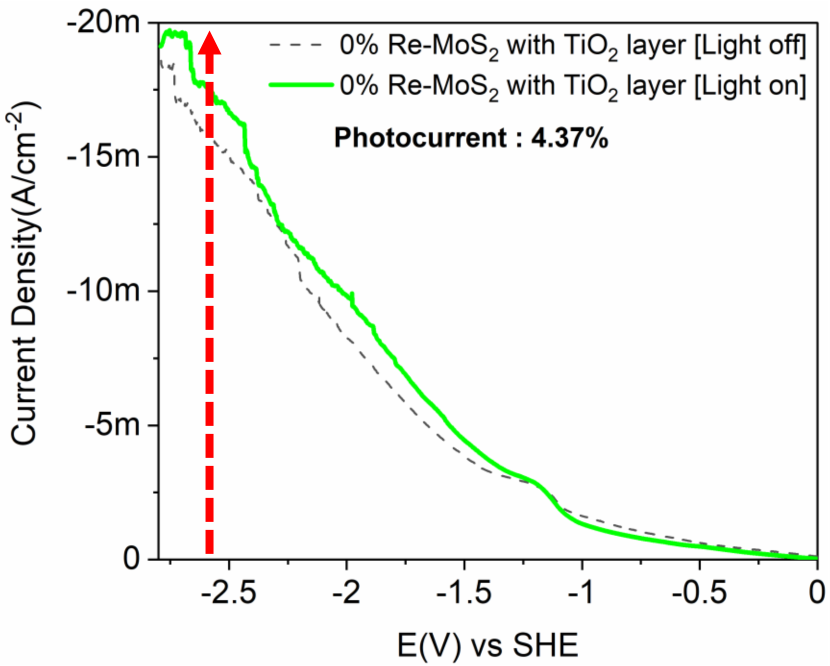
Re-Doped MoS_2 and WS_2 with ~ 7 – 10 nm of TiO2 Capping layer in 0.1M KOH (Ph 13)

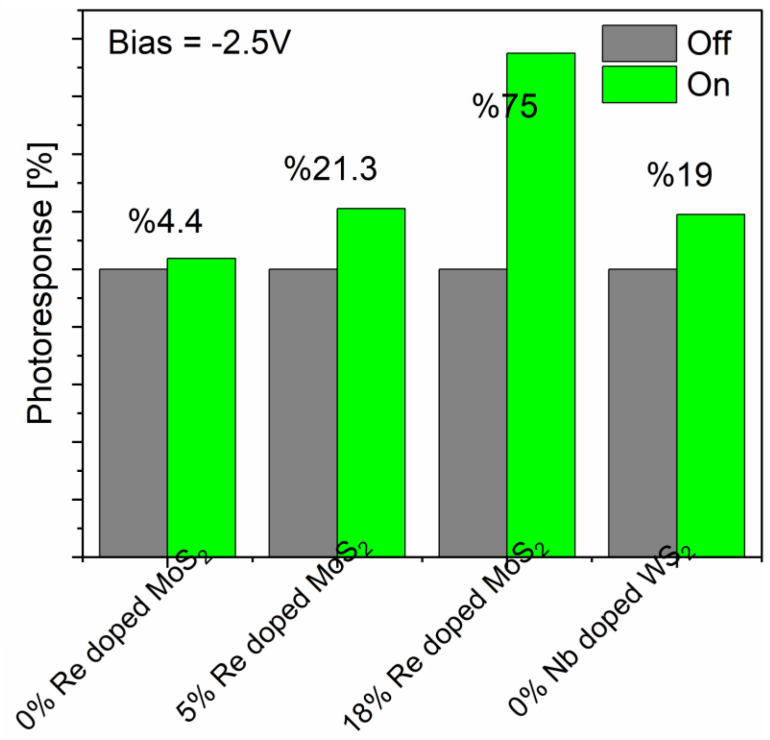
0% Re-MoS_2



0% Re-MoS_2



0% 5% and 18 Re-MoS_2
0% Nb-WS_2



$$Photoresponse\ \%\ [PR] = \frac{I\ on - I\ off}{I\ off}\ x\ 100$$

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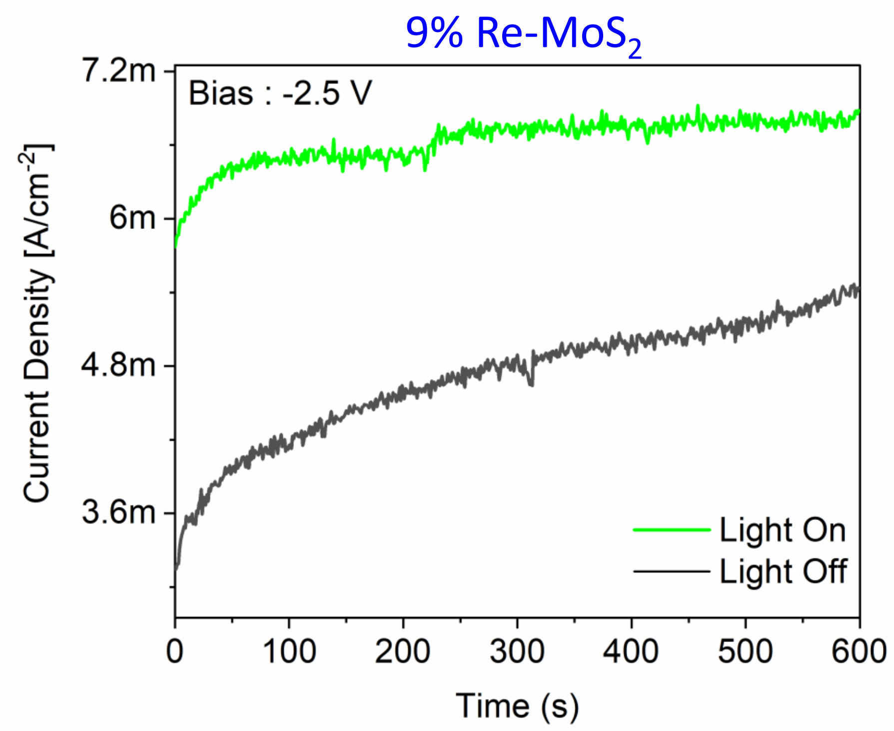
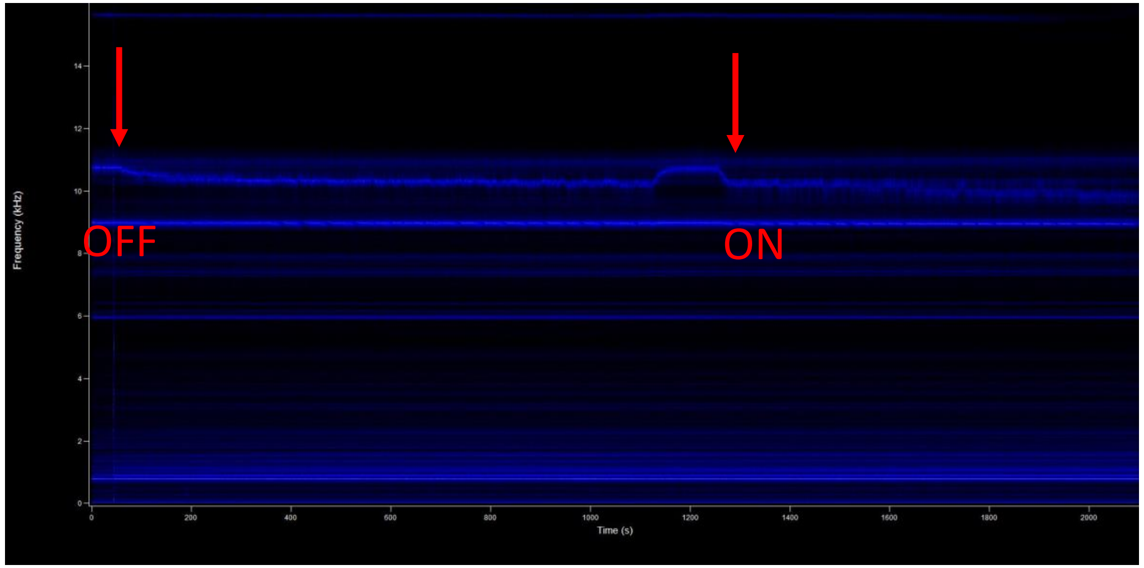
Solar-To-Hydrogen Efficiency



BARDS: Sonic resonance response analysis of H_2



Kang *et al*,. ACS Appl. Energy Mater. 2020, 3, (2000)

- Solar Simulator, AM1.5g 1.1 Suns (equivalent to AM1.5g 0.99 Suns)
- Photocurrent J_{sc} ~ 2.6 mA/cm^2 & H_2 ~ 59.2 nmol/s
- Estimated Solar-To-Hydrogen (STH), **η_{STH} ~ 5.3 %**

Segev *et al*, J. Phys. D: Appl. Phys. 55 323003, (2022)

Alley *et al*,. Methods. 10, (2022)

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Conclusions

✓ TMD films with introducing different amount of dopants

✓ Detection of photo resistance change with doping

✓ Detection of photocurrent in photoelectrochemical assessment with doping

✓ Estimated STH efficiency

Future Work

- More characterisation with the current single layer projects.
- Multilayer structure
- Band gap engineering with different layers
- Defect passivating, high absorbent capping layers

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This project work has received funding from the European Union under grant agreement No 101084261 (FreeHydroCells)

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Questions?