How I Became a Non-Chemist
(with considerable help from Jim Lawler)

Ken Menningen

Department of Physics & Astronomy

With Special Thanks To:

Dr. John A. Turner

National Renewable Energy Laboratory

Golden, CO

Diamond growth plasma

Diamond growth plasma

Diamond crystal growth by plasma chemical vapor deposition

C.-P Chang, D. L. Flamm, D. E. Ibbotson, and J. A. Mucha
AT&T Bell Laboratories, 600 Mountain Avenue, Murray Hill, New Jersey 07974

(Received 3 August 1987; accepted for publication 10 November 1987)

We have grown diamond crystals and polycrystalline diamond films from $CH_4/H_2/O_2$ gas feeds in a simple, high-power density, 2450-MHz discharge tube reactor. Single-crystal growth rates over 20 μm/h have been achieved. The material has been analyzed using Raman spectroscopy, Auger spectroscopy, and x-ray diffraction. Control of nucleation is a major problem for growing sound films, and the high temperatures currently required for growth will limit applications. Oxygen additions were necessary to deposit diamonds over the range of feed composition we studied.

Diamond growth plasma

Diamond crystal growth by plasma chemical vapor deposition

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Contents lists available at ScienceDirect

Diamond & Related Materials

journal homepage: www.elsevier.com/locate/diamond

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DIAMOND AND RELATED MATERIALS

High-rate growth of single crystal diamond in microwave plasma in CH4/H2 and CH4/H2/Ar gas mixtures in presence of intensive soot formation

CrossMark

A.P. Bolshakov [a,b,*], V.G. Ralchenko [a,b], V.Y. Yurov [a,b], A.F. Popovich [a,c], I.A. Antonova [a,b], A.A. Khomich [a,c], E.E. Ashkinazi [a,b], S.G. Ryzhkov [a], A.V. Vlasov [a], A.V. Khomich [a,c]

[a] A.M. Prokhorov General Physics Institute RAS, Vavilov str. 38, Moscow 119991, Russia
[b] National Research Nuclear University MEPhI, Moscow 115409, Russia
[c] Institute of Radio Engineering and Electronics RAS, Pl. Vvedenskogo 1, Fryazino 141190, Russia

Dia

Diamon ... tion

C.-
AT

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We ... O_2 gas
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rat ... an
spe ... najor

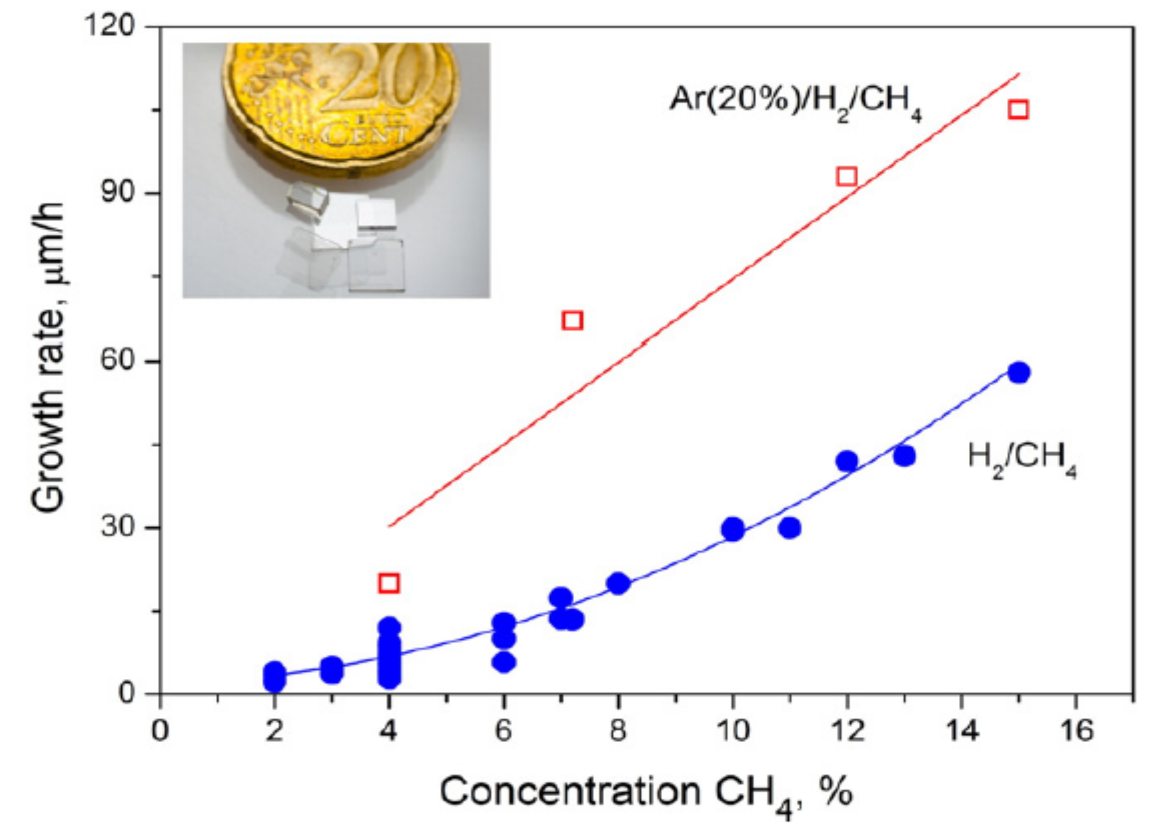

Fig. 2. Dependence of growth rate for SC diamond as function of methane concentration in gas mixtures CH_4/H_2 at 130 Torr, 500 sccm (circles) and Ar (20%)/CH_4/H_2 at 200 Torr, 625 sccm (squares). Inset: a collection of SC CVD diamond plates separated from the substrate and polished. The lines are guides for eye.

ELSEVIER

DIAMOND AND RELATED MATERIALS

High-rat
H2 and

CrossMark

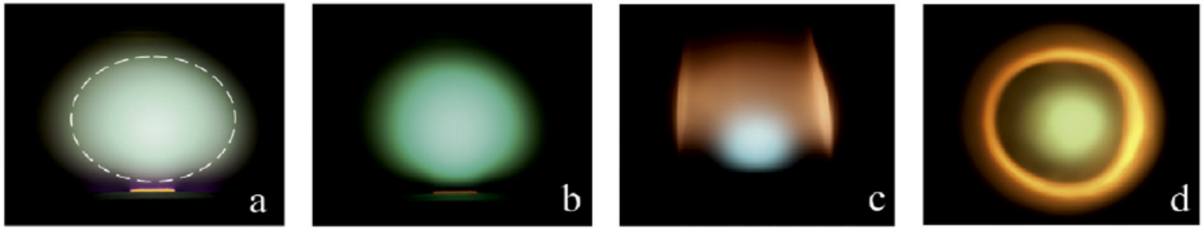

Fig. 3. Photographs of plasma in CH_4/H_2 gas mixture (a) and in CH_4/H_2/20%Ar mixture (b,c,d) with methane content of 4% (a, b) and 15% (c,d). The other process parameters are: microwave power P = 3.0 kW, pressure p = 130 Torr, total gas flow rate 500 sccm in CH_4/H_2 mixture (a), and 3.0 kW, 200 Torr, 625 sccm (b,c,d) in CH_4/H_2/20%Ar. The orange emission from soot is seen in side-view (c) and top-view (d) of the same plasma cloud. The white dashed elliptical contour in image (a) denotes half maximum brightness level used to measure the plasma volume and MW power density (300 W/cm^3 for the particular regime).

A.P. Bolsh ... a,c
E.E. Ashkir

[a] A.M. Prokhorov
[b] National Research Nuclear University MEPhI, Moscow 115409, Russia
[c] Institute of Radio Engineering and Electronics RAS, Pl. Vvedenskogo 1, Fryazino 141190, Russia

Two Stories (Part I)

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- **Diamond growth by molecular beam**

Two Stories (Part I)

- **Diamond growth by molecular beam**
- **Single-crystal diamonds by CVD**

Two Stories (Part I)

Growth of Diamond from Atomic Hydrogen and a Supersonic Free Jet of Methyl Radicals

Szetsen Steven Lee, David W. Minsek, Daniel J. Vestyck, Peter Chen*

The growth of small (~10-micrometer) diamond particles (on 0.1- or 0.25-micrometer seed crystals) using an effusive glow discharge nozzle for H· and a separate supersonic pyrolysis jet for $\cdot CH_3$ is reported. Laser micro-Raman, scanning electron microscopy, and x-ray photoelectron spectroscopy data are presented as evidence that well-crystallized diamond is indeed formed. Resonant multiphoton ionization spectroscopy is used as a diagnostic for the gas-phase chemistry indicating that the radical sources are clean and quantitative and that there is no detectable interconversion of $\cdot CH_3$ to C_2H_2 under the conditions of the experiment. Diamond growth is found at substrate temperatures greater than or equal to 650°C with no marked increase in the rate of growth up to 850°C. Acetylene does not give good quality diamond under similar conditions.

Science New Series, **263**, (Mar. 18, 1994), pp. 1596-1598

Highly sensitive absorption spectroscopy

Hig
spe

Detection of CH_3 during CVD growth of diamond by optical absorption

M.A. Childs, K.L. Menningen, P. Chevako, N.W. Spellmeyer, L.W. Anderson and J.E. Lawler
Department of Physics, University of Wisconsin–Madison, 1150 University Avenue, Madison, WI 53706, USA

Received 6 August 1992; revised manuscript received 17 September 1992; accepted for publication 18 September 1992
Communicated by B. Fricke

Methyl radical production in a hot filament CVD system

K.L. Menningen, M.A. Childs, P. Chevako, H. Toyoda [1], L.W. Anderson and J.E. Lawler
Department of Physics, University of Wisconsin – Madison, 1150 University Avenue, Madison, WI 53706, USA

Received 6 October 1992; in final form 22 December 1992

Ultraviolet spectroscopy of gaseous species in a hot filament diamond deposition system when C_2H_2 and H_2 are the input gases

H. Toyoda,[a] M. A. Childs, K. L. Menningen, L. W. Anderson, and J. E. Lawler
Department of Physics, University of Wisconsin-Madison, 1150 University Avenue, Madison, Wisconsin 53706

(Received 13 May 1993; accepted for publication 23 November 1993)

Evaluation of a substrate pretreatment for hot filament CVD of diamond

K. L. Menningen, M. A. Childs, H. Toyoda,[a] L. W. Anderson, and J. E. Lawler
Department of Physics, University of Wisconsin–Madison, 1150 University Avenue, Madison, Wisconsin 53706

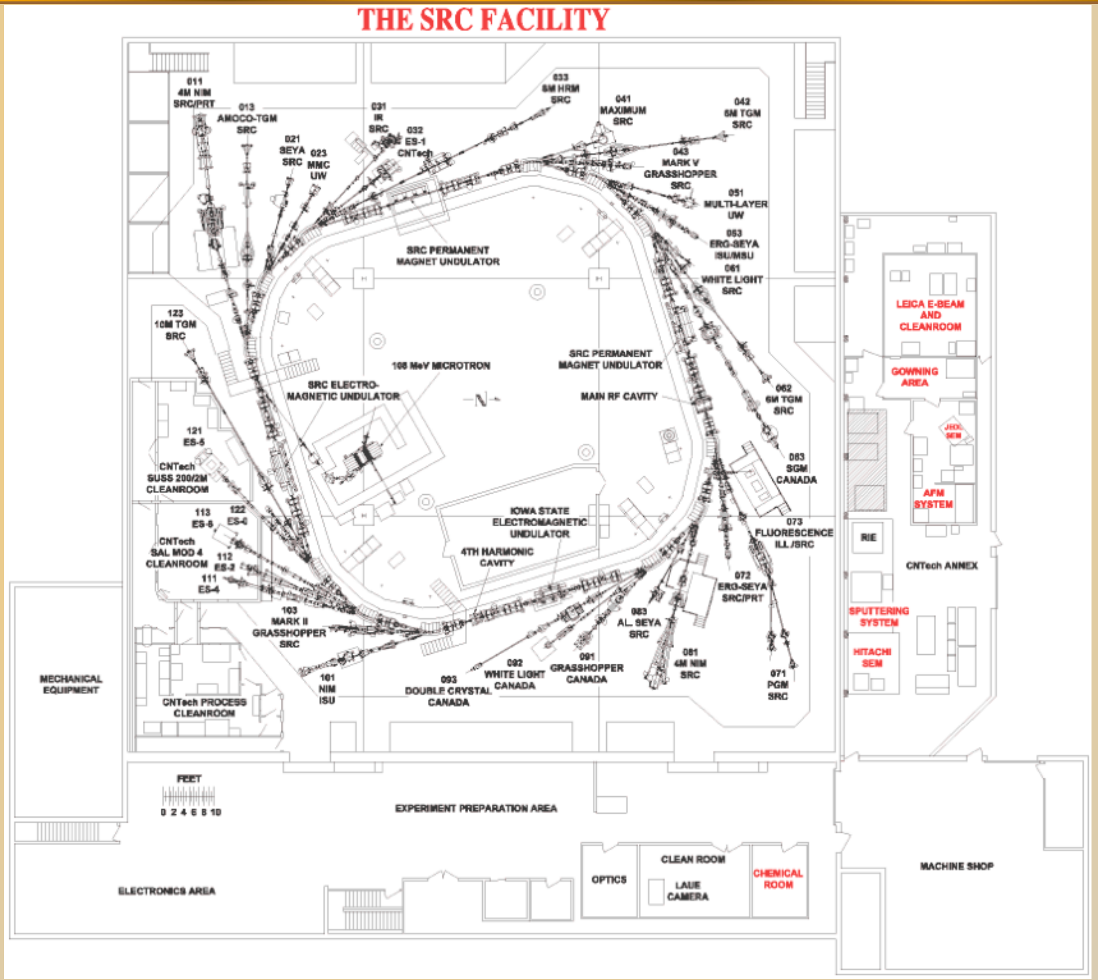
(Received 6 July 1993; accepted 9 December 1993)

Measurement of CH_3 and CH Densities in a Diamond Growth d.c. Discharge.

M. A. Childs, K. L. Menningen, H. Toyoda (*)
L. W. Anderson and J. E. Lawler
Department of Physics, University of Wisconsin-Madison
1150 University Ave., Madison, WI 53706, USA

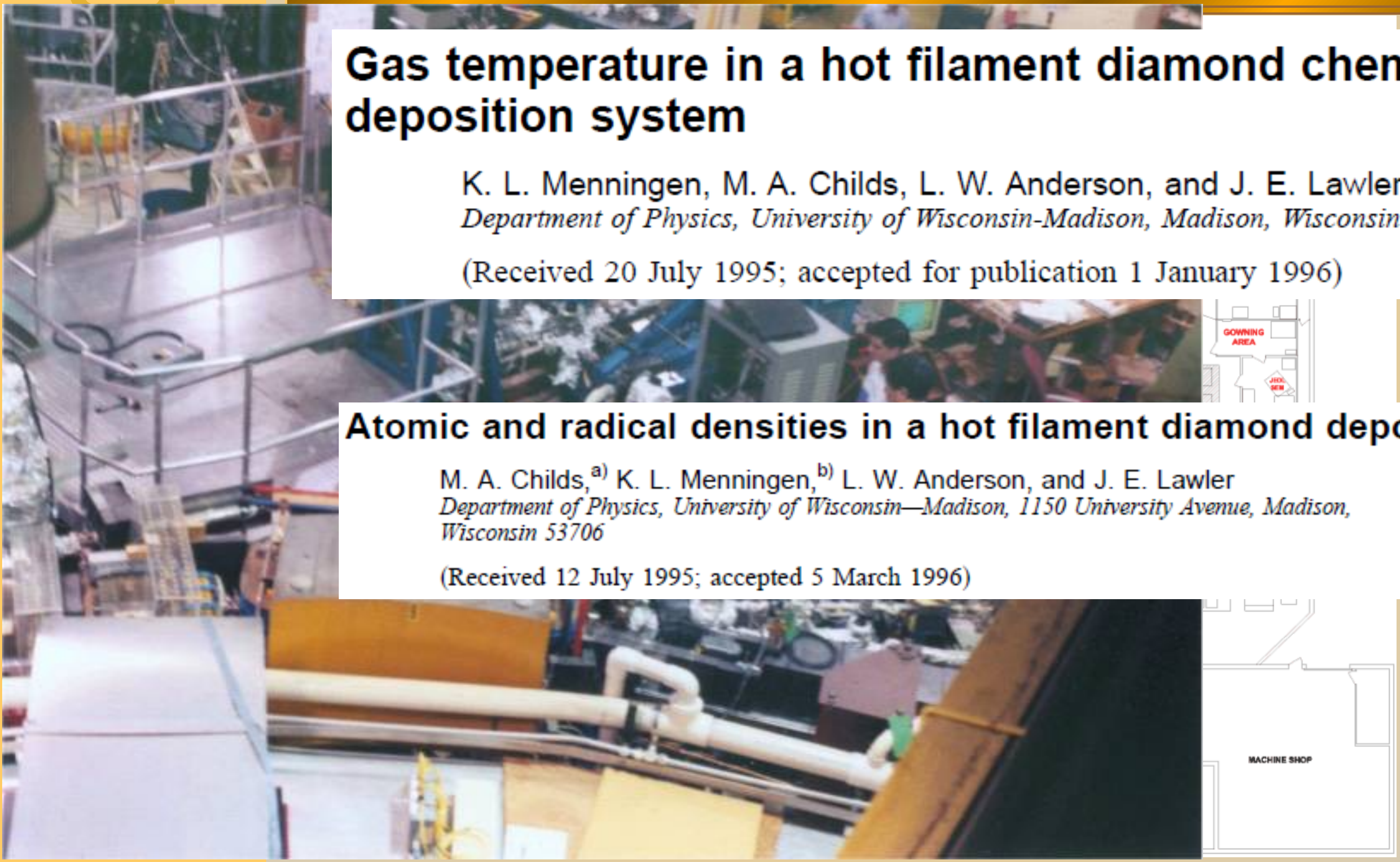
(received 2 November 1993; accepted in final form 27 January 1994)

Synchrotron experiments



Synchrotron experiments

Synchrotron experiments



Gas temperature in a hot filament diamond chemical vapor deposition system

K. L. Menningen, M. A. Childs, L. W. Anderson, and J. E. Lawler
Department of Physics, University of Wisconsin-Madison, Madison, Wisconsin 53706

(Received 20 July 1995; accepted for publication 1 January 1996)

Atomic and radical densities in a hot filament diamond deposition system

M. A. Childs,[a] K. L. Menningen,[b] L. W. Anderson, and J. E. Lawler
Department of Physics, University of Wisconsin—Madison, 1150 University Avenue, Madison, Wisconsin 53706

(Received 12 July 1995; accepted 5 March 1996)

Synchrotron experiments

Synchrotron experiments

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RELATIVE BAND OSCILLATOR STRENGTHS FOR CARBON MONOXIDE: $A\ ^1\Pi - X\ ^1\Sigma^+$ TRANSITIONS

S. R. FEDERMAN,[1] K. L. MENNINGEN,[2] WEI LEE,[1] AND J. B. STOLL[2]
Received 1996 November 12; accepted 1996 December 18

ABSTRACT

Band oscillator strengths for CO transitions between the electronic states $A\ ^1\Pi$ and $X\ ^1\Sigma^+$ were measured via absorption with a synchrotron radiation source. When referenced to the well-characterized (5, 0) band oscillator

THE ASTROPHYSICAL JOURNAL SUPPLEMENT SERIES, 134:133–138, 2001 May
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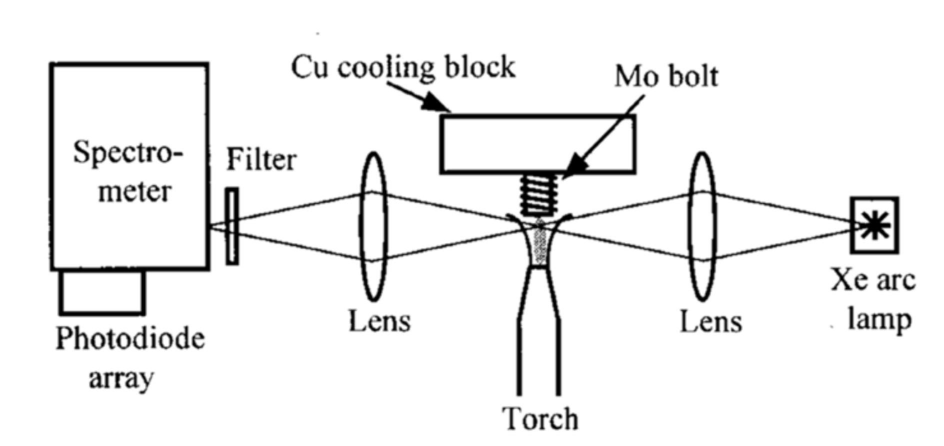
OSCILLATOR STRENGTHS FOR *B–X*, *C–X*, AND *E–X* TRANSITIONS IN CARBON MONOXIDE

S. R. FEDERMAN,[1] M. FRITTS,[1] S. CHENG,[1] K. M. MENNINGEN,[2] DAVID C. KNAUTH,[1] AND K. FULK[2]
Received 2000 August 3; accepted 2000 December 27

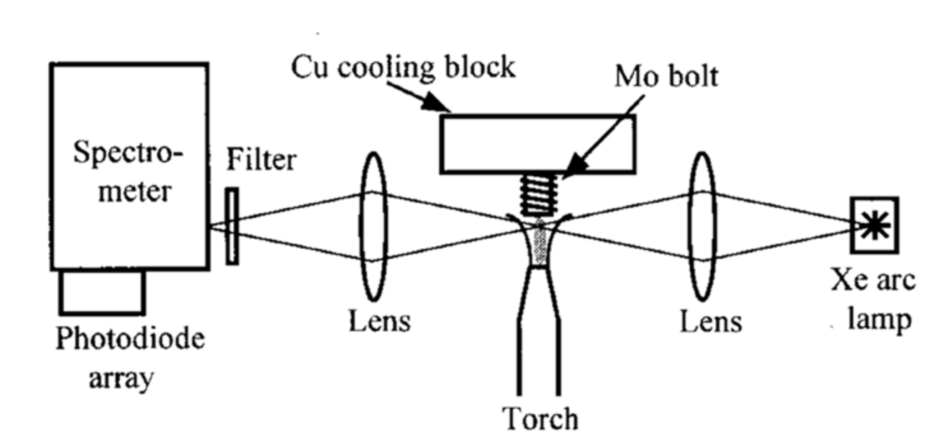
ABSTRACT

Band oscillator strengths for electronic transitions in CO were obtained at the Synchrotron Radiation Center of the University of Wisconsin–Madison. Our focus was on transitions observed in interstellar

Torch papers



Torch papers



Radical density measurements in an oxyacetylene torch diamond growth flame

M. D. Welter and K. L. Menningen[a)]
Department of Physics, University of Wisconsin—Whitewater, 800 West Main Street, Whitewater, Wisconsin 53190

(Received 29 January 1997; accepted for publication 5 May 1997)

J. Phys. D: Appl. Phys. **32** (1999) 937–941. Printed in the UK   PII: S0022-3727(99)98735-1

Radical density measurements in an atmospheric pressure oxyacetylene torch

S J Firchow and K L Menningen

Department of Physics, University of Wisconsin - Whitewater, 800 West Main Street,
Whitewater, WI 53190, USA

Received 27 October 1998, in final form 8 January 1999

Hg radiation trapping papers

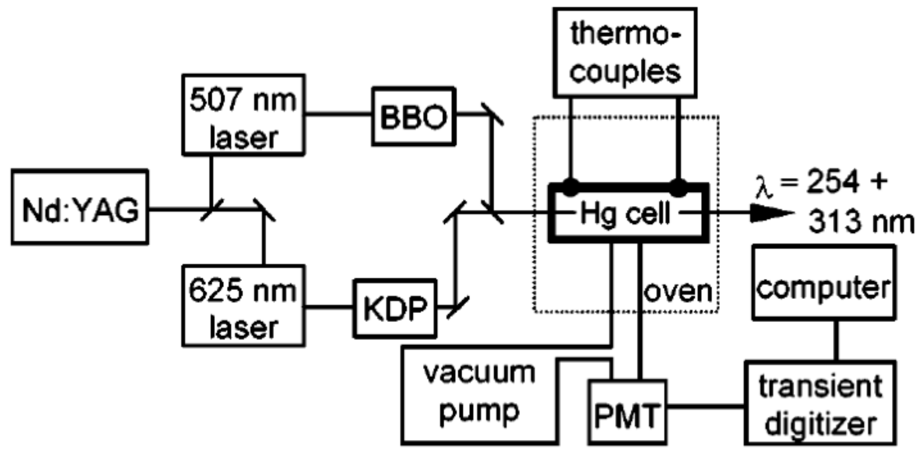


FIG. 1. Schematic diagram of the experiment.

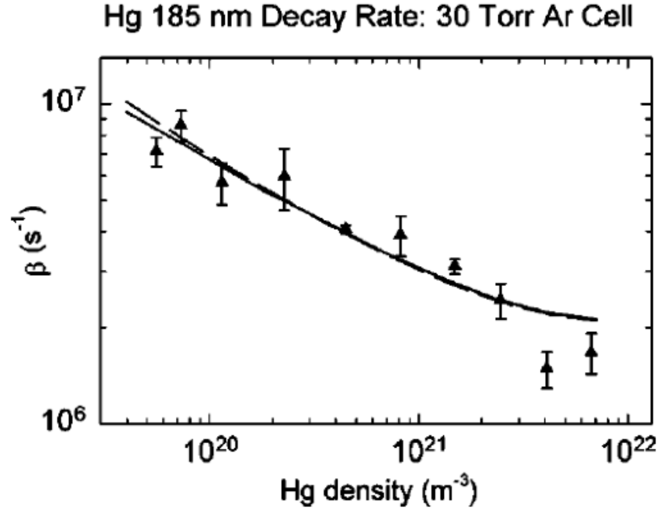


FIG. 4. Measured Hg 185 nm decay rates as a function of Hg density in a sealed cell of radius $h = 10.5$ mm and with 30 Torr of Ar buffer gas. The error bars represent scatter in the measurements of a single run. The solid line indicates the Monte Carlo simulation results, and the dashed line is the analytic formula.

Hg radiation trapping papers

JOURNAL OF APPLIED PHYSICS VOLUME 88, NUMBER 6 15 SEPTEMBER 2000

Radiation trapping of the Hg 185 nm resonance line

K. L. Menningen[a] and J. E. Lawler

Department of Physics, University of Wisconsin-Madison, 1150 University Avenue, Madison, Wisconsin 53706

(Received 29 December 1999; accepted for publication 20 June 2000)

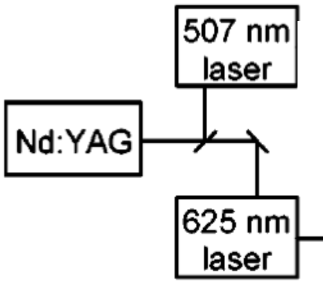


FIG. 1. Sche

INSTITUTE OF PHYSICS PUBLISHING JOURNAL OF PHYSICS D: APPLIED PHYSICS

J. Phys. D: Appl. Phys. **38** (2005) 3304–3311 doi:10.1088/0022-3727/38/17/S35

Radiation trapping of the Hg 254 nm resonance line

M T Herd[1], J E Lawler[1] and K L Menningen[2]

[1] Department of Physics, University of Wisconsin, Madison, WI 53705, USA
[2] Department of Physics and Astronomy, University of Wisconsin, Stevens Point, WI 54481, USA

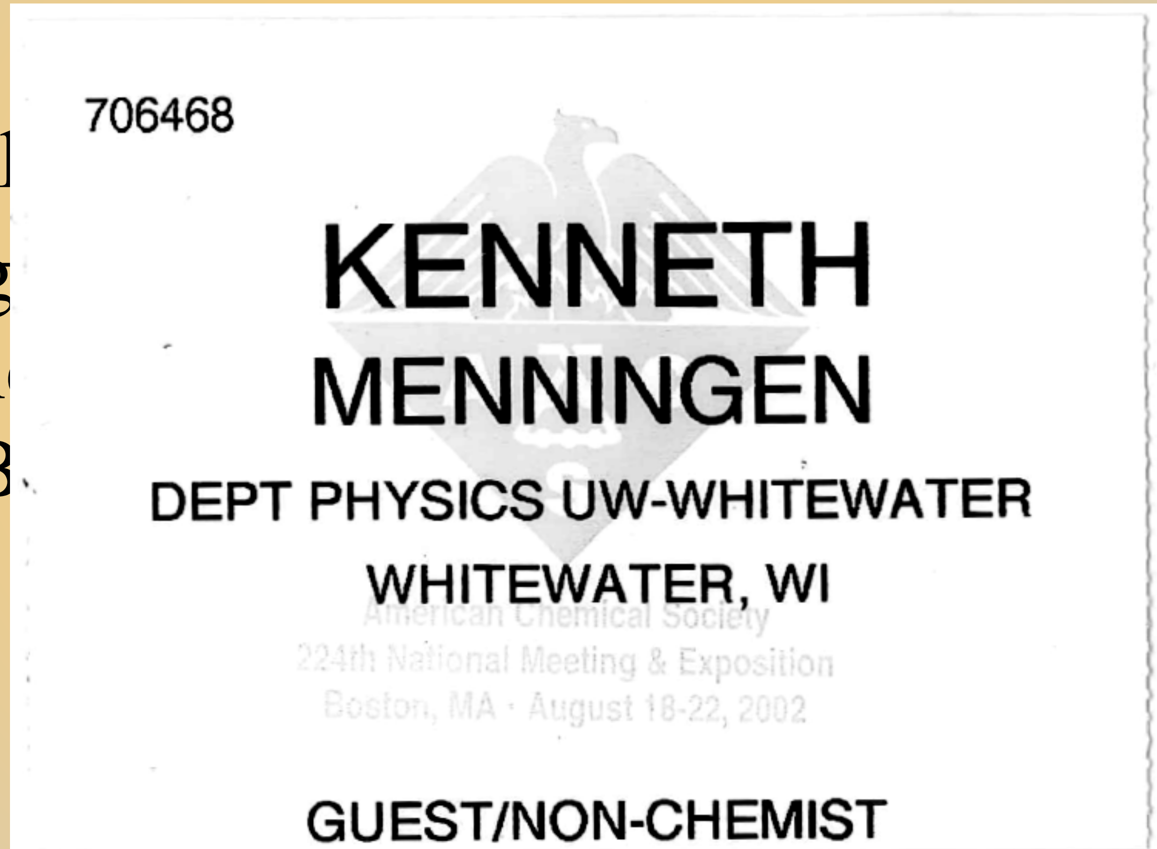
Received 13 February 2005, in final form 16 March 2005
Published 19 August 2005
Online at stacks.iop.org/JPhysD/38/3304

Post Hg Radiation trapping

- Help with the energy crisis?
- Begin exploring options by attending American Chemical Society meeting 2002 in Boston, MA

Post Hg Radiation trapping

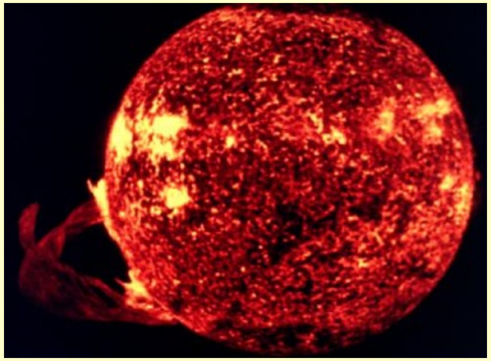
- Hel
- Beg
  Am 2002
  in B



2003 NREL Sabbatical

- Moved family to Lakewood, CO
- Worked with Dr. John Turner
- Spectroscopic evaluation of porphyrin surface treatment

The Electron Economy



Inputs:

Solar Energy

(which also powers wind, waves, etc.)

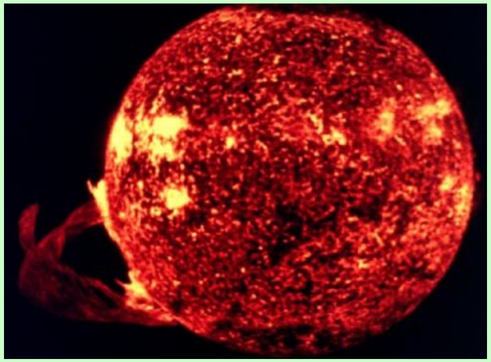
→ High Performance Battery →

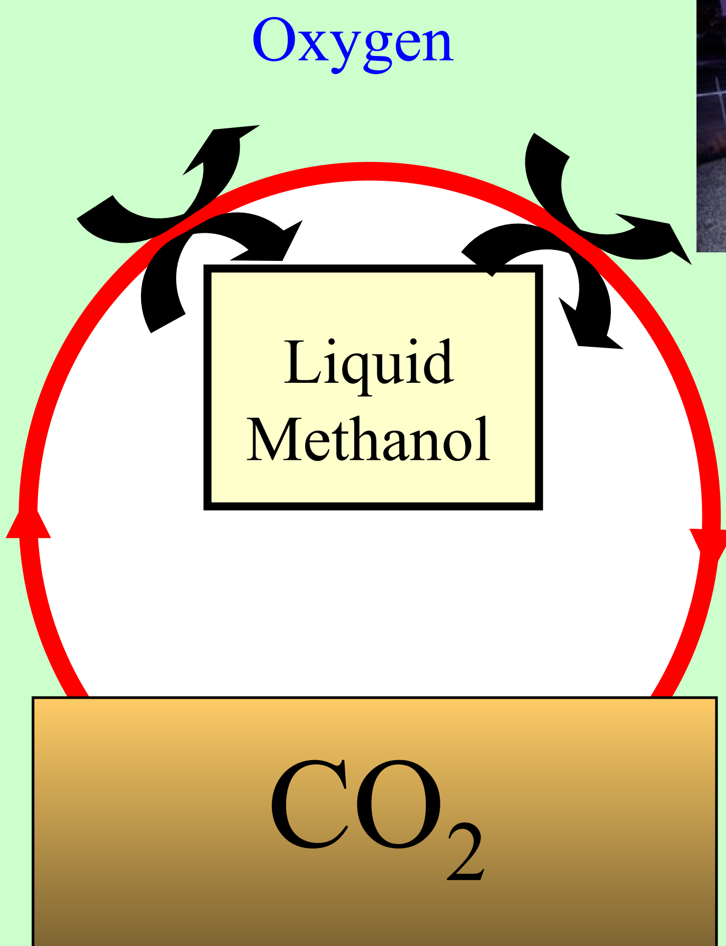
Outputs:

Electricity, Heat

NREL

CO_2 *Economy Closed Energy Cycle*





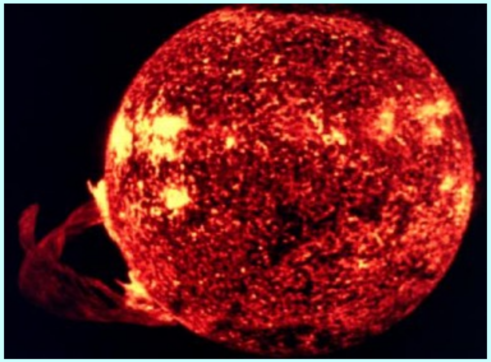
Inputs:

Solar Energy and Water

Outputs:

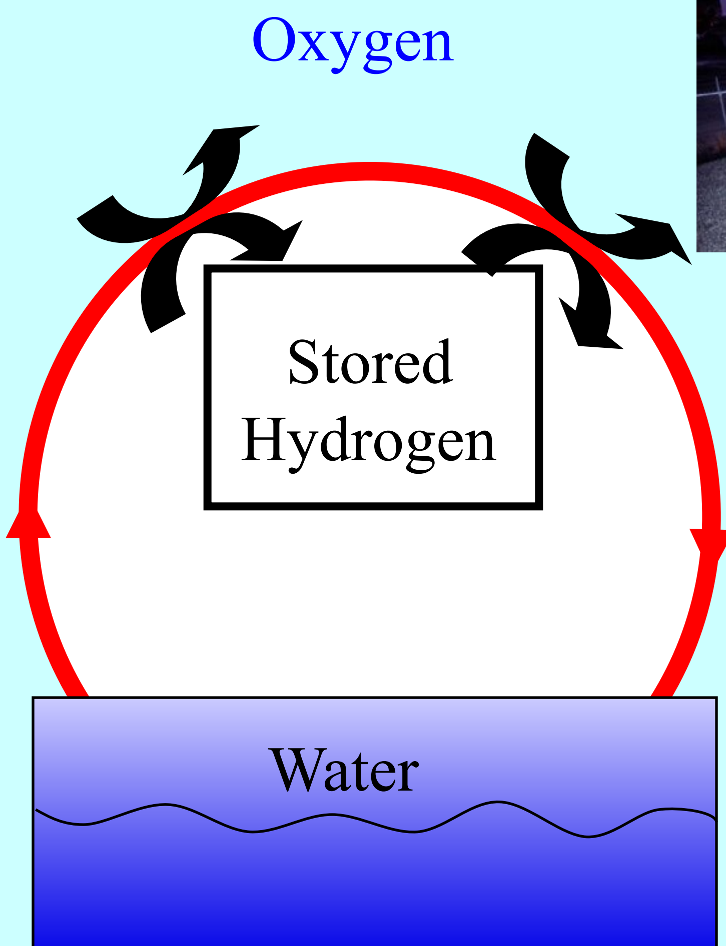
Electricity, Heat and Water

Hydrogen Economy Closed Energy Cycle



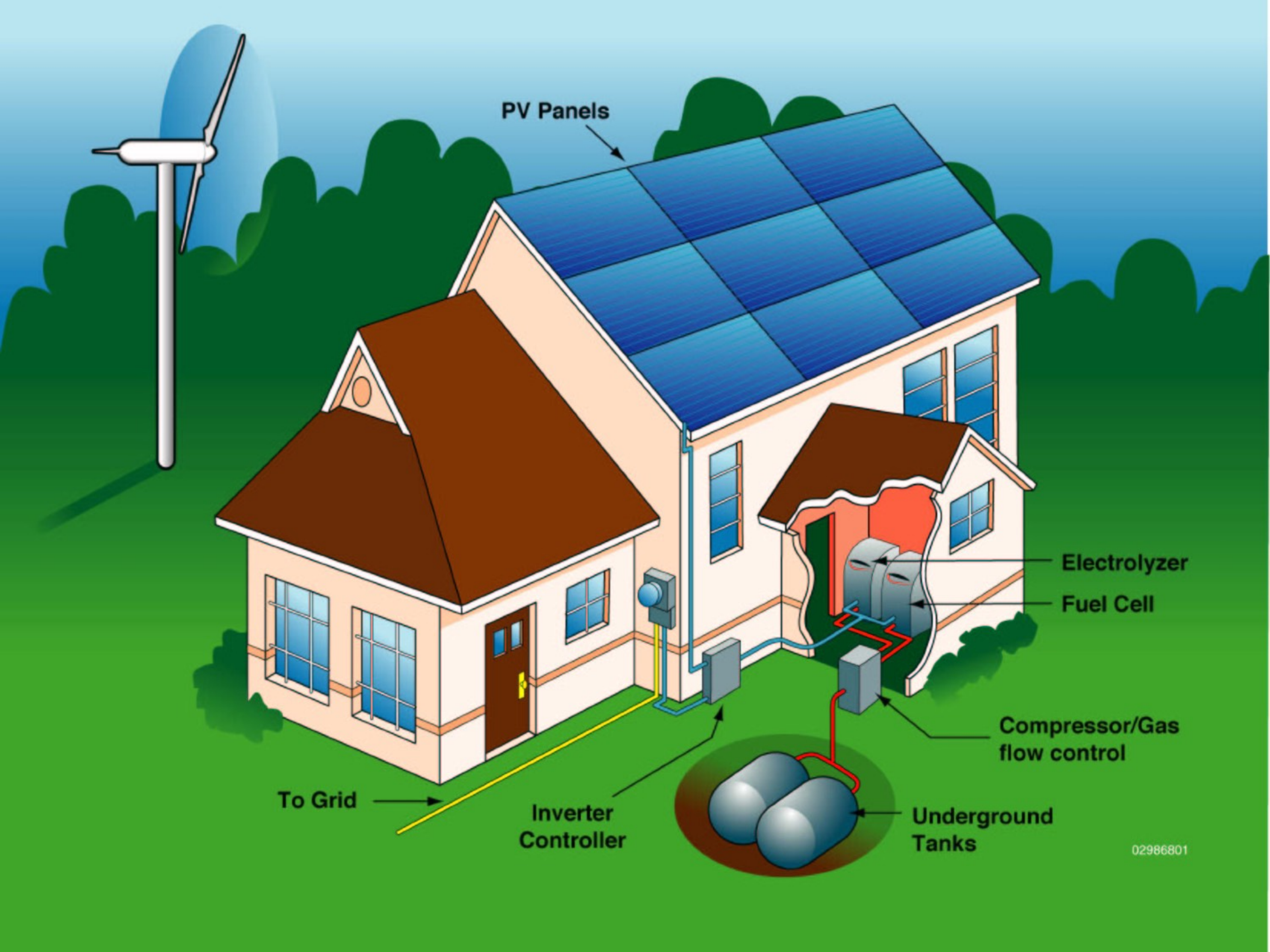
Inputs:

Solar Energy and Water



Outputs:

Electricity, Heat and Water

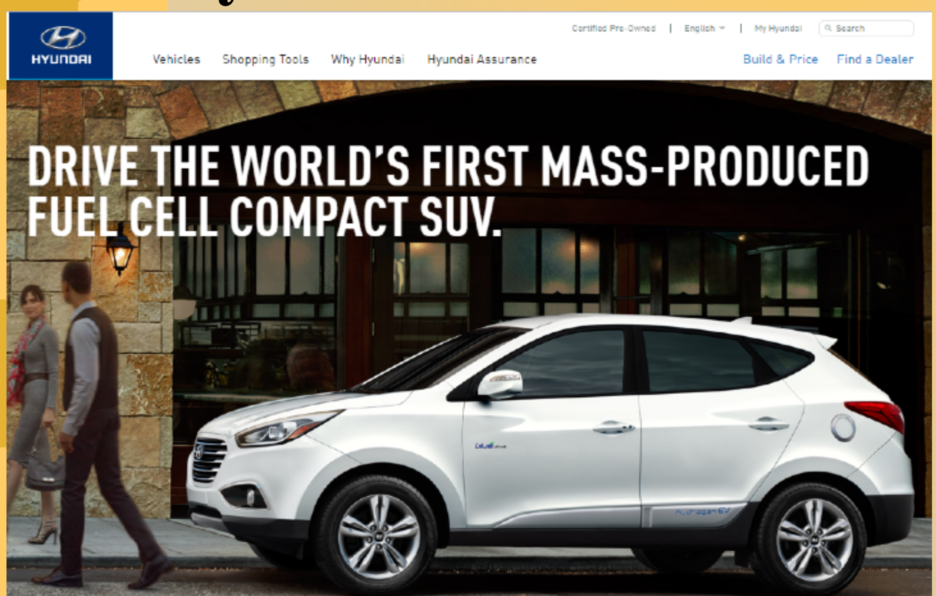

PV Panels
Electrolyzer
Fuel Cell
Compressor/Gas flow control
Underground Tanks
Inverter Controller
To Grid
02986801

2017 Hydrogen cars

You can buy *three* fuel cell cars in California:

Toyota Mirai

Hyundai Tucson

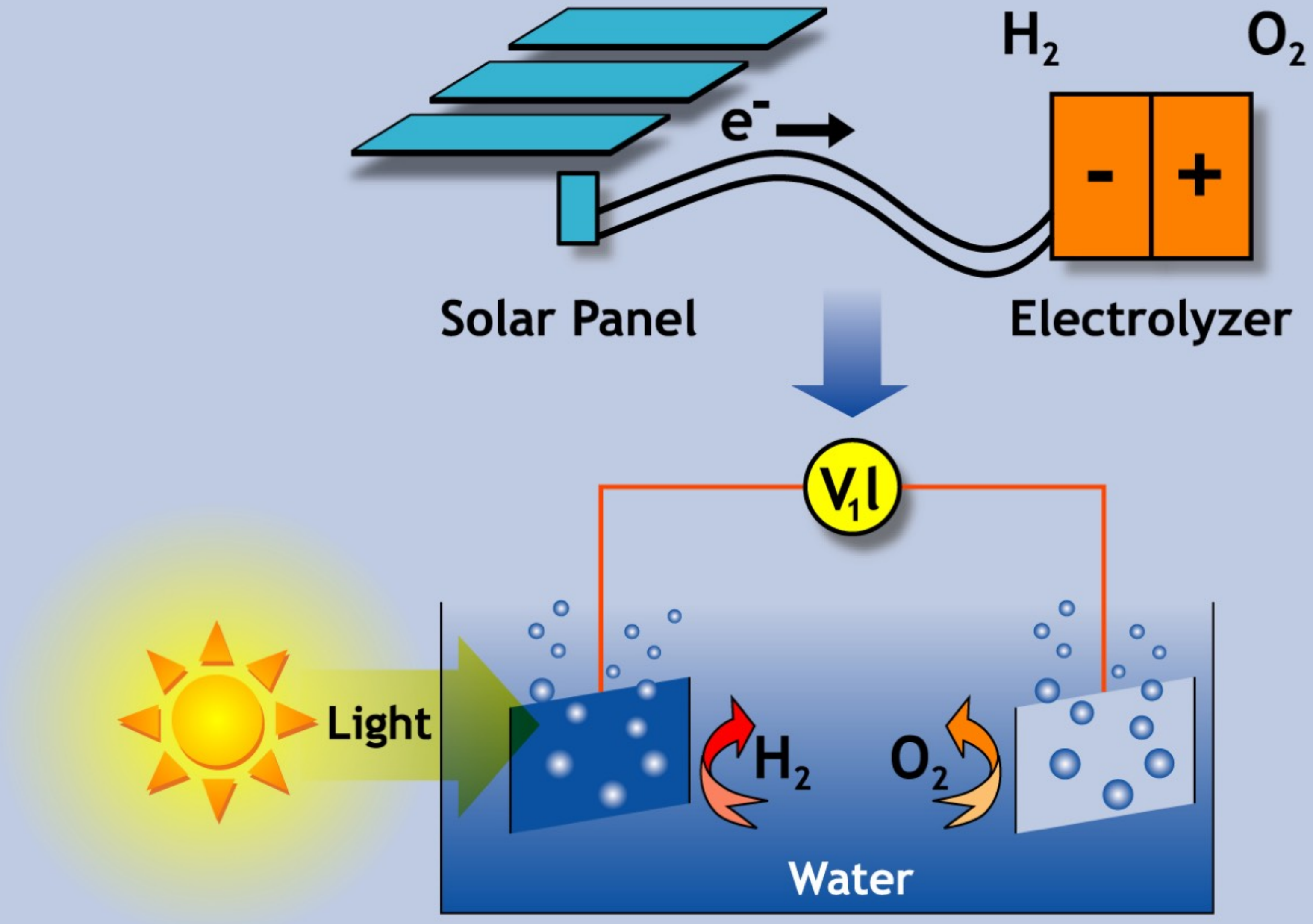


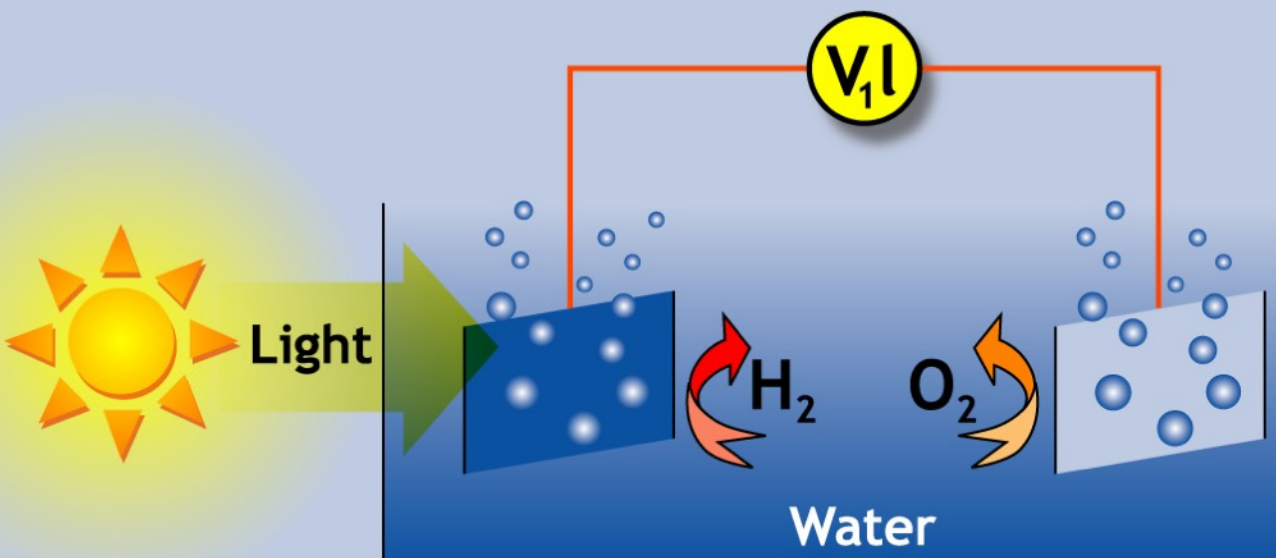
Honda Clarity

Safety

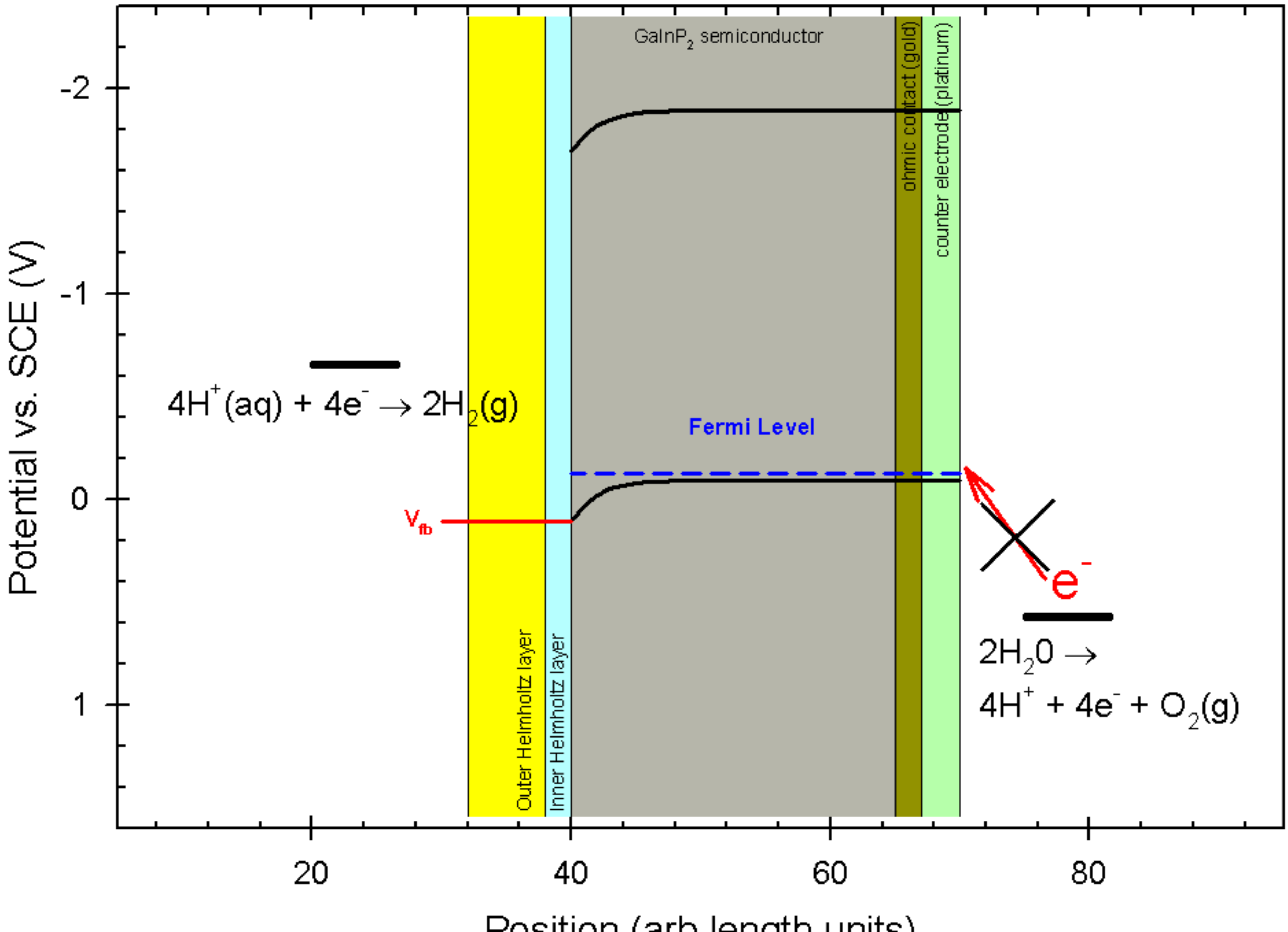
- Fuel leak simulation
  - ➔hydrogen on left
  - ➔gasoline on right
  - ●equivalent energy release
- Hydrogen has safety advantages as well as energy security and environmental advantages.

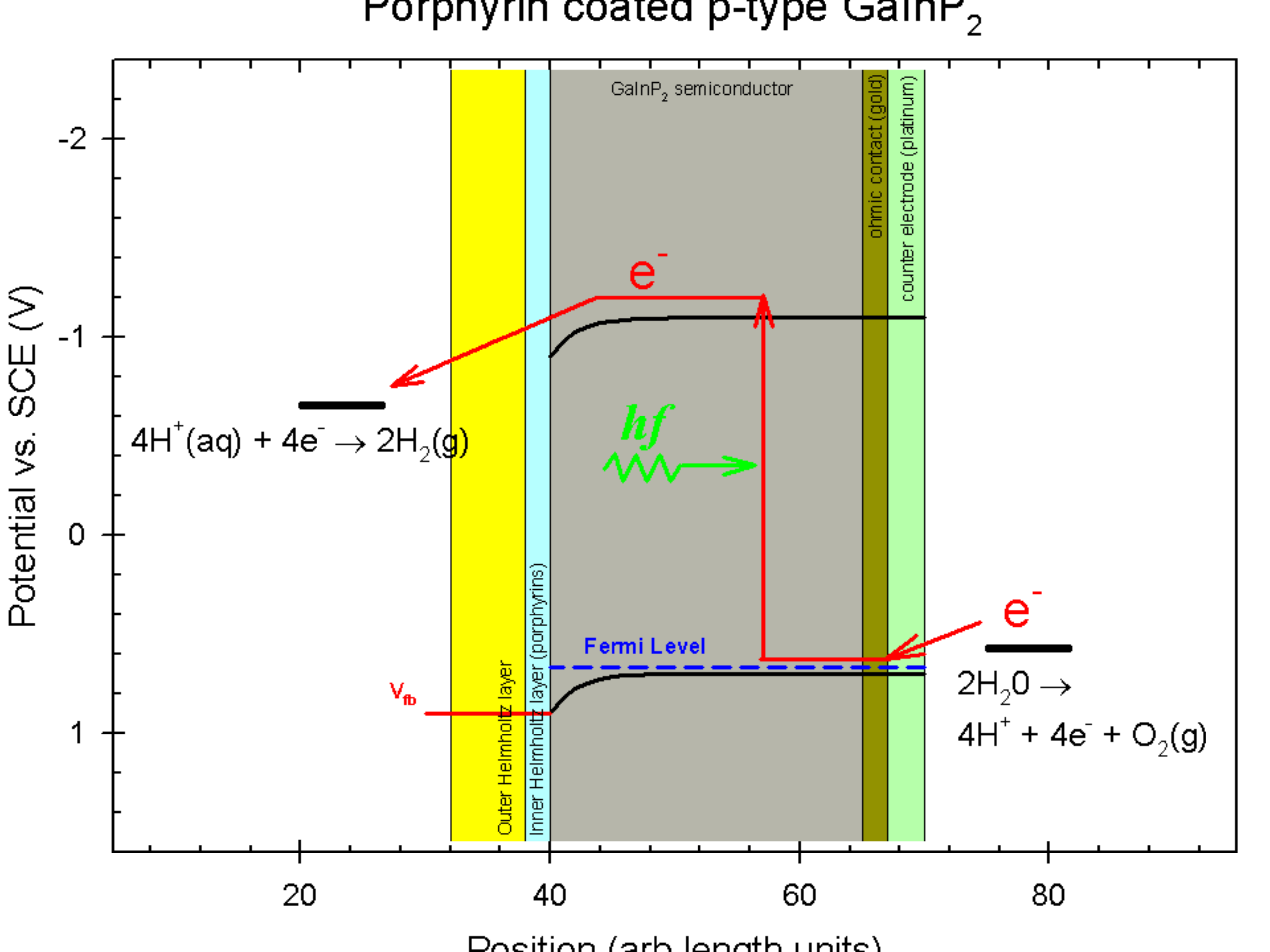
Goal of the Research




V,I
Light
H2
O2
Water

Clean, bare $GaInP_2$





Porphyrin coated p-type GaInP2
GaInP2 semiconductor
ohmic contact (gold)
counter electrode (platinum)
e-
hf
4H+(aq) + 4e- → 2H2(g)
e-
Fermi Level
Vfb
Outer Helmholtz layer
Inner Helmholtz layer (porphyrins)
2H20 →
4H+ + 4e- + O2(g)
-2
-1
0
1
20
40
60
80
Potential vs. SCE (V)
Position (arb length units)

Brief review of PEC water splitting

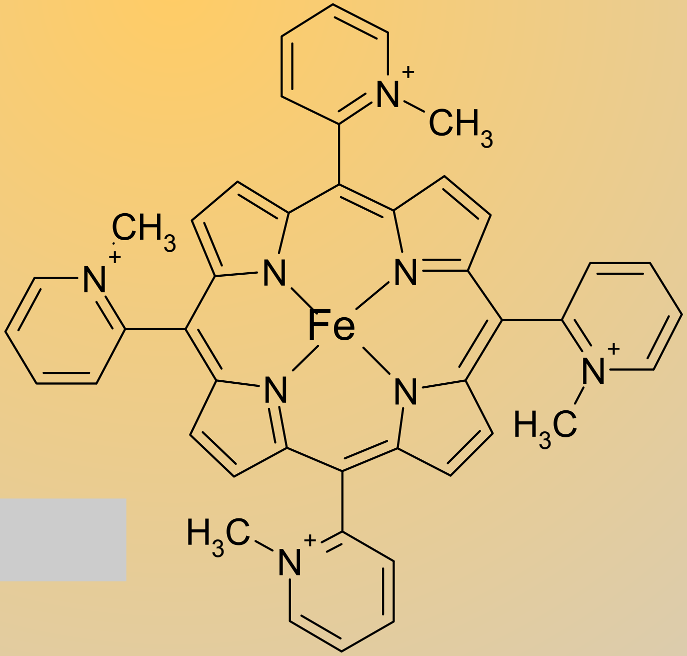
- The ideal semiconductor must:
  - have a band gap ~1.6-1.8 eV
  - have band edges that straddle water redox potentials
  - be stable against corrosion >10,000 hrs
  - efficiently transfer charge across surface
- No such semiconductor has been identified!

Project Goals

- Modify the surface of $GaInP_2$ to
- Shift the band edges and
- Catalyze charge transfer so that it will
- Split water using only sunlight

One Surface Treatment

- Iron tetrakis(N-methyl-2-pyridyl) porphyrin {FeTMPyP(2)} is among the best performers
- Mixing different porphyrins enhances the effect



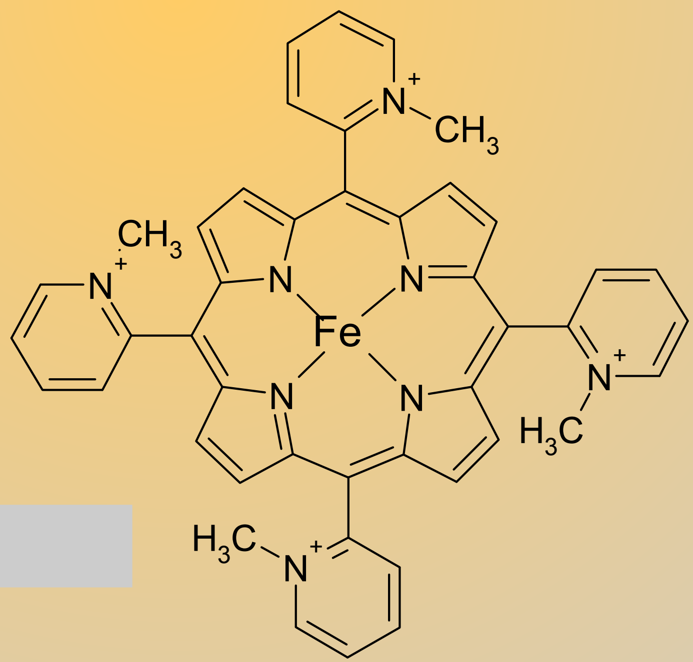
One Surface Treatment

Displacement of the Bandedges of $GaInP_2$ in Aqueous Electrolytes Induced by Surface Modification

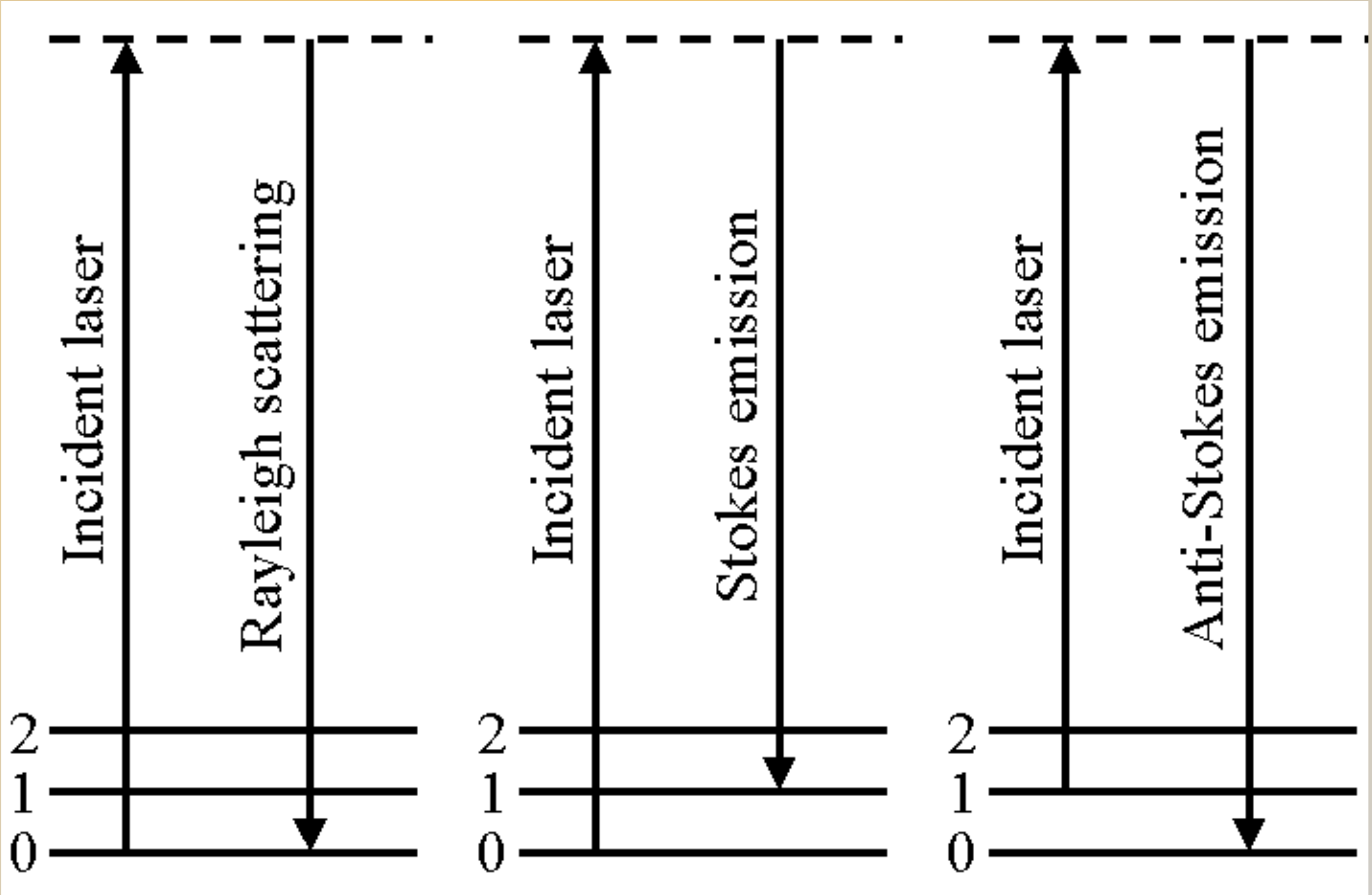
Shyam S. Kocha* and John A. Turner*

National Renewable Energy Laboratory, Photoconversion Branch, Golden, Colorado 80401, USA

- Mixing different porphyrins enhances the effect

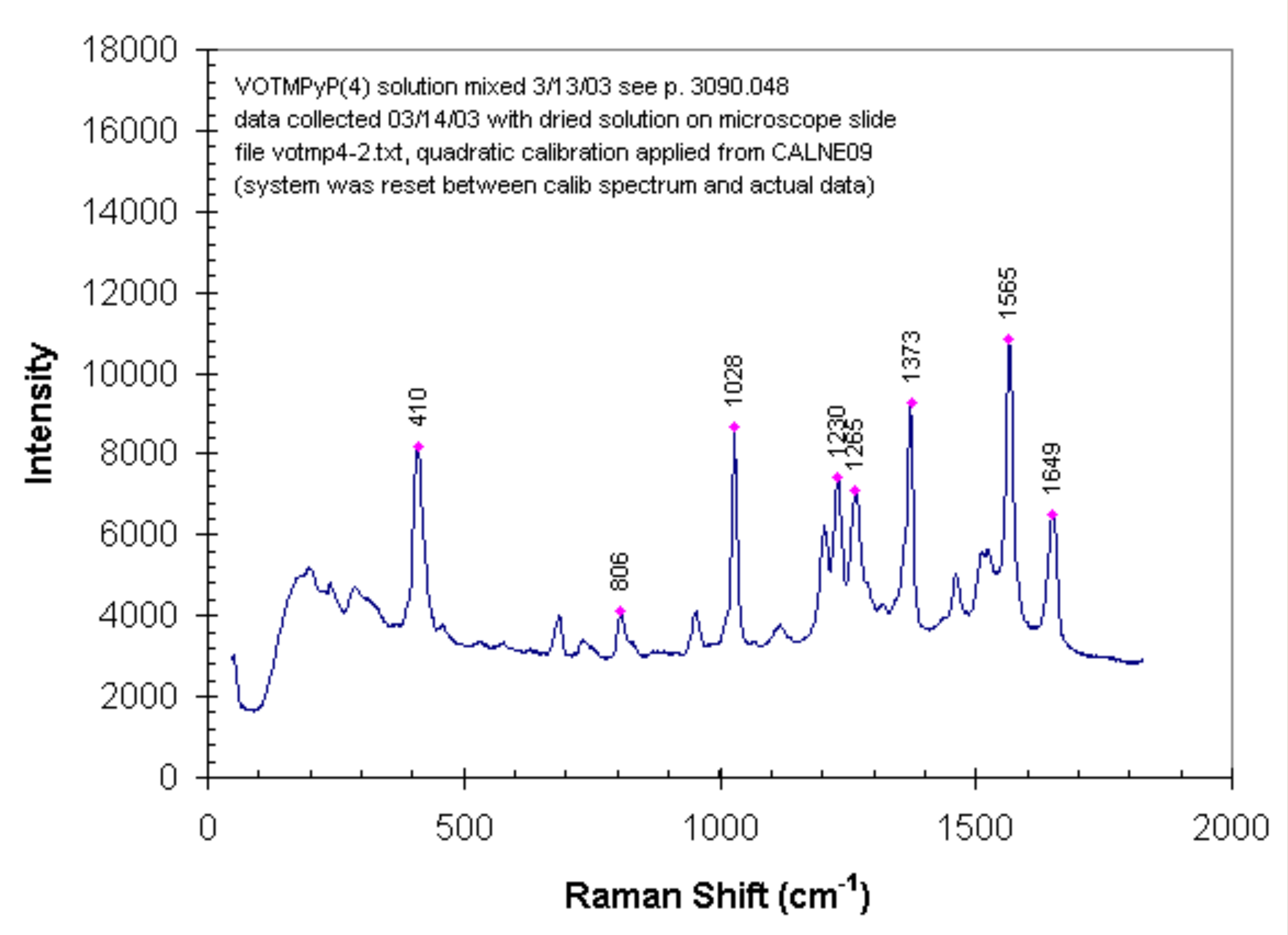


Raman Overview

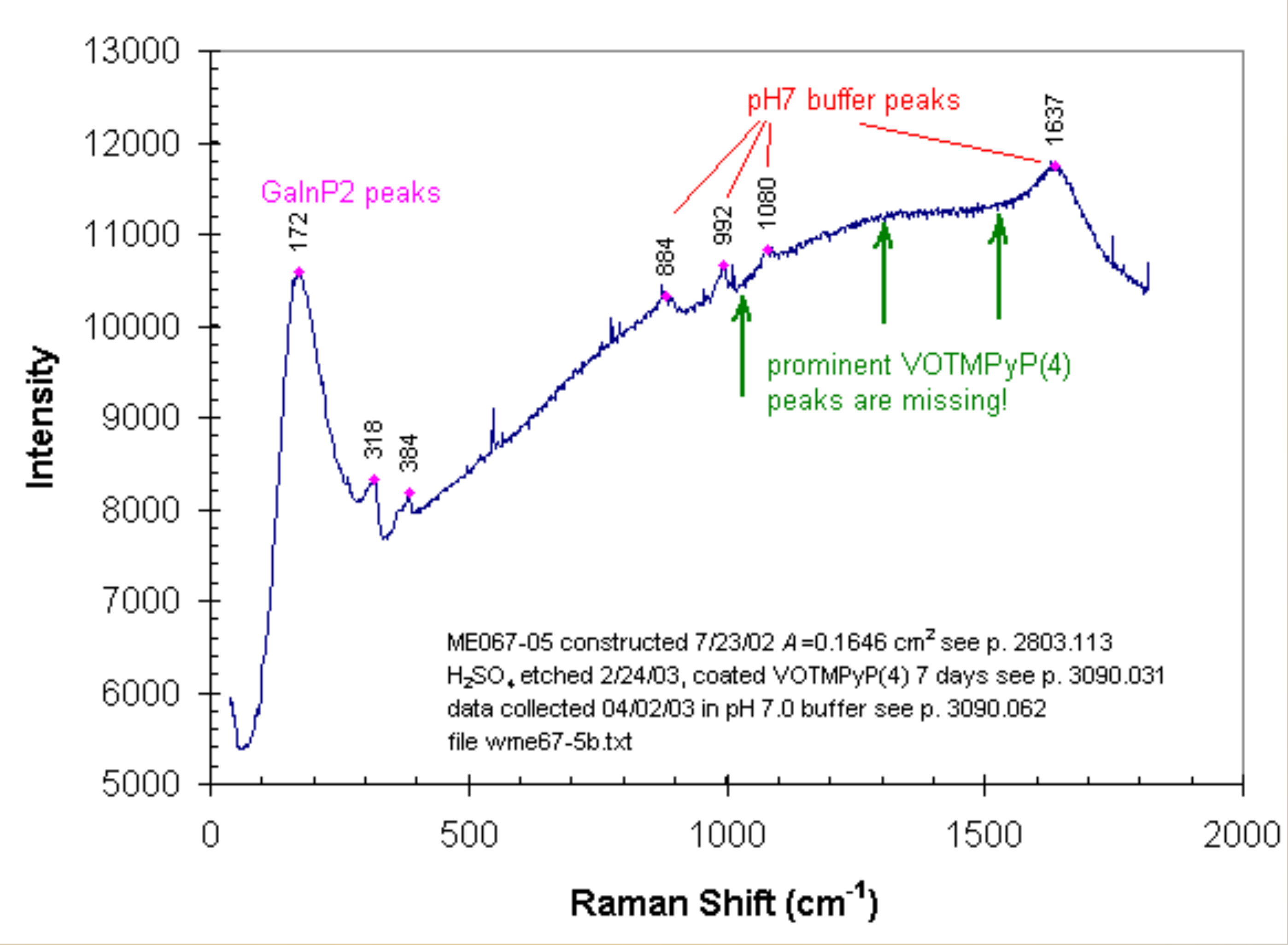


Raman Overview

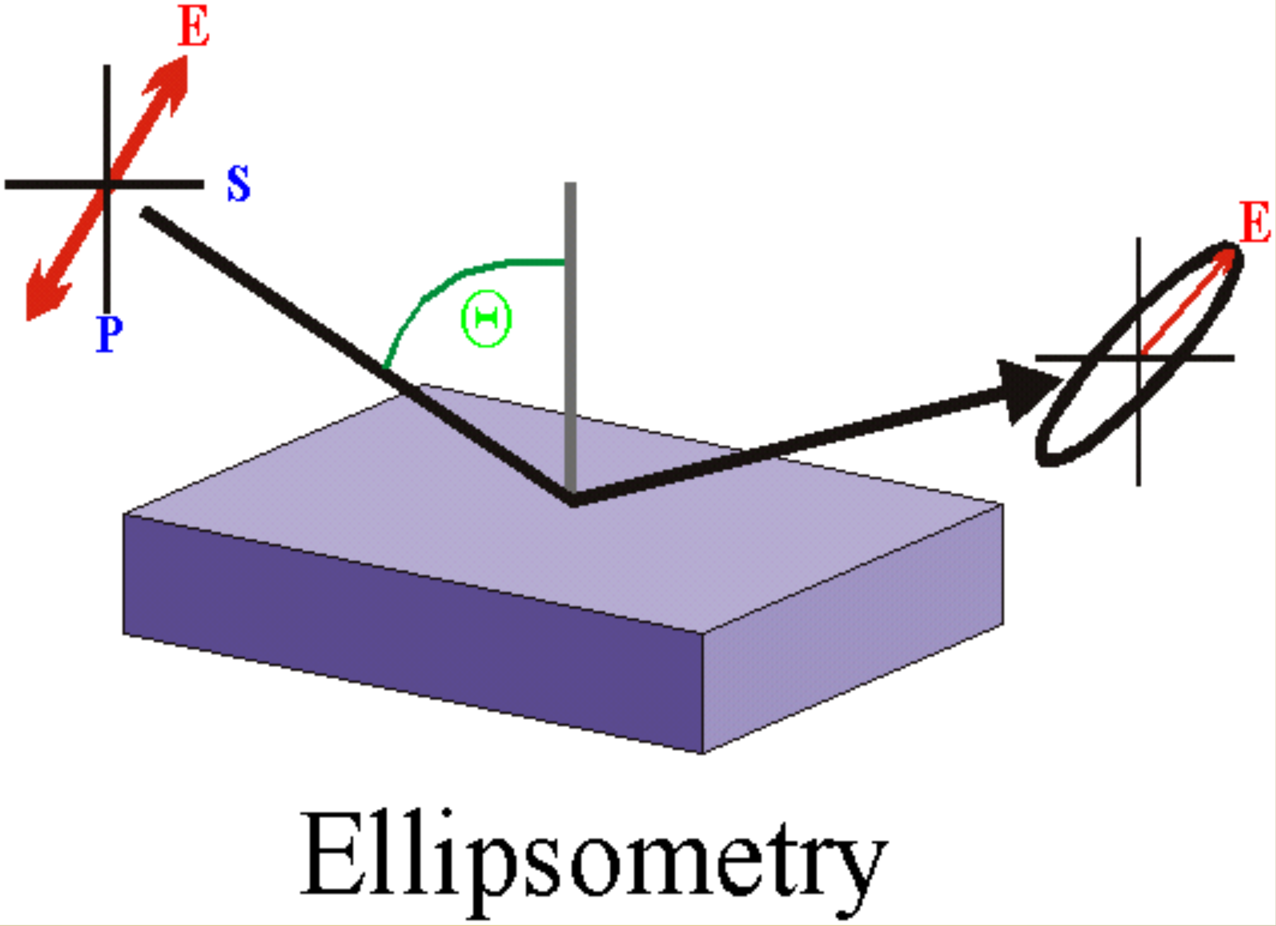
VOTMPyP(4) on glass slide



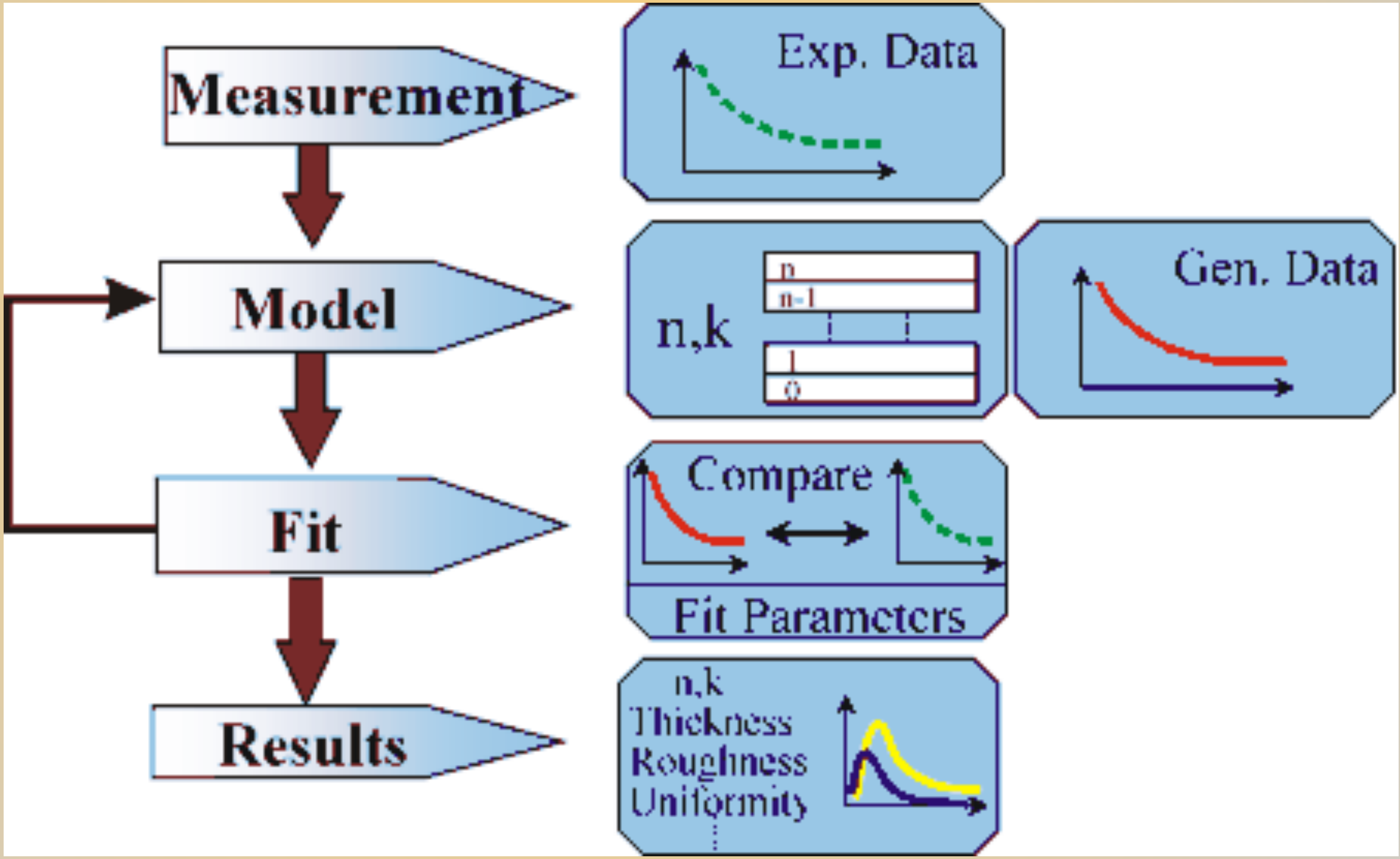
VOTMPyP(4) on wet $GaInP_2$



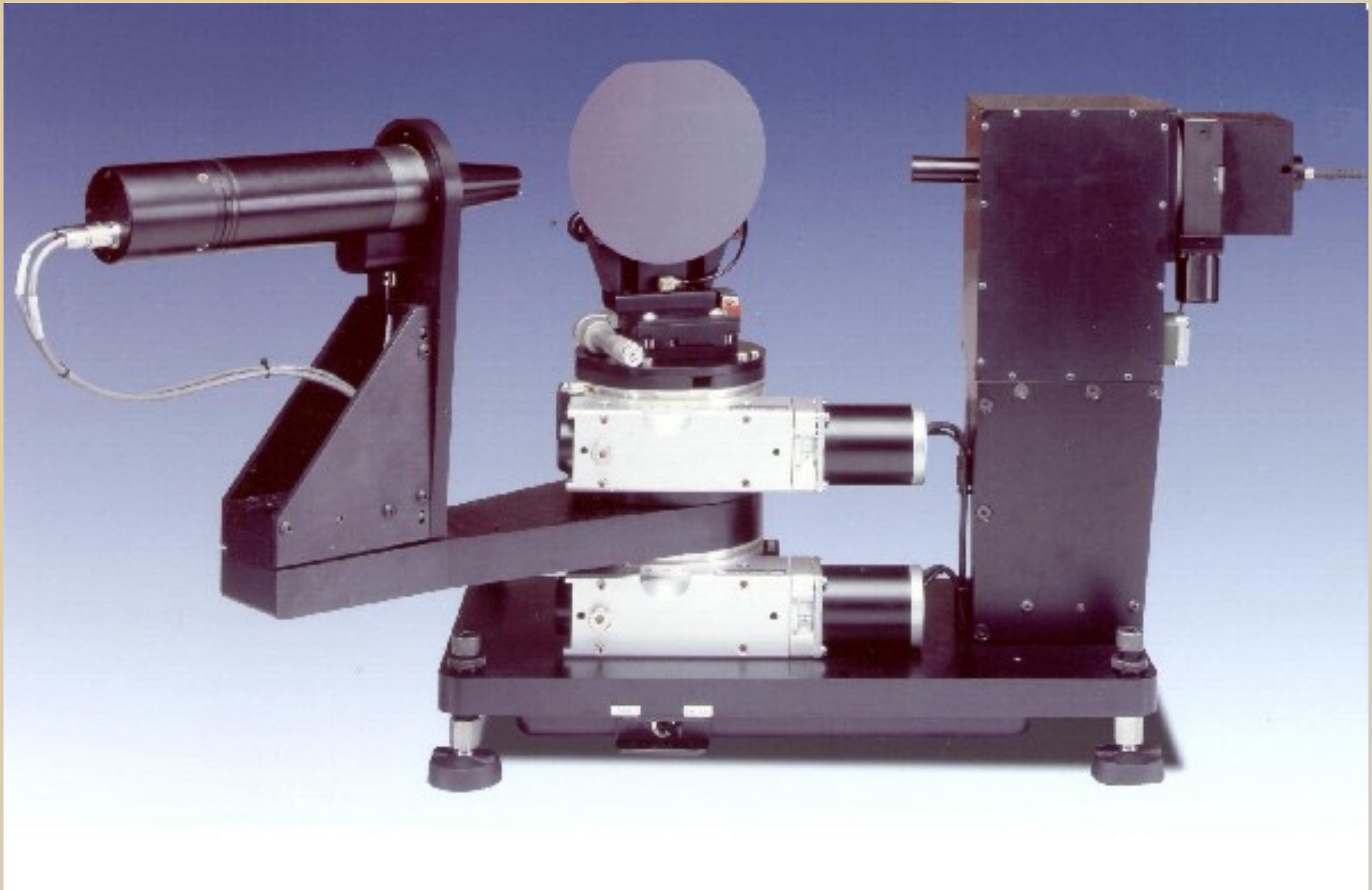
Ellipsometry overview



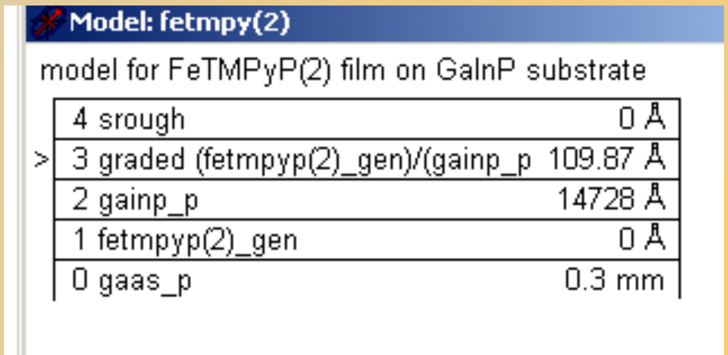
Ellipsometry overview

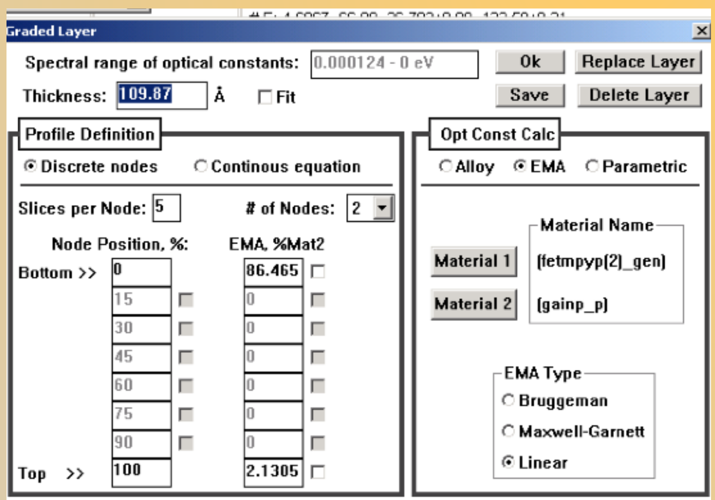


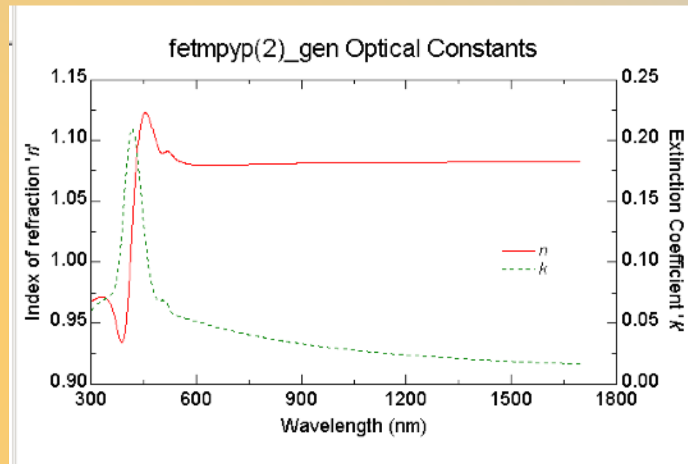
Ellipsometry overview

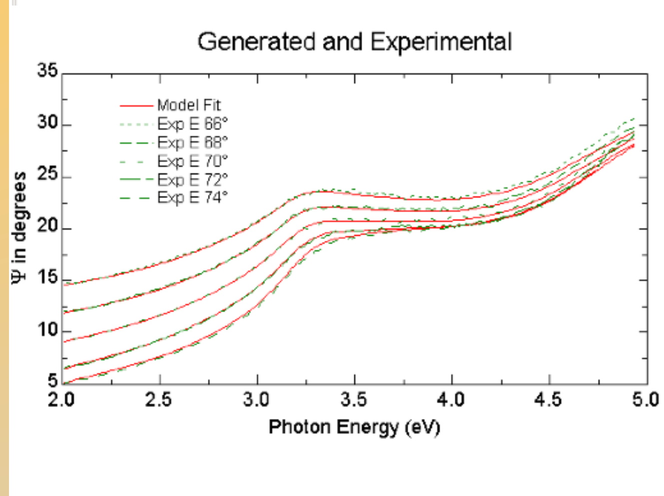


FeTMPyP(2) 4 day soak

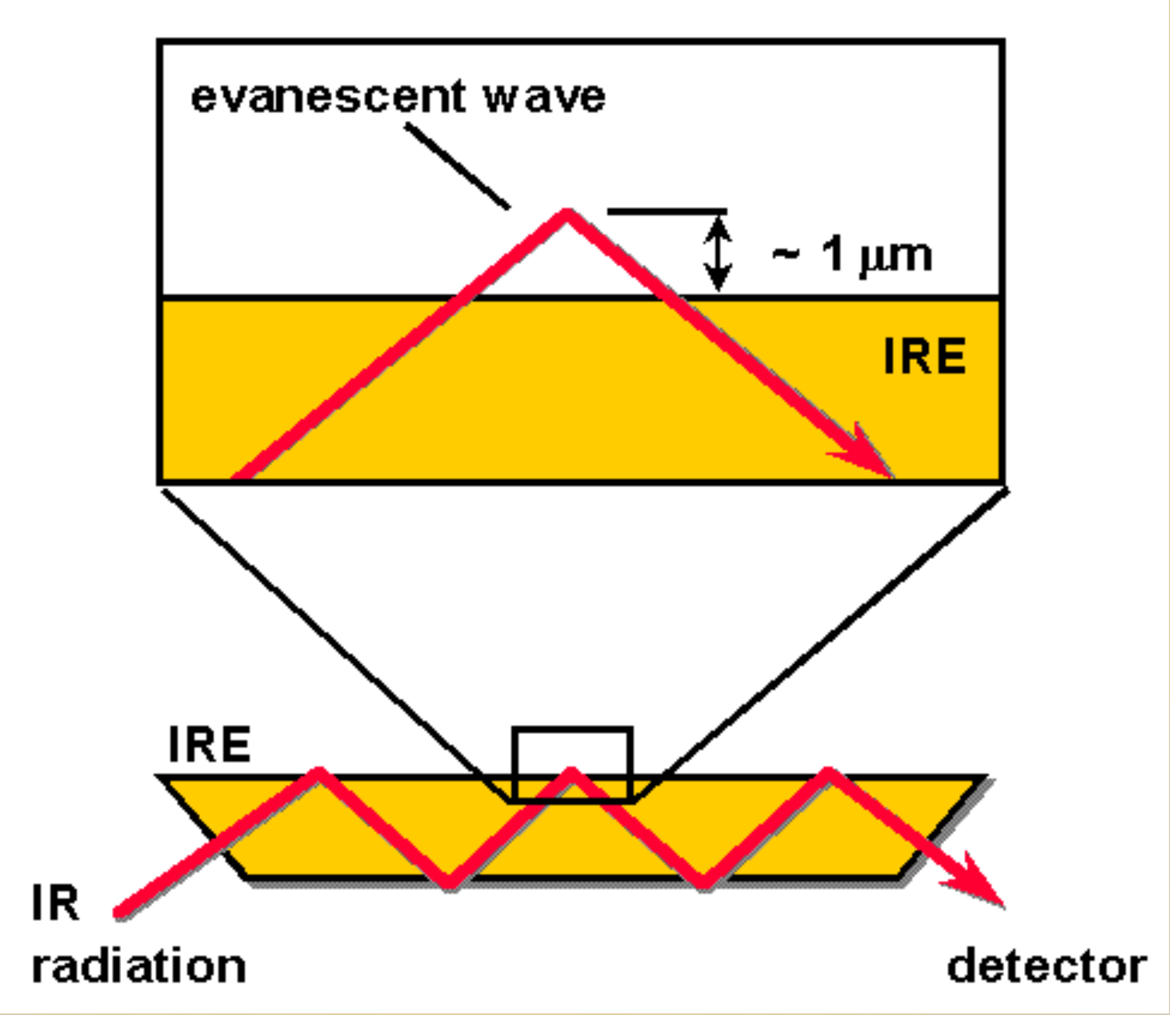




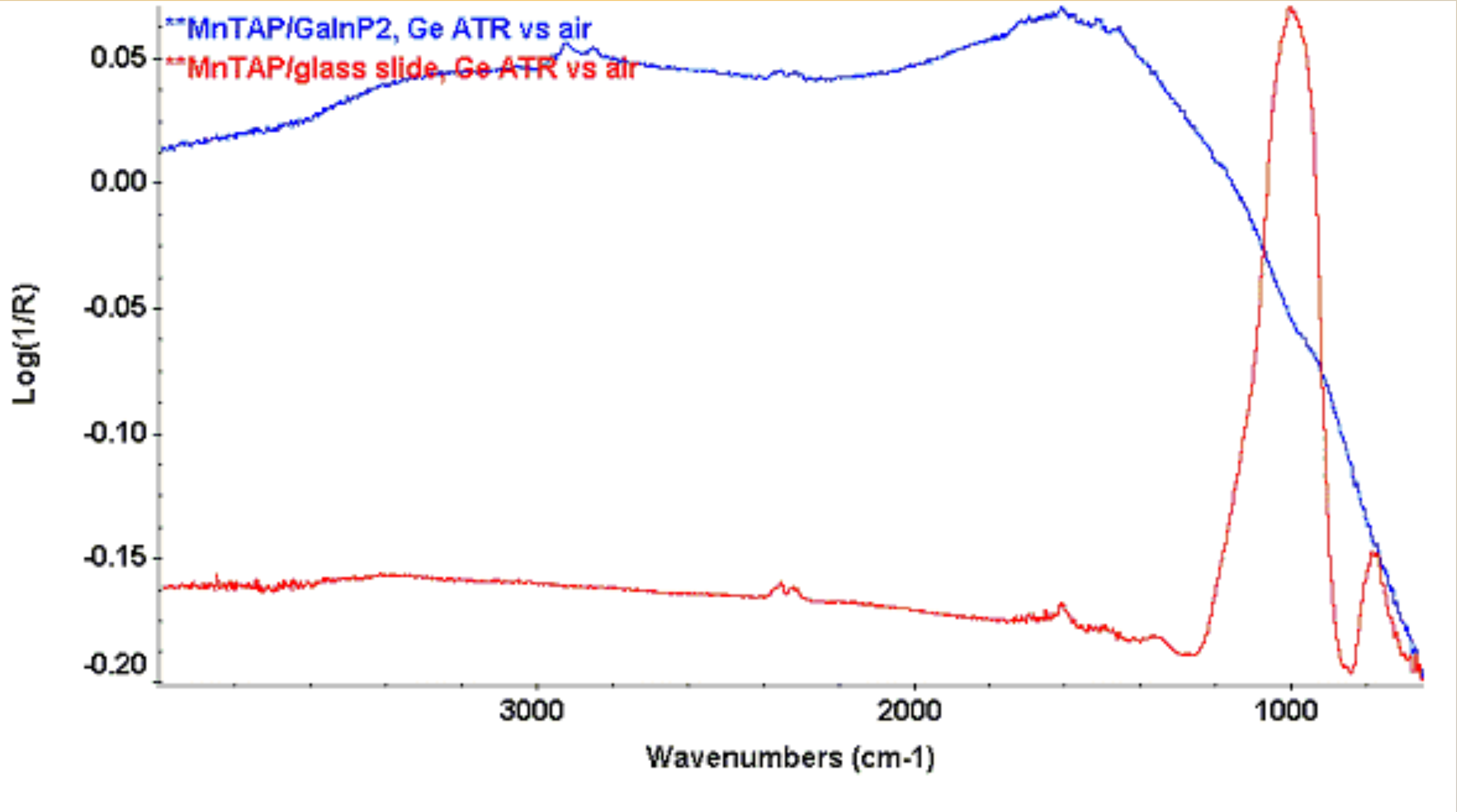




Attenuated Total Reflectance

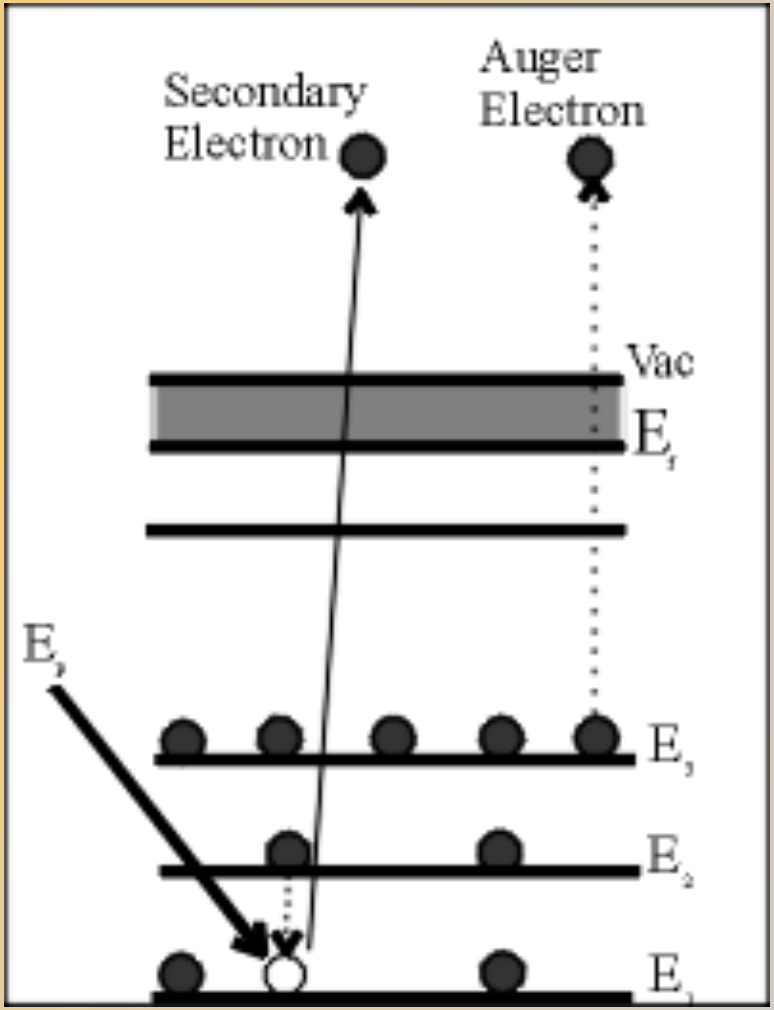


ATR spectrum of MnTAP



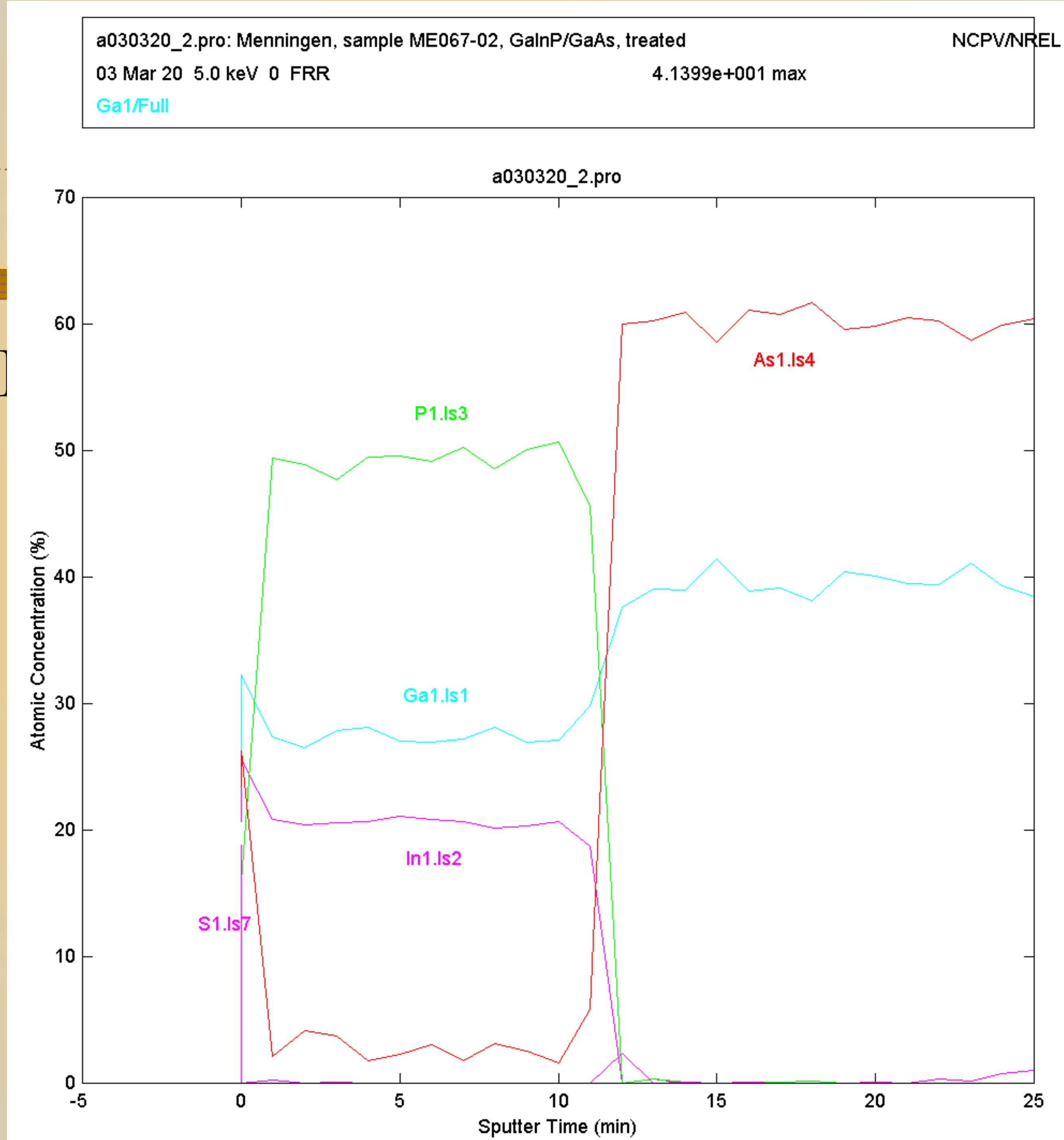
AES depth profiling

- 3 keV Ne^+ ion gun sputters into sample, then Auger Electron Spectroscopy is performed
- Composition as a function of depth for all elements >1% mole fraction



AES depth profile – ME067-2

- FeTMPyP(2) w/ 3<u>M</u> H_2SO_4, 1 week soak


a030320_2.pro: Menningen, sample ME067-02, GaInP/GaAs, treated
NCPV/NREL
03 Mar 20 5.0 keV 0 FRR
4.1399e+001 max
Ga1/Full
a030320_2.pro
Atomic Concentration (%)
Sputter Time (min)
P1.ls3
As1.ls4
Ga1.ls1
In1.ls2
S1.ls7
70
60
50
40
30
20
10
0
-5
0
5
10
15
20
25

What we learned from what didn't work:

- The porphyrin layer is probably very thin, likely 100 Å or less
- Acid etching does not seem to harm the sample very much
- Optical detection of the porphyrins is very challenging!

What we learned from what worked:

- Electrodes should soak in buffer before making Mott-Schottky measurements
- Mixing porphyrins amplifies the effect
- Results from RuOEP treatment highly dependent upon technique
- H_2SO_4 seems to prepare sites for porphyrin deposition
- Porphyrin treatments shift the band edges but do not facilitate charge transfer enough

Two Stories (Part II)

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- **Diamond growth by molecular beam**

Two Stories (Part II)

- **Diamond growth by molecular beam**
- **Single-crystal diamonds by CVD**

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- **Sing ... CVD**

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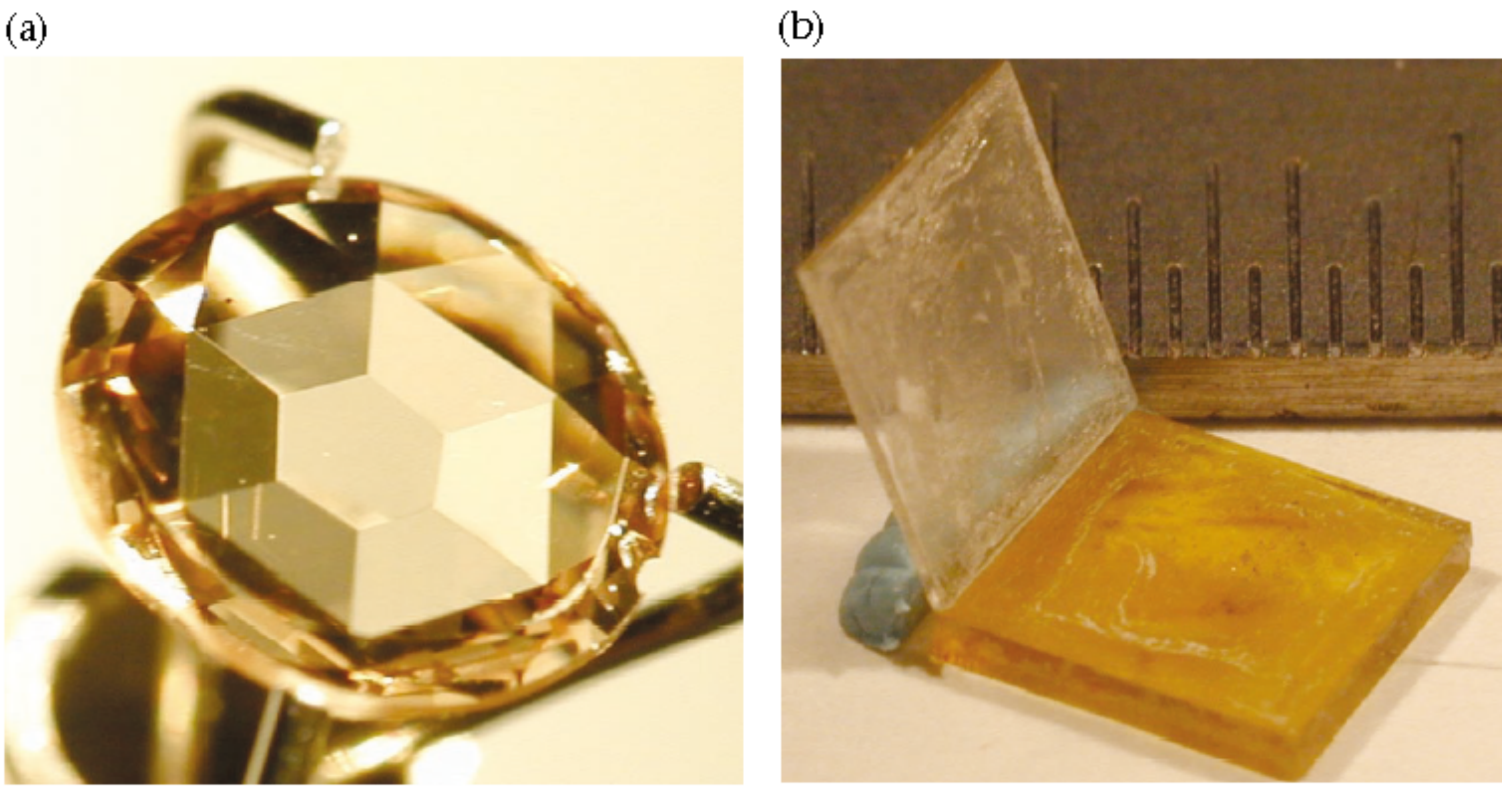

FIG. 1. Photographs of single crystal diamond grown by chemical vapor deposition (CVD) (Courtesy of Apollo Diamond): (a) a faceted stone ca. 3 mm diameter, in which single qubit operations for quantum computing have been demonstrated at room temperature using the N-V defect; and (b) a homoepitaxial layer ca. 4 x 4 x 1 mm (white/clear) after separation from the adjacent to the original HPHT seed crystal (yellow).

Fig. 1. Photographs o[f]
crystal diamond gr[own by]
chemical vapor dep[osition]
(CVD) (Courtesy of
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News

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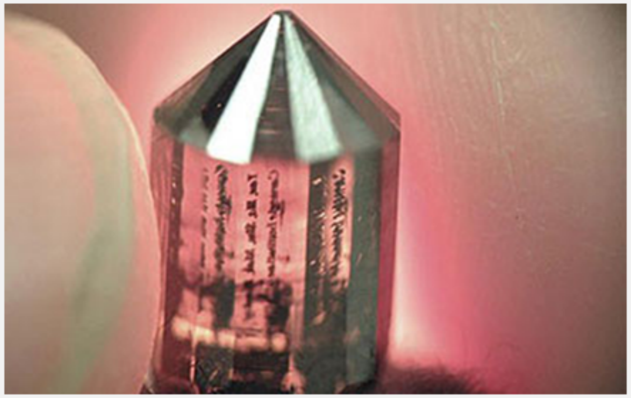
Press Release 05-080

New Technique Produces 10-carat Diamond

Crystal-clear material is better for optics, scientific applications

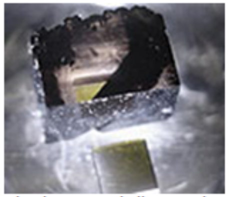


Five-carat diamond laser-cut from a 10-carat single crystal produced by high-growth rate CVD
Credit and Larger Version

May 16, 2005

Researchers at the Carnegie Institution of Washington, D.C. have produced 10-carat, half-inch thick single-crystal diamonds at rapid growth rates (100 micrometers per hour) using a chemical vapor deposition (CVD) process. The size is approximately five times that of commercially available diamonds produced by the standard high-pressure/high-temperature (HPHT) method and other CVD techniques.

In addition, the team has made colorless single-crystal diamonds, transparent from the ultraviolet to infrared wavelengths with their CVD process.

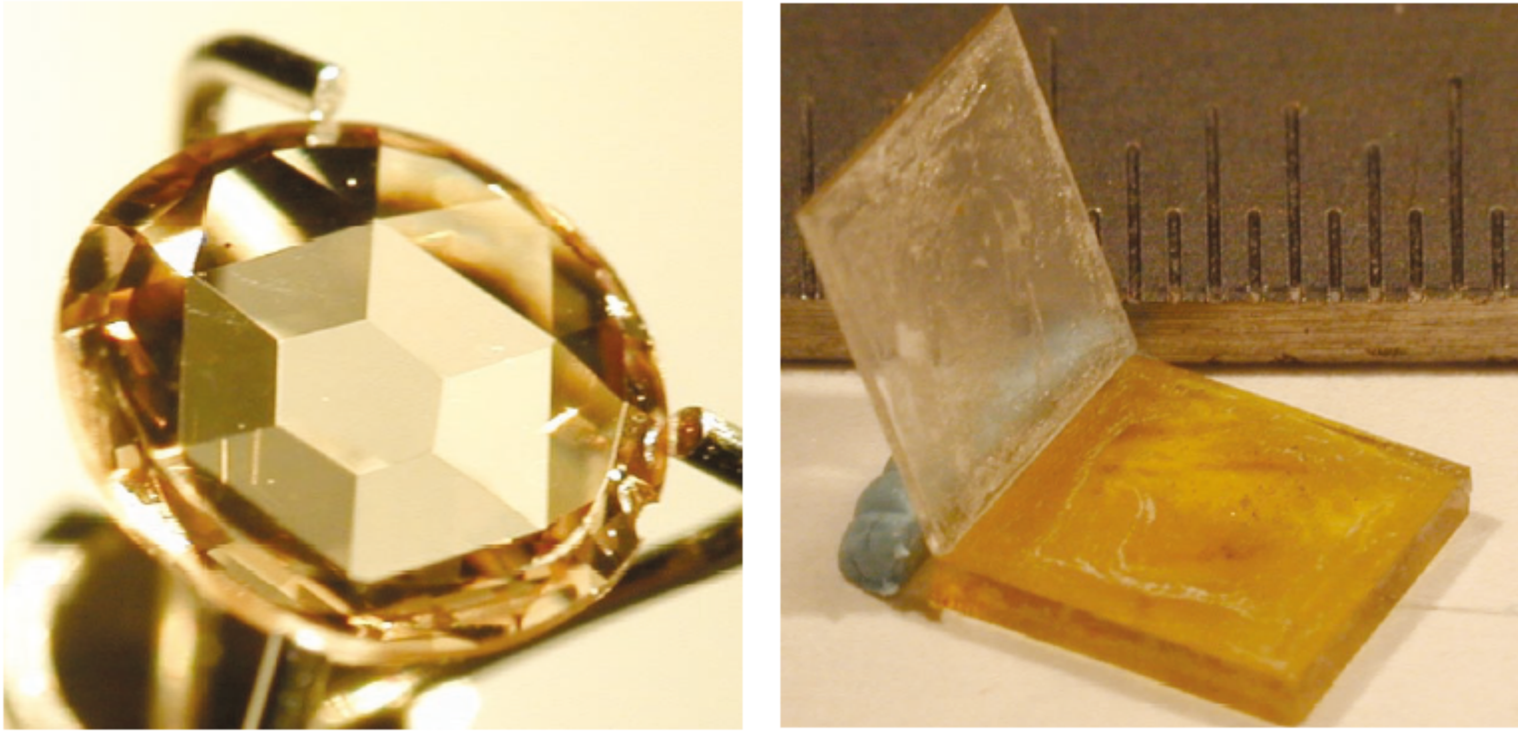


Single-crystal diamond block formed by CVD process
Credit and Larger Version

The CVD process can produce a variety of single crystal diamonds.
Credit and Larger Version

Two Si

(a) (b)



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Two S

- Dia lar
  bea
- Sing CVD

Two Stories (Part II)

- **Diamond growth by molecular beam**
- **Single-crystal diamonds by CVD**

What we've tried at UWSP

- Adding nanomagnets to the interface
- Use RuS_2 for anode
- Etching $GaInP_2$ to make it porous
- Adding nitrogen to the surface
- Building a dual photoelectrode system
- Explore Fe and W oxide semiconductors
- Explore Ni and Co oxide semiconductors
- Deposit $BiVO_4$ as protective coating

Photoelectrochemical water splitting for hydrogen production using combination of CIGS2 solar cell and RuO_2 photocatalyst

Neelkanth G. Dhere*, Anant H. Jahagirdar

Morphology and Strongly Enhanced Photoresponse of GaP Electrodes Made Porous by Anodic Etching

B. H. Erné, D. Vanmaekelbergh, and J. J. Kelly*

Debye Institute, University of Utrecht, 3508 TA Utrecht, the Netherlands

Journal of The Electrochemical Society, 155 (9) B903-B907 (2008)

Photoelectrochemical Characterization and Durability Analysis of GaInPN Epilayers

Todd G. Deutsch, Jeff L. Head, and John A. Turner

Significant Efficiency Increase in Self-Driven Photoelectrochemical Cell for Water Photoelectrolysis

R. C. Kainthla, B. Zelenay, and J. O'M. Bockris*

Hydrogen Research Center, Department of Chemistry, Texas A&M University, College Station, Texas 77843

J Solid State Electrochem (1998) 2: 170–175 © Springer-Verlag 1998

ORIGINAL PAPER

Mahmoud M. Khader · Mahmoud M. Saleh · Emad M. El-Naggar

Photoelectrochemical characteristics of ferric tungstate

Photocatalytic Water Splitting DOI: 10.1002/anie.201007987

Splitting Water with Cobalt

Vincent Artero,* Murielle Chavarot-Kerlidou, and Marc Fontecave*

ARTICLE

Received 6 Apr 2013 | Accepted 25 Jun 2013 | Published 29 Jul 2013 DOI: 10.1038/ncomms3195

Efficient solar water splitting by enhanced charge separation in a bismuth vanadate-silicon tandem photoelectrode

Fatwa F. Abdi[1], Lihao Han[2], Arno H.M. Smets[2], Miro Zeman[2], Bernard Dam[1] & Roel van de Krol[1,3]

What we've tried at UWSP

Electrochemical and Solid-State Letters, 9 (2) A43-A45 (2006) A43

Magnetized Nickel Electrodes for Improved Charge and Discharge Rates in Nickel Metal Hydride and Nickel Cadmium Batteries

Pengcheng Zou and Johna Leddy

Department of Chemistry, University of Iowa, Iowa City, Iowa 52242, USA

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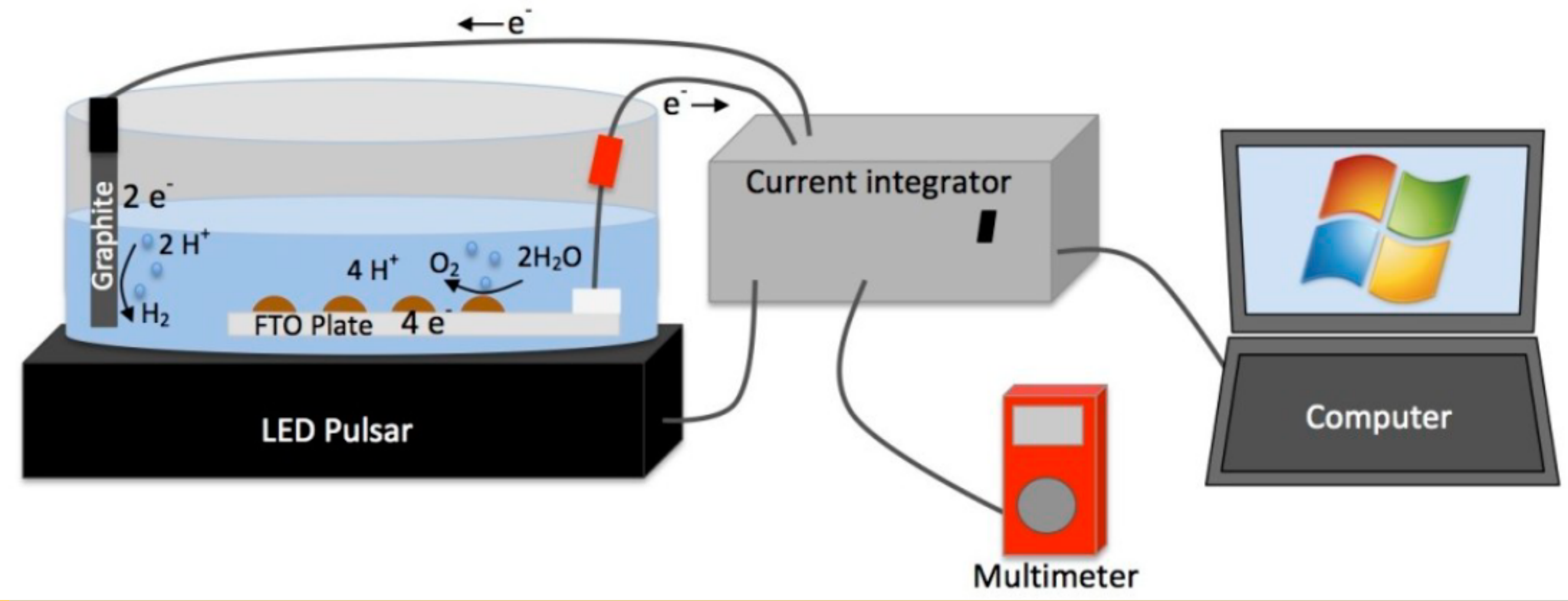
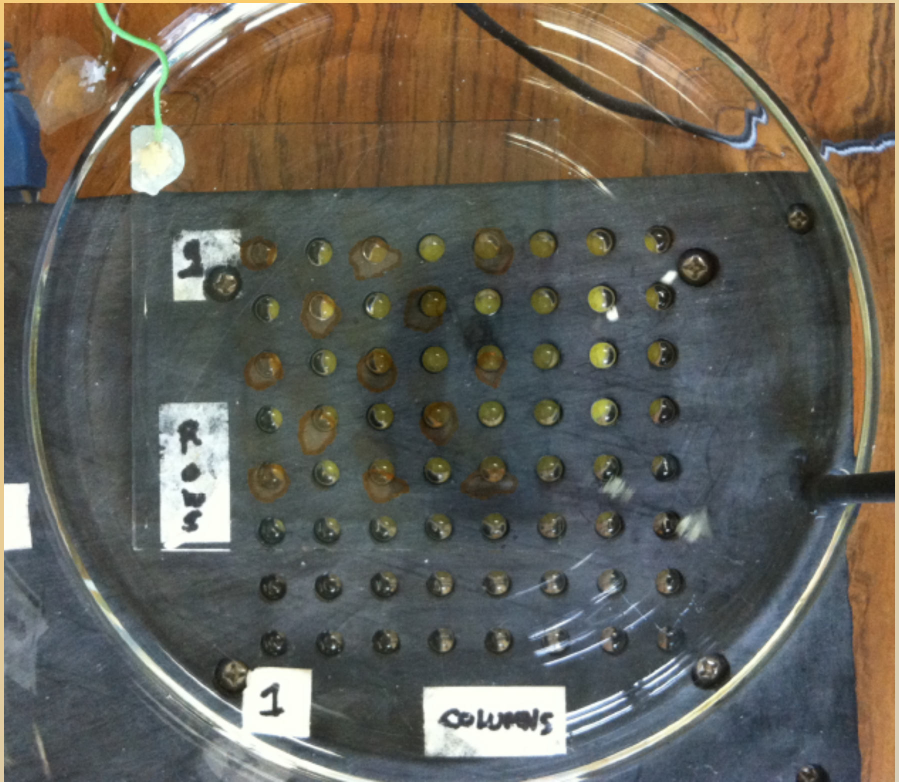
Solar Energy Activity Lab

- Goal is to quickly measure photocurrent from oxide semiconductors.

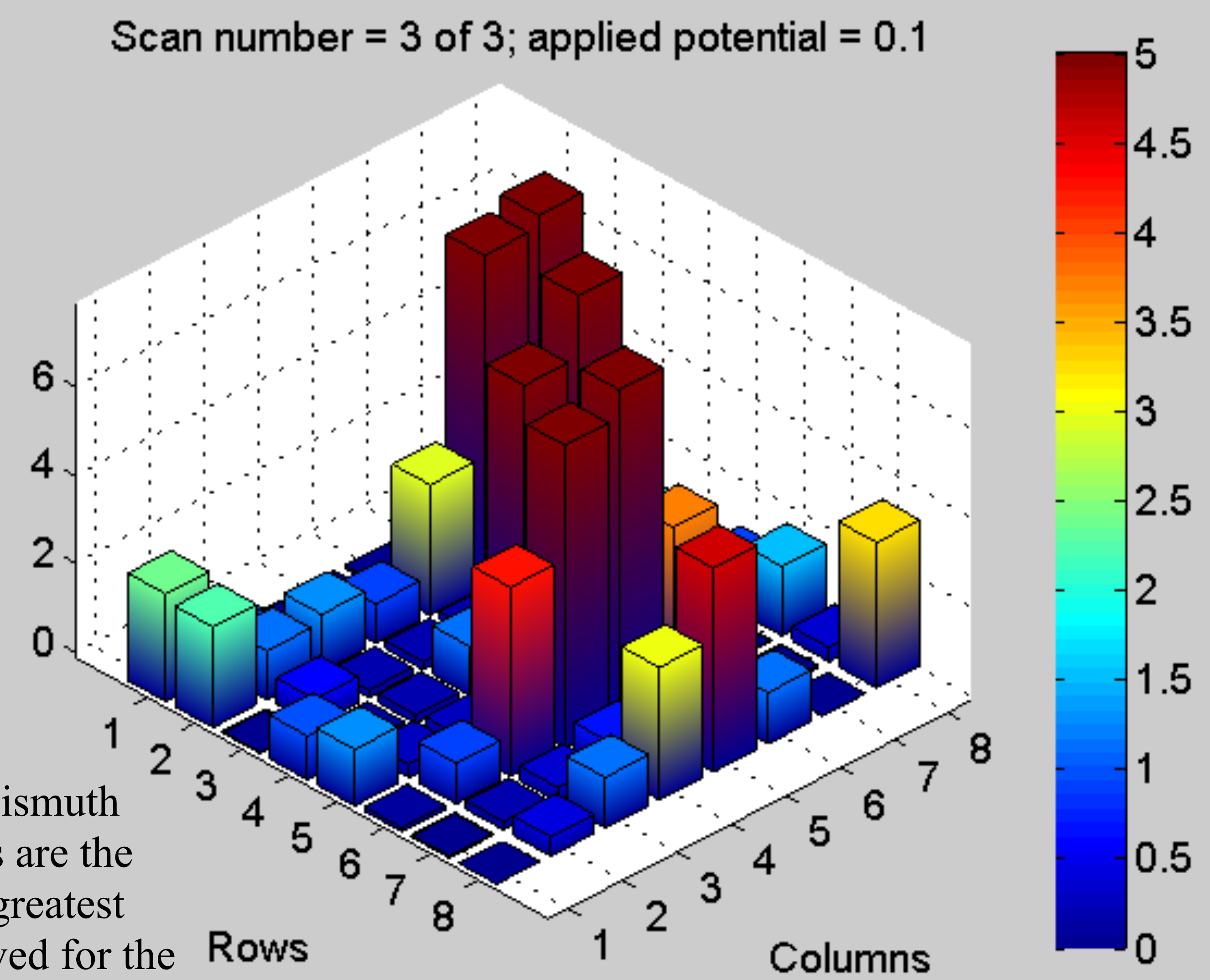


Spotted plate

- Each plate can hold up to 64 unique recipes
- Most have 15 recipes with redundant spots



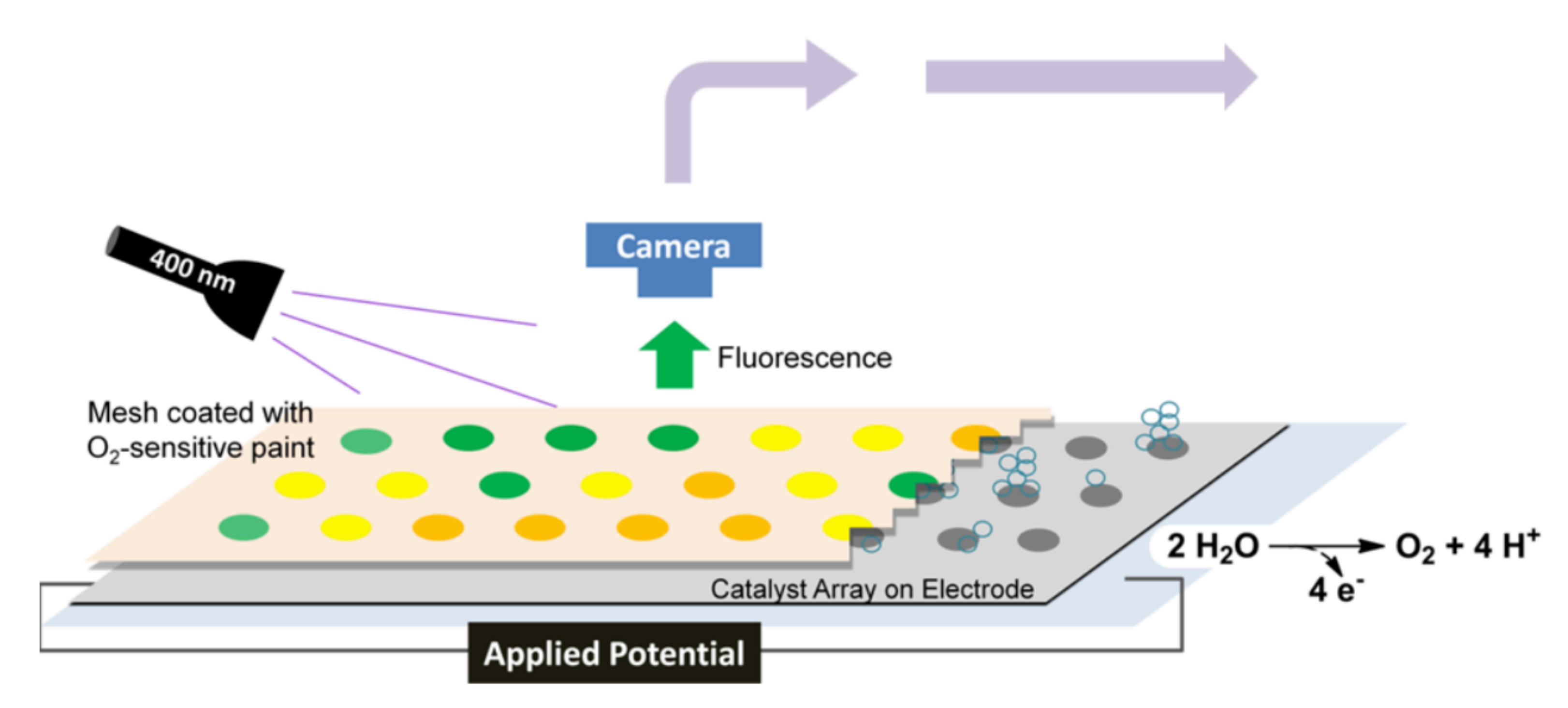
Bismuth Vanadate



Photocurrent scan of a bismuth vanadium plate. Corners are the reference spots and the greatest photocurrents are observed for the [1:9] and [2:9] **Bi:V** mixtures.

***H**eterogeneous **A**nodes **R**apidly **P**erused for **O**xygen **O**verpotential **N**eutralization*

- Goal is to quickly measure ability to catalyze oxygen production.

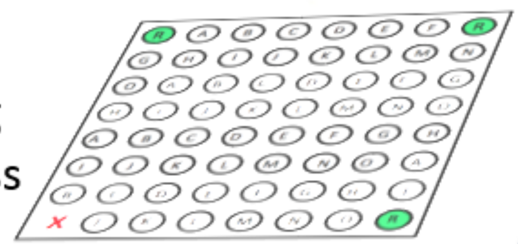


Spotting plates

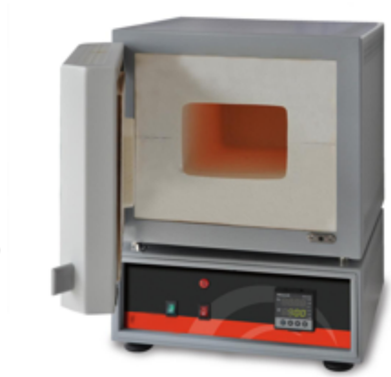
Experimental: Spotting Metal Oxide Plates

1. Aqueous metal nitrate solutions are individually prepared
   - HARPOON: 5 mM
   - SEAL: 40 mM

2. Varying ratios of metal nitrate solutions are pipetted onto a transparent conducting oxide-coated (FTO) glass substrate

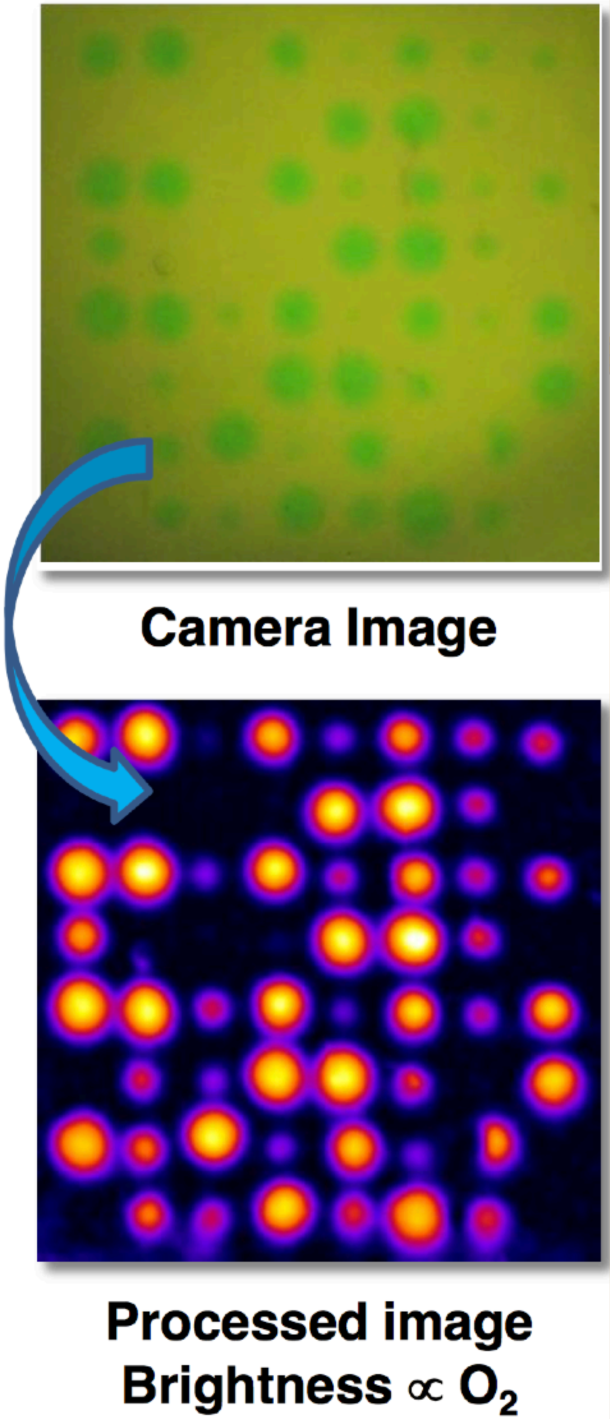


The template allows for up to 64 different metal oxide combinations to be tested!

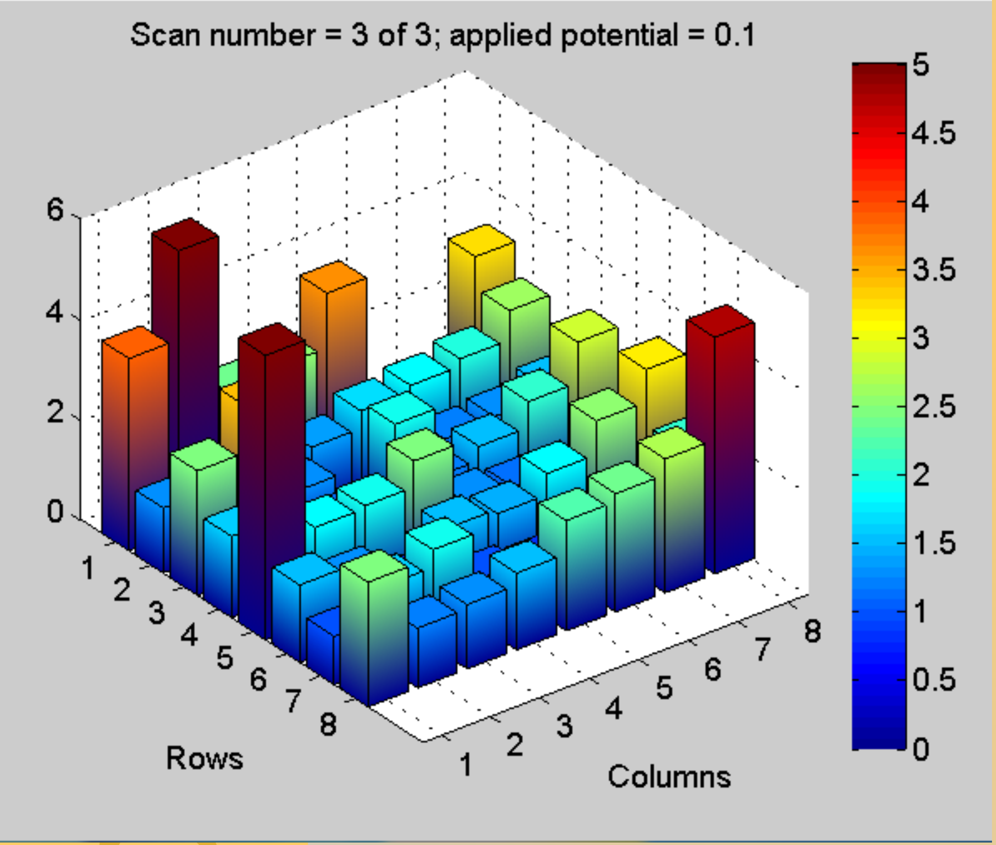


3. The FTO spotted plate is annealed at 500 °C for 6 hr in a furnace to convert the salts to oxides

HARPOON scan



Fe:Bi plate



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6
4
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8
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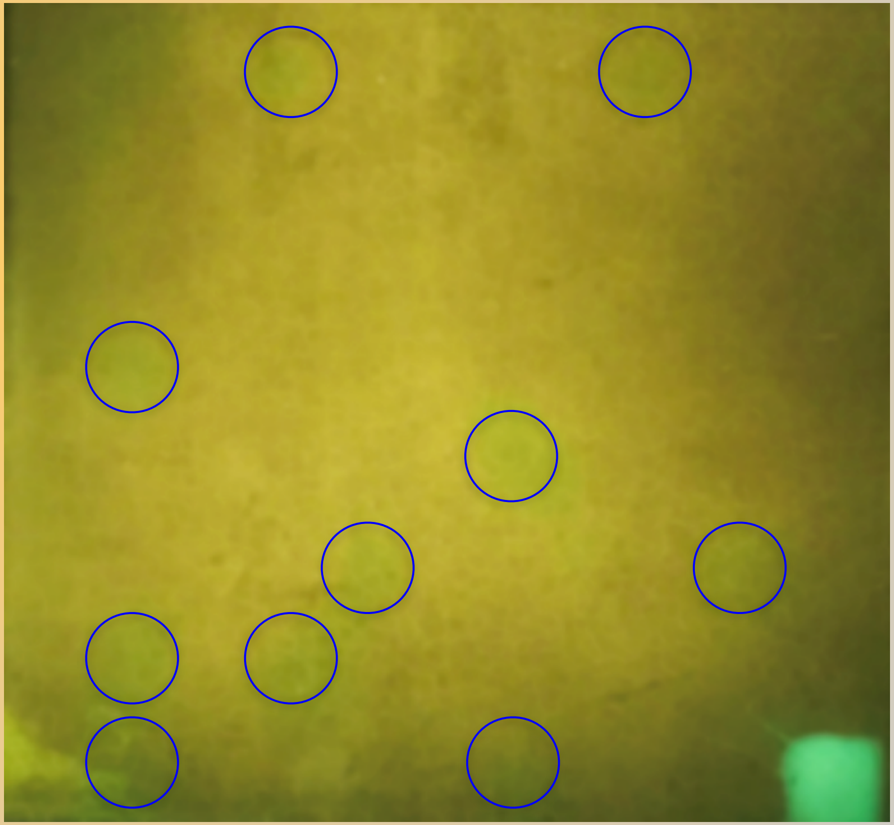
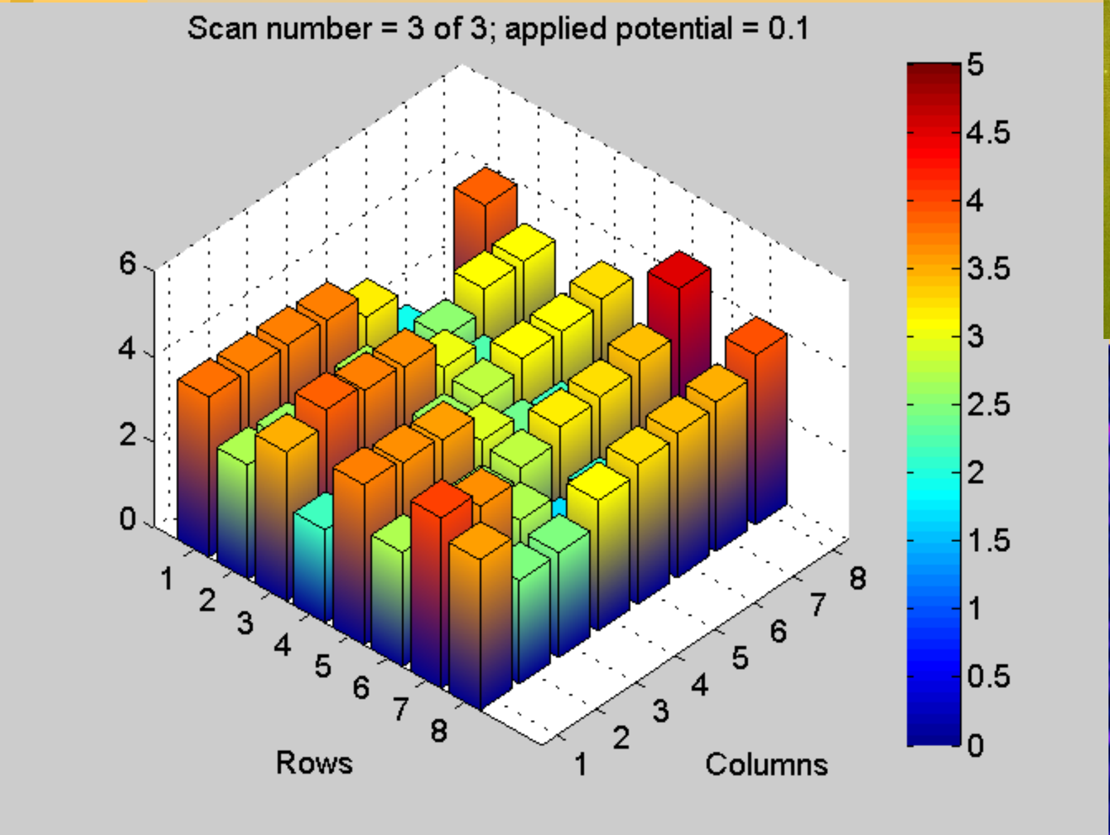
0.5
0



Fe:Cr plate


Scan number = 3 of 3; applied potential = 0.1
6
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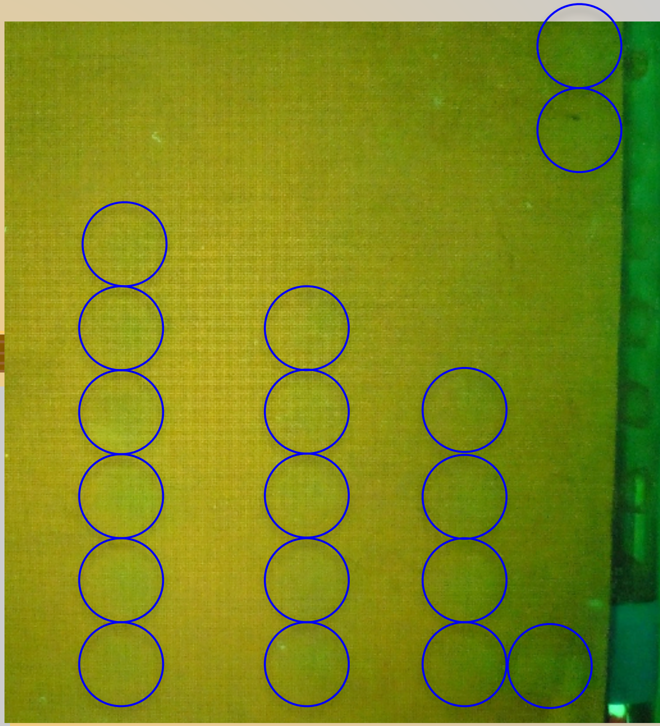

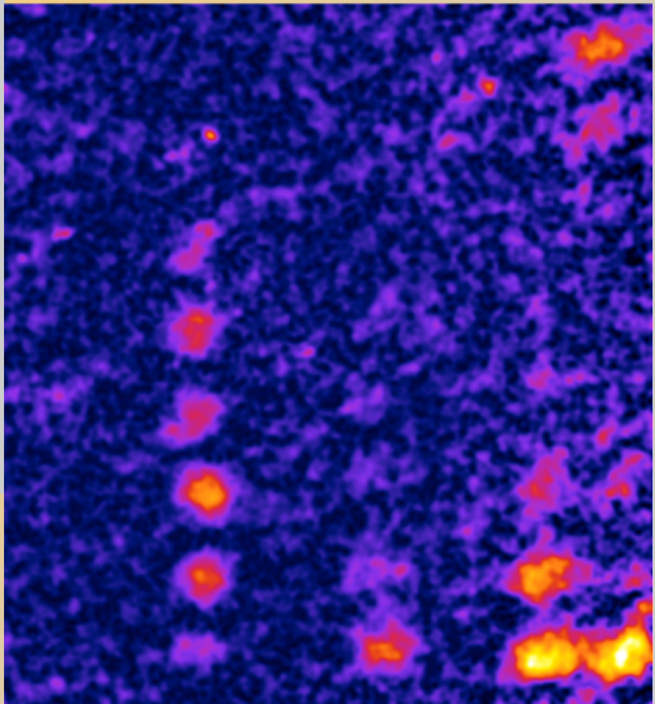

What should be done next

- Improve consistency between the two measurement techniques
- Quantify the overall suitability of candidates
- Fabricate semiconductor nanowires from the best candidates

What I learned from Jim

- Work steadily to collect good data but also keep a venturesome spirit
- Develop and sustain collaborations
- Cultivate a broad array of interests