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International Centre for Theoretical Physics

United Nations
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International Atomic
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World Year of PHYSICS 2005

Workshop on "Physics for Renewable Energy"
October 17 - 29, 2005

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"Photoelectrochemical & Biological Production of Hydrogen & Storage of Hydrogen of Carbon Nanotubles "

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A national laboratory of the U.S. Department of Energy
Office of Energy Efficiency & Renewable Energy

National Renewable Energy Laboratory

Innovation for Our Energy Future

Photoelectrochemical and Biological Production of Hydrogen and Storage of Hydrogen by Carbon Nanotubes

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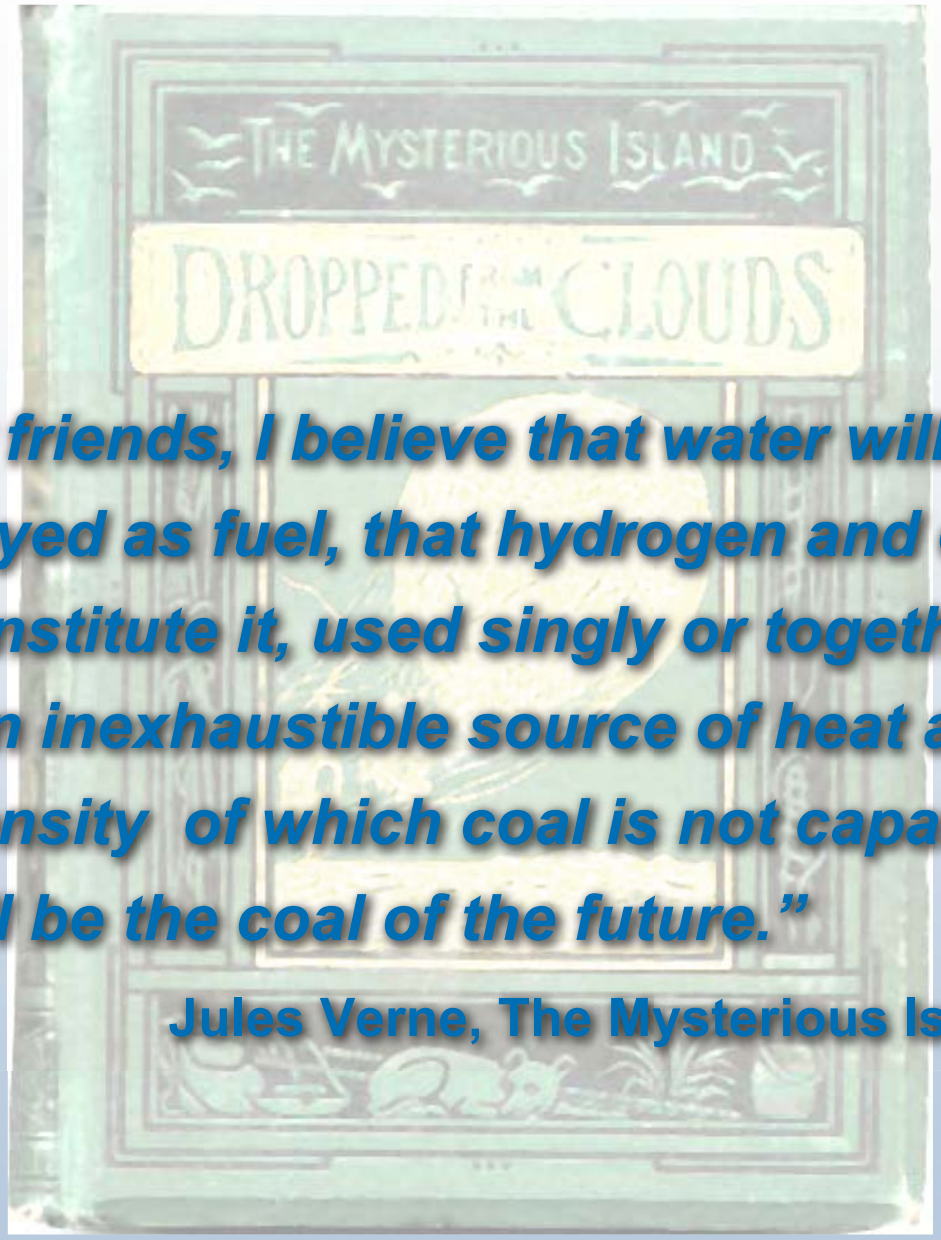
Golden, CO 80401

Presented at the Workshop on Physics for Energy Sources
International Center for Theoretical Physics (ICTP)
Trieste, Italy
October 17–29, 2005



"Yes, my friends, I believe that water will one day be employed as fuel, that hydrogen and oxygen which constitute it, used singly or together, will furnish an inexhaustible source of heat and light, of an intensity of which coal is not capable… Water will be the coal of the future."

Jules Verne, The Mysterious Island (1874)

Hydrogen: A National Initiative

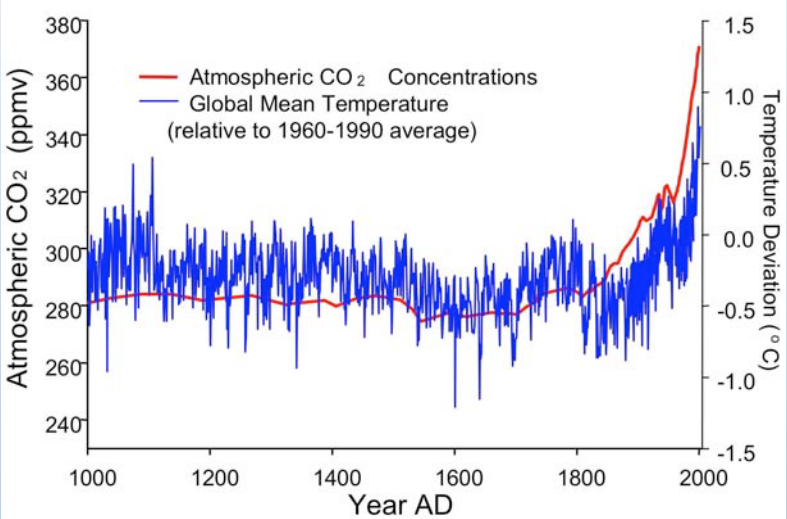
"Tonight I'm proposing $1.2 billion in research funding so that America can lead the world in developing clean, hydrogen-powered automobiles… With a new national commitment, our scientists and engineers will overcome obstacles to taking these cars from laboratory to showroom, so that the first car driven by a child born today could be powered by hydrogen, and pollution-free."

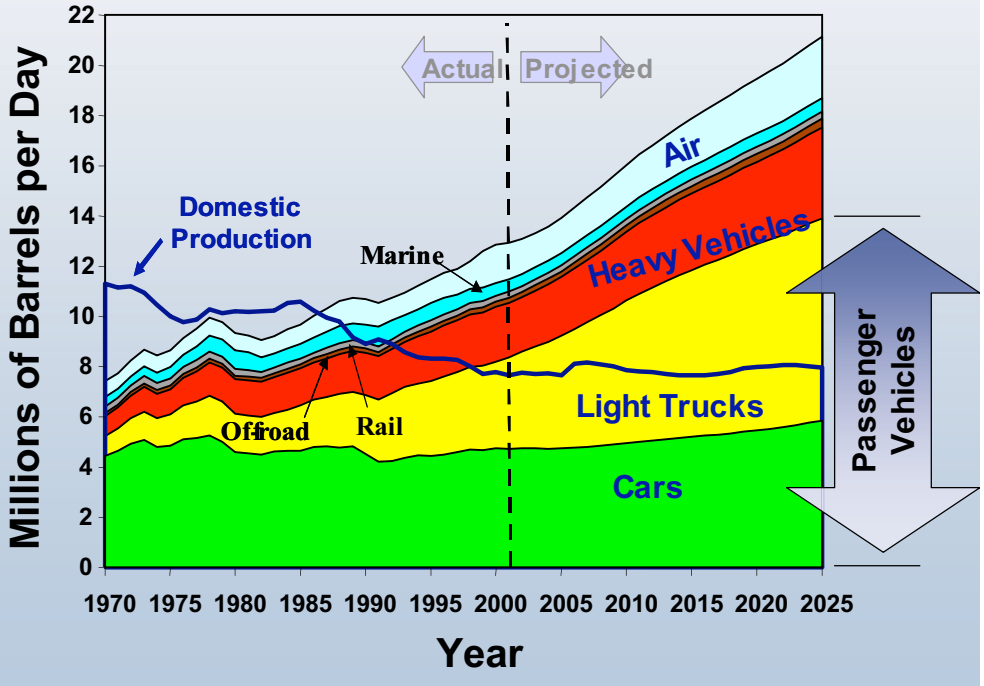
President Bush, State-of the-Union Address, January 28, 2003

Drivers for the Hydrogen Economy:

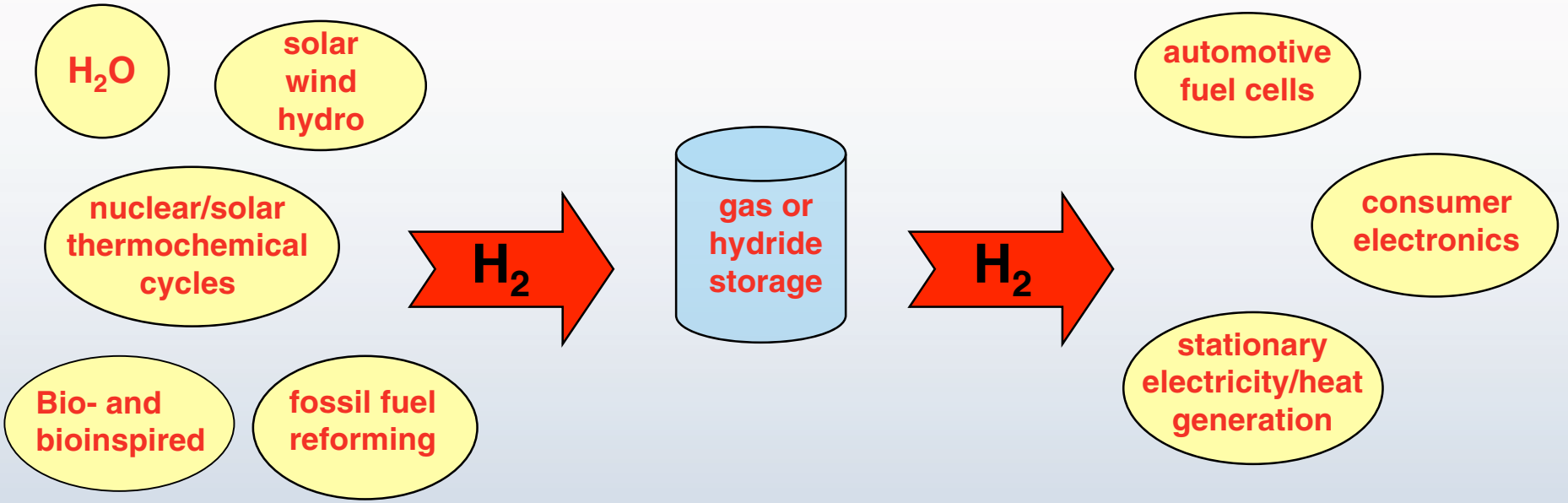
- ***Reduce Reliance on Fossil Fuels***
- ***Reduce Accumulation of Greenhouse Gases***

Energy Source	% of U.S. Electricity Supply	% of Total U.S. Energy Supply
Oil	3	39
Natural Gas	15	23
Coal	51	22
Nuclear	20	8
Hydroelectric	8	4
Biomass	1	3
Other Renewables	1	1





The Hydrogen Economy



production	***storage***	***use in fuel cells***
9M tons/yr	**4.4 MJ/L (Gas, 10,000 psi)** **8.4 MJ/L (LH_2)**	**$3000/kW**
40M tons/yr **(Transportation only)**	**9.72 MJ/L** **(2015 FreedomCAR Target)**	**$35/kW** **(Internal Combustion Engine)**

The Hydrogen Economy

The production of hydrogen, primarily from water but also from other feedstocks, its distribution and utilization as an energy carrier.

Electricity Generation

- Fossil fuels
- Biomass
- Nuclear
- Geothermal
- Renewable e-
  - Solar
  - Wind
  - Hydro

Production

- Electrolysis
- Thermolysis
- Conversion

Feedstock

- Water
- Fossil fuels
- Biomass

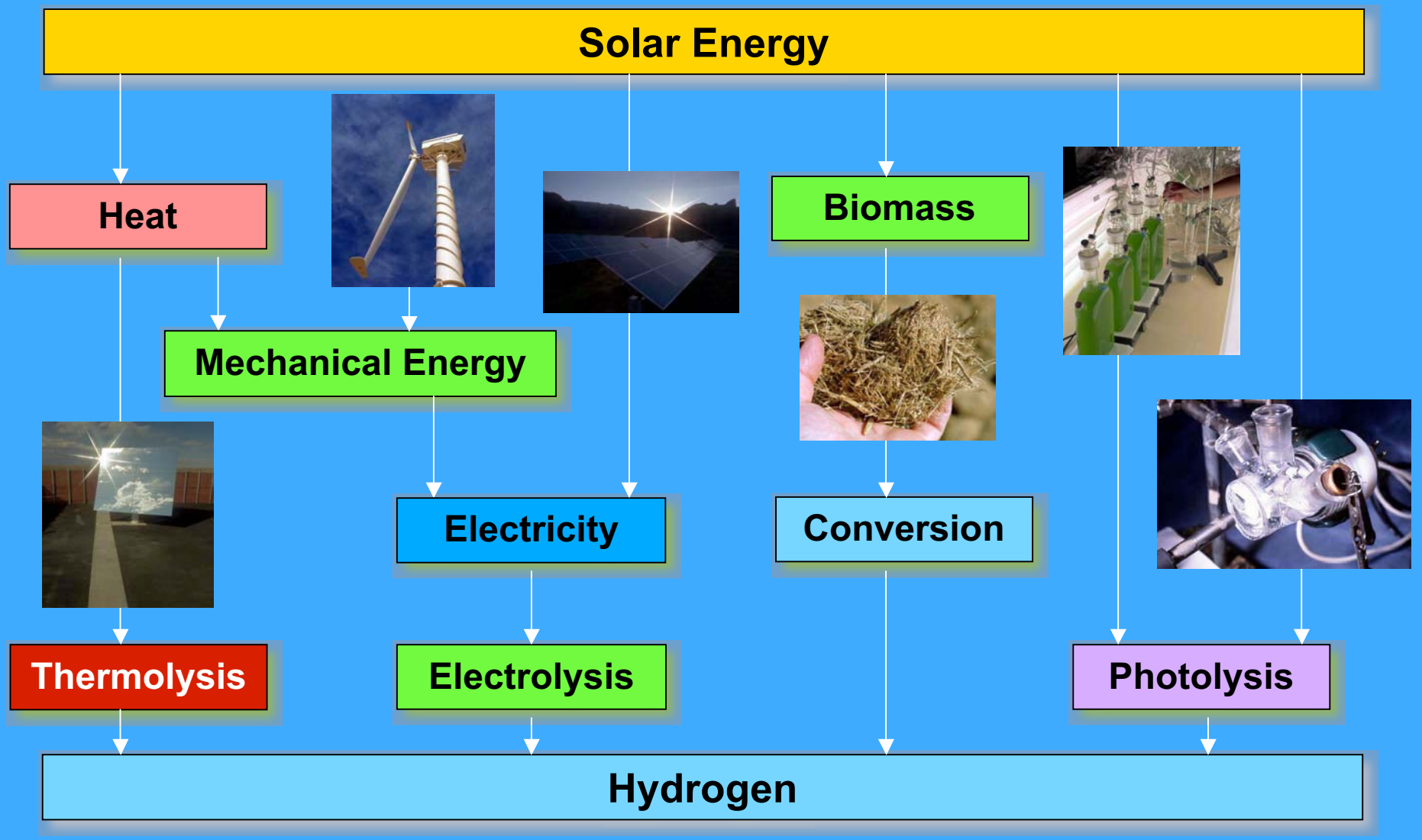
Distribution

- Used onsite
- Pipelines
- Compressed gas
- Liquid

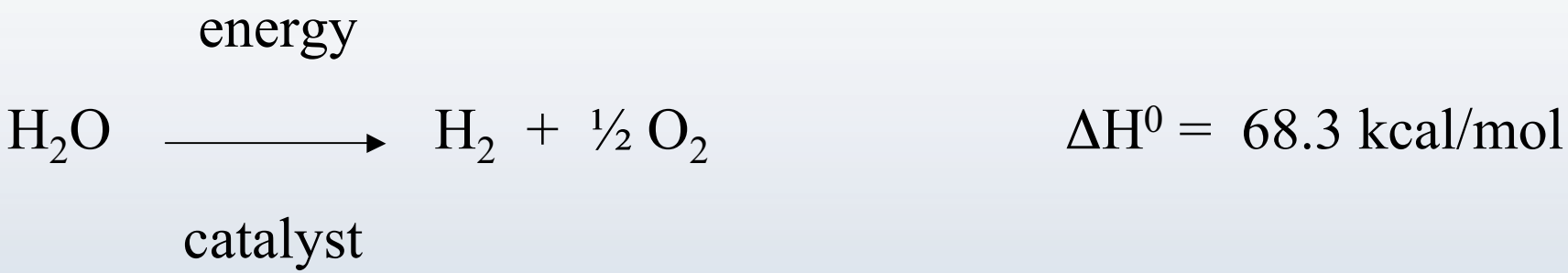
Utilization

- Fuel cells
- Turbines
- IC Engines

Sustainable Paths to Hydrogen



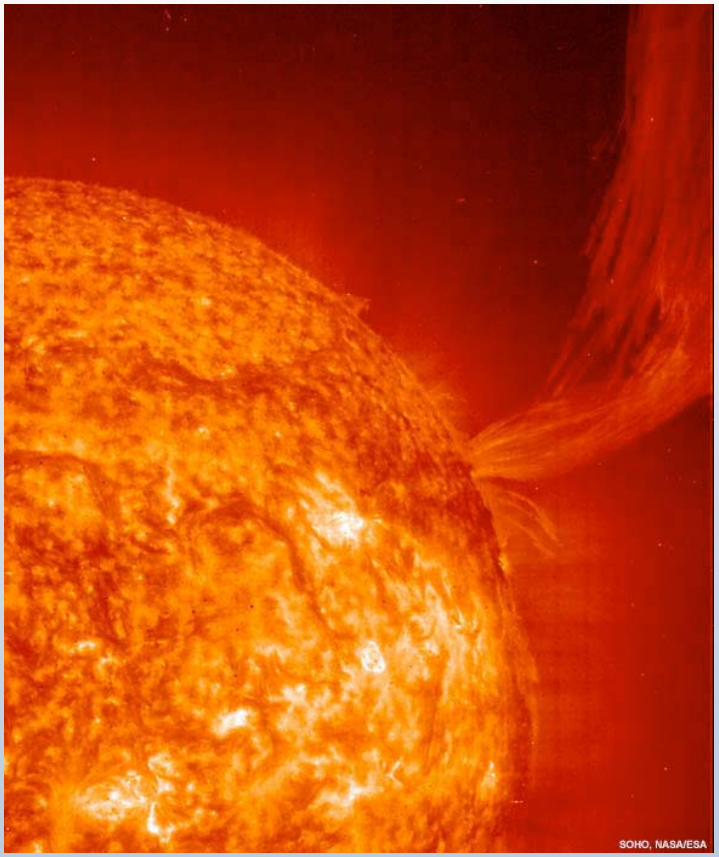
Hydrogen:
A Future Energy Carrier



- **High energy content on a weight basis**
- **If from a renewable source:**
  - **Displaces need for fossil fuels**
  - **Non-poluting**
  - **Carbon neutral**
  - **Addresses global change issues**
  - **Enhances energy security**

Direct Conversion Systems

Combination of a Light Harvesting System and a Water Splitting System



- Semiconductor photoelectrolysis
- Photobiological Systems
- Homogeneous water splitting
- Heterogeneous water splitting
- Thermal cycles

(Sunlight and Water to Hydrogen with No External Electron Flow)

Energy Conversion Strategies

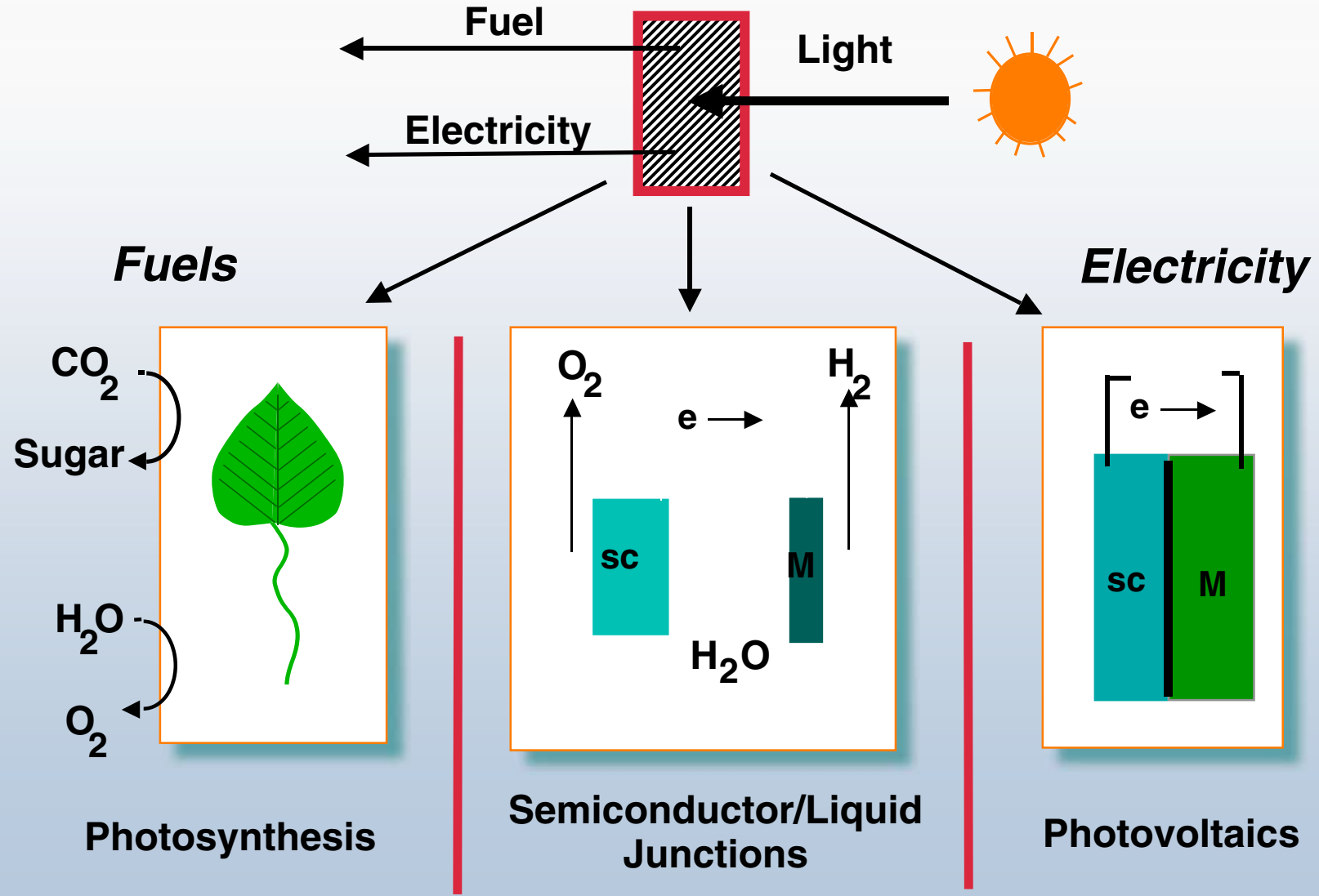
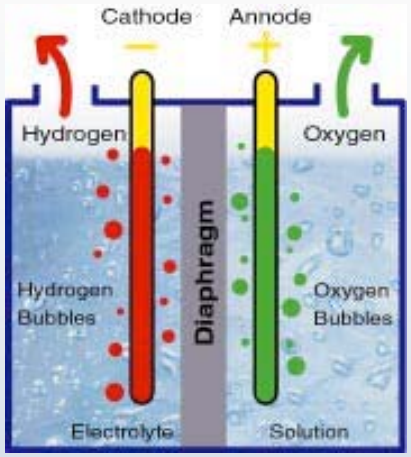


Photo-Electrochemical Production of Hydrogen from Water

Efficiency Considerations



- Energy efficiency of electrolysis for PEC comparison (LHV) =

$$\frac{\text{Chemical potential of } H_2}{\text{Electrolysis potential } (@\ 1A/cm^2)} = \frac{1.23\ V}{1.9\ V} = 65\%$$

- Coupling to a 12% PV array gives a solar-to-hydrogen efficiency of:

.12*.65 = 7.8%

Hydrogen from Visible Light and Water

- Visible light has enough energy to split water (H_2O) into hydrogen (H_2) and oxygen (O_2).
  - *Fortunately water is transparent and does not absorb this energy.*
- The combination of a light harvesting system and a water splitting system is necessary to be able to use sunlight to split water.
- Photoelectrochemical processes along with certain algae can use this light to produce hydrogen from water.

$$2H_2O \xrightarrow{\text{Visible Light}} 2H_2 + O_2$$

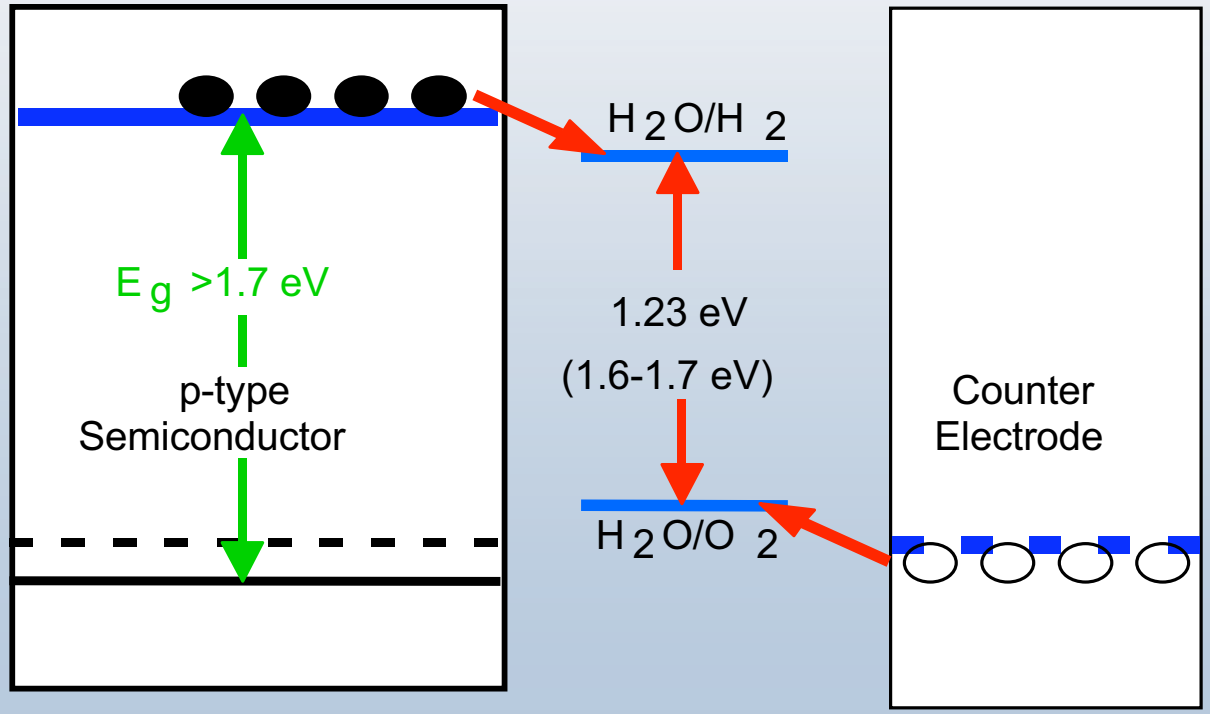
Photoelectrochemical Conversion Material Requirements

Bandgap

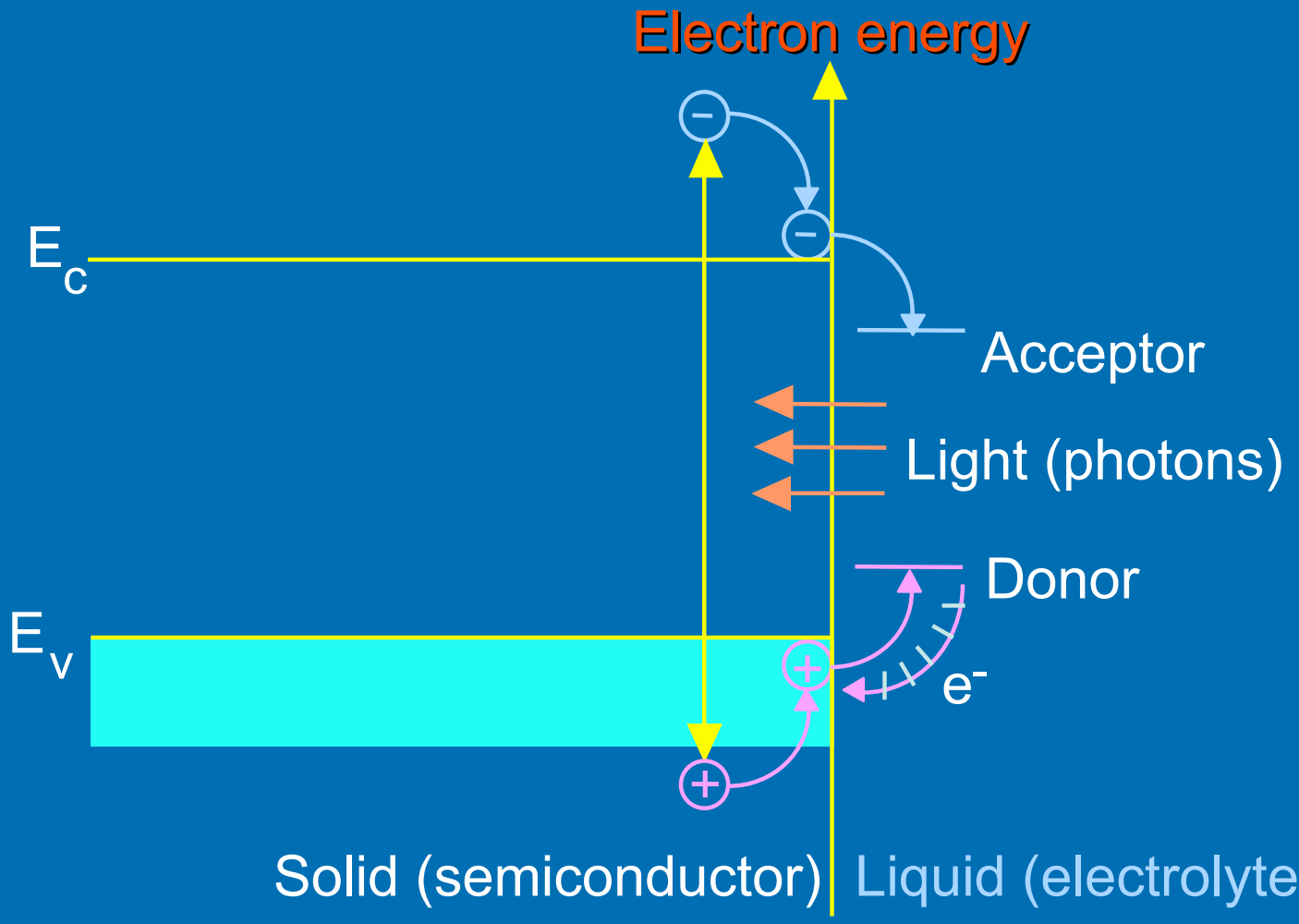
Band edge overlap

Fast charge transfer

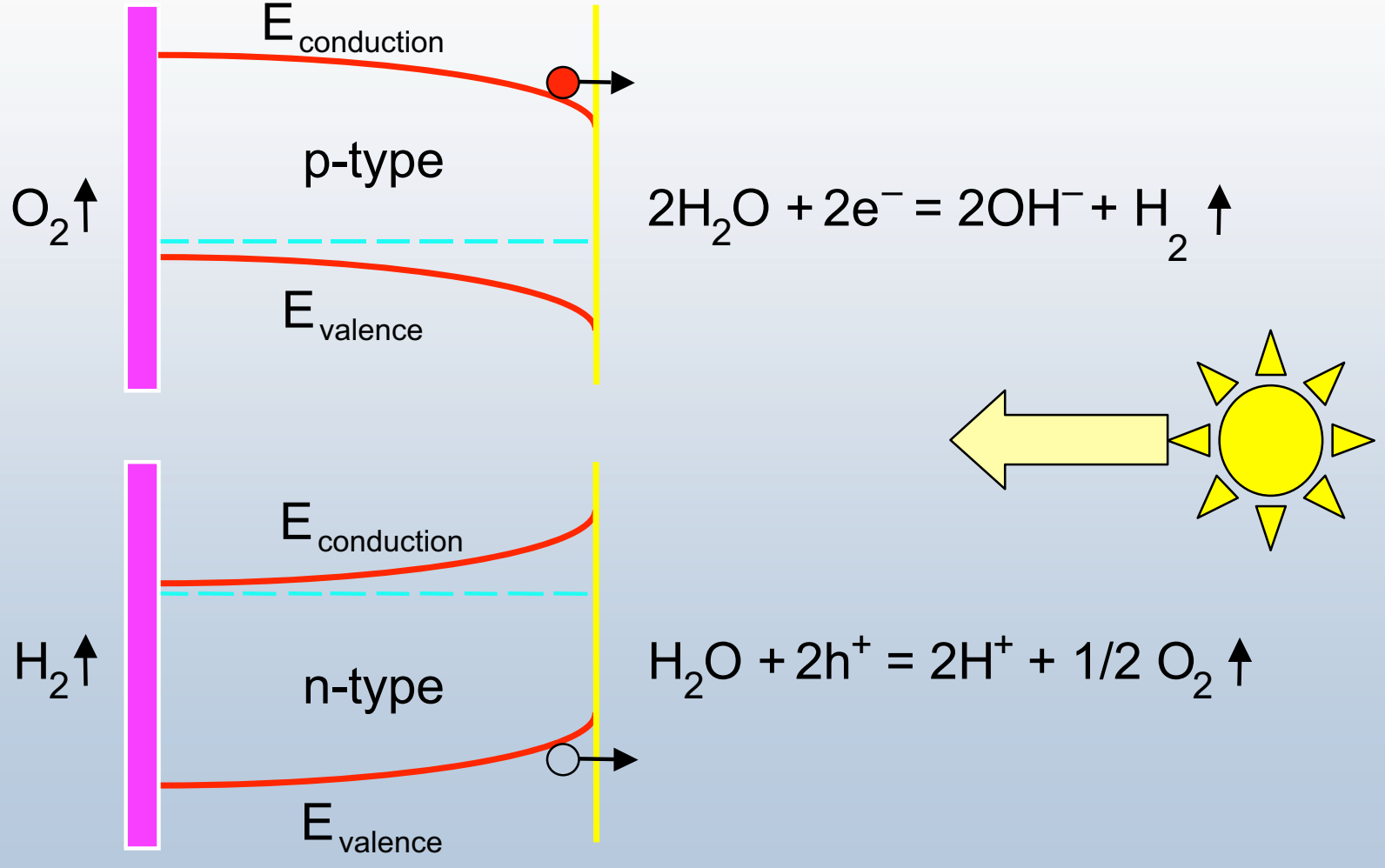
All three conditions must be satisfied SIMULTANEOUSLY



Chemical Reactions at a Semiconductor Electrolyte Interphase

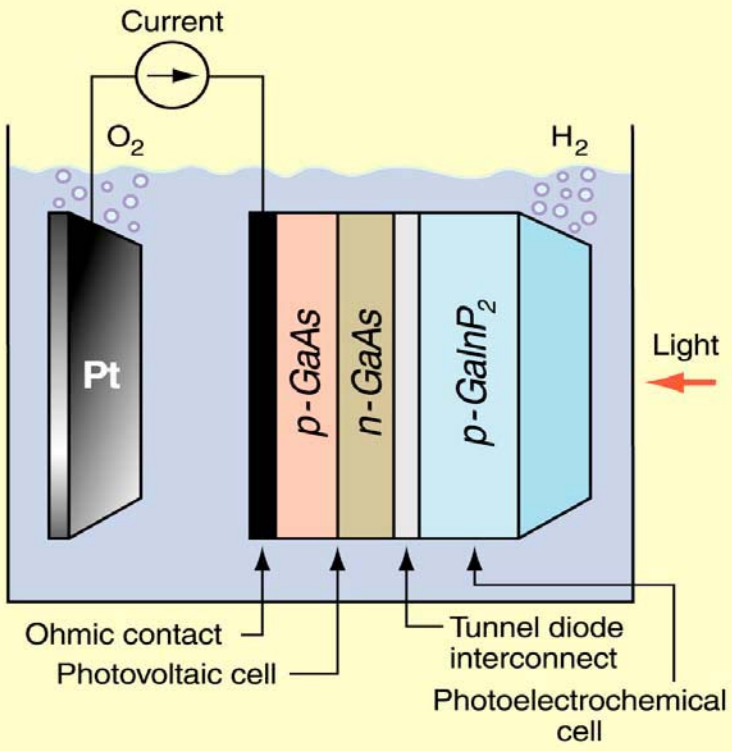


Band Edges of p- and n-Type Semiconductors Immersed in Aqueous Electrolytes to Form Liquid Junctions



PV/PEC Photoelectrolysis Device

Novel cell uses light to produce H_2 at 12.4% efficiency



Note: *n* and *p* refer to *n*- and *p*-type semiconductors
Credit: Adapted with permission from Science, copyright 1996 AAAS

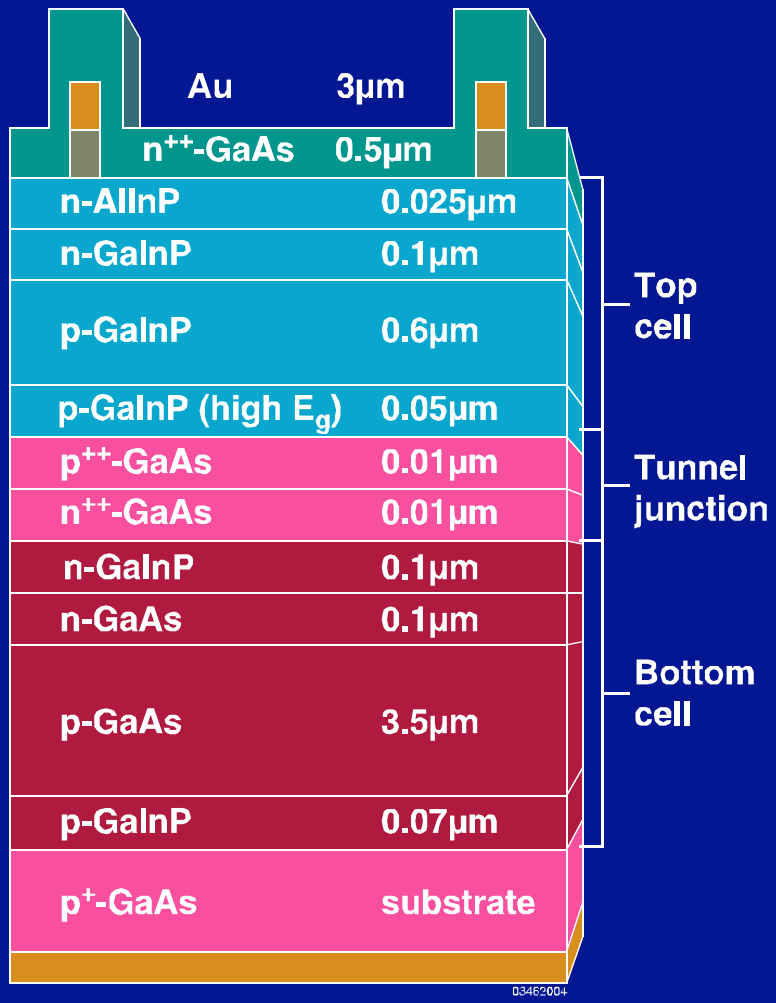
- ✓Direct water electrolysis.
- ✓Unique tandem (PV/PEC) design.
- ✓12.4% Solar-to-hydrogen

Experimental Cell

World Record Efficiency Solar Cell

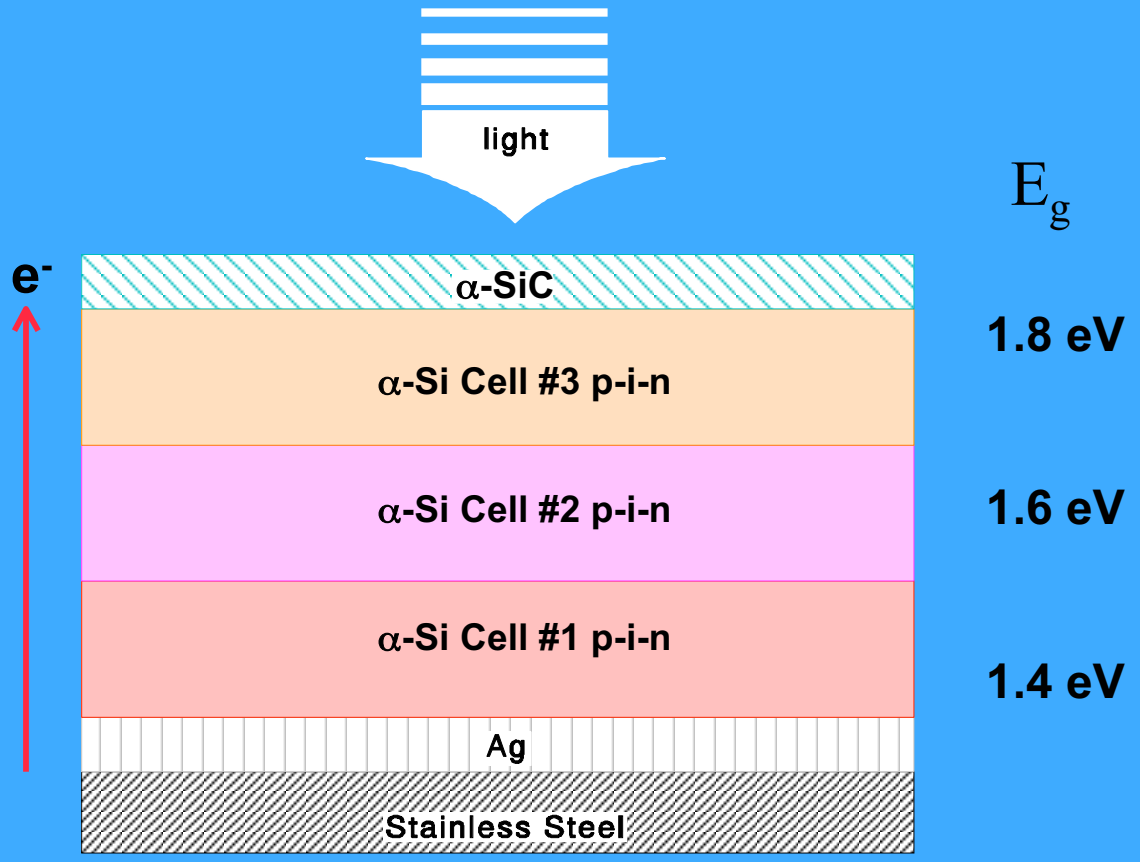
Several World Record Efficiencies:

- 29.5% 1 sun flat plate cell
- 30.2% 140-180 sun concentrator cell
- 25.7% 1 sun space cell



Gallium Indium Phosphide/Gallium Arsenide Photovoltaic Solar Cell

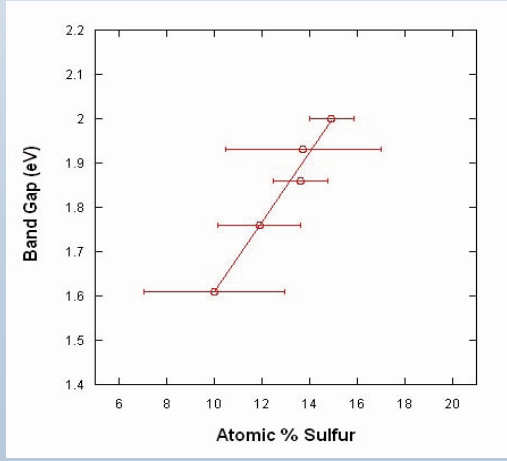
Amorphous Silicon Triple Cell
(reverse design for water splitting)



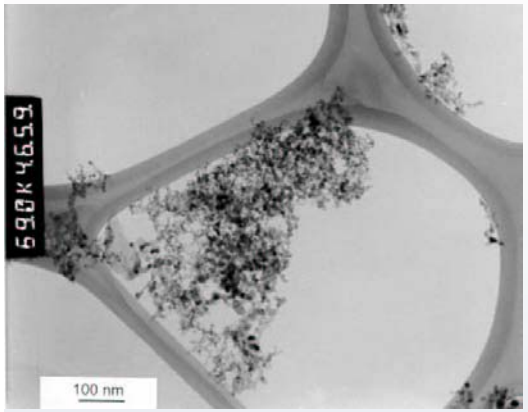
High Efficiency Semiconductor Materials

- III-V materials have the highest PV efficiency of any semiconductor material.
  - Largest range of available bandgaps
  - ….but
    - Unstable in aqueous solution (exception nitrides)
    - Band-edge mismatch (exception nitrides).

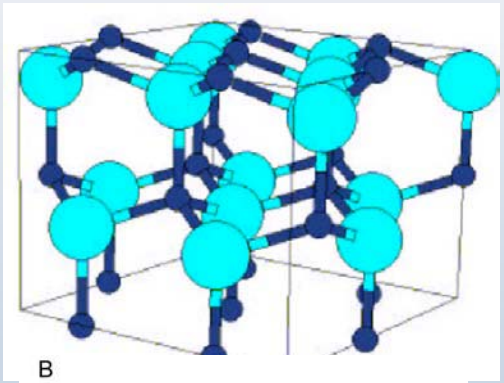
- I-III-VI materials offer low-cost manufacturing.
  - Synthesis procedures for desired bandgap unknown.
  - ….but
    - Unstable in aqueous solution?
    - Band-edge mismatch?

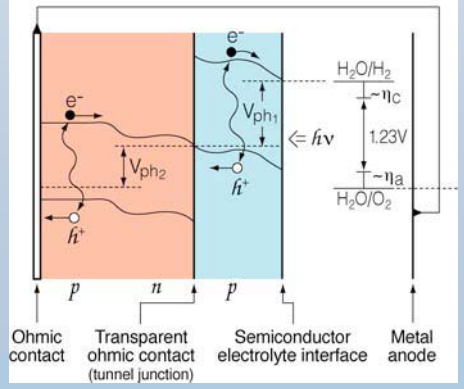


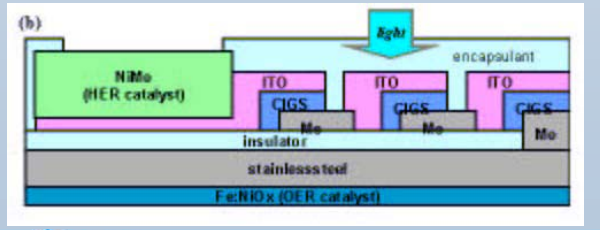
Current Areas of Effort



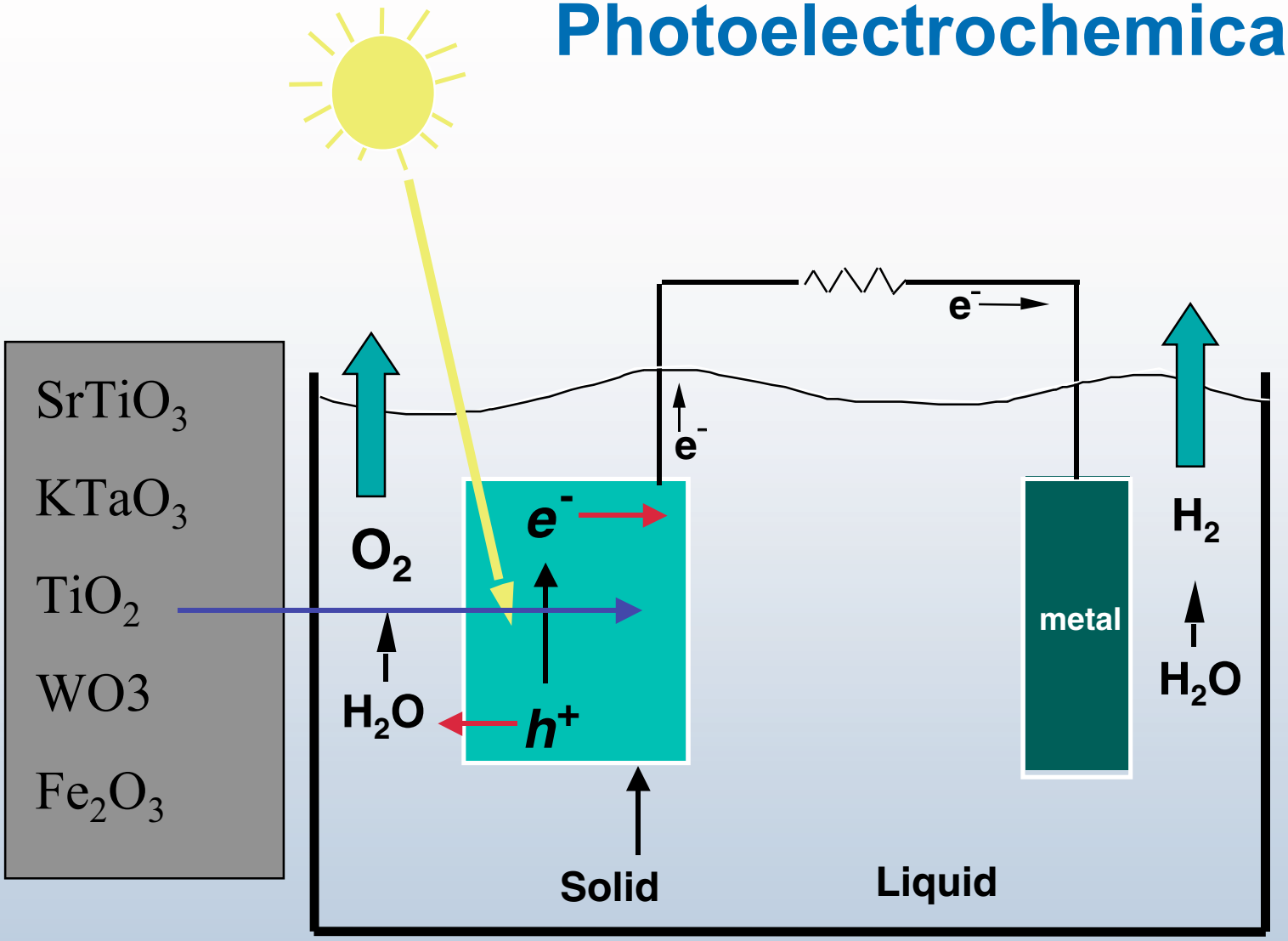
- Metal oxides (mixed and single).
  - Most studied area
  - Largest possibility of materials
  - Greatest stability, lowest efficiency to date
- Novel and new materials
  - Typically from the PV industry
  - PV materials are not always directly applicable to PEC systems, so some modifications needed
- Advanced structures/hybrid designs
  - Tandem cells, triple junctions, p-n combinations.
  - Specialty designs
- Characterization Tools
- Catalysts







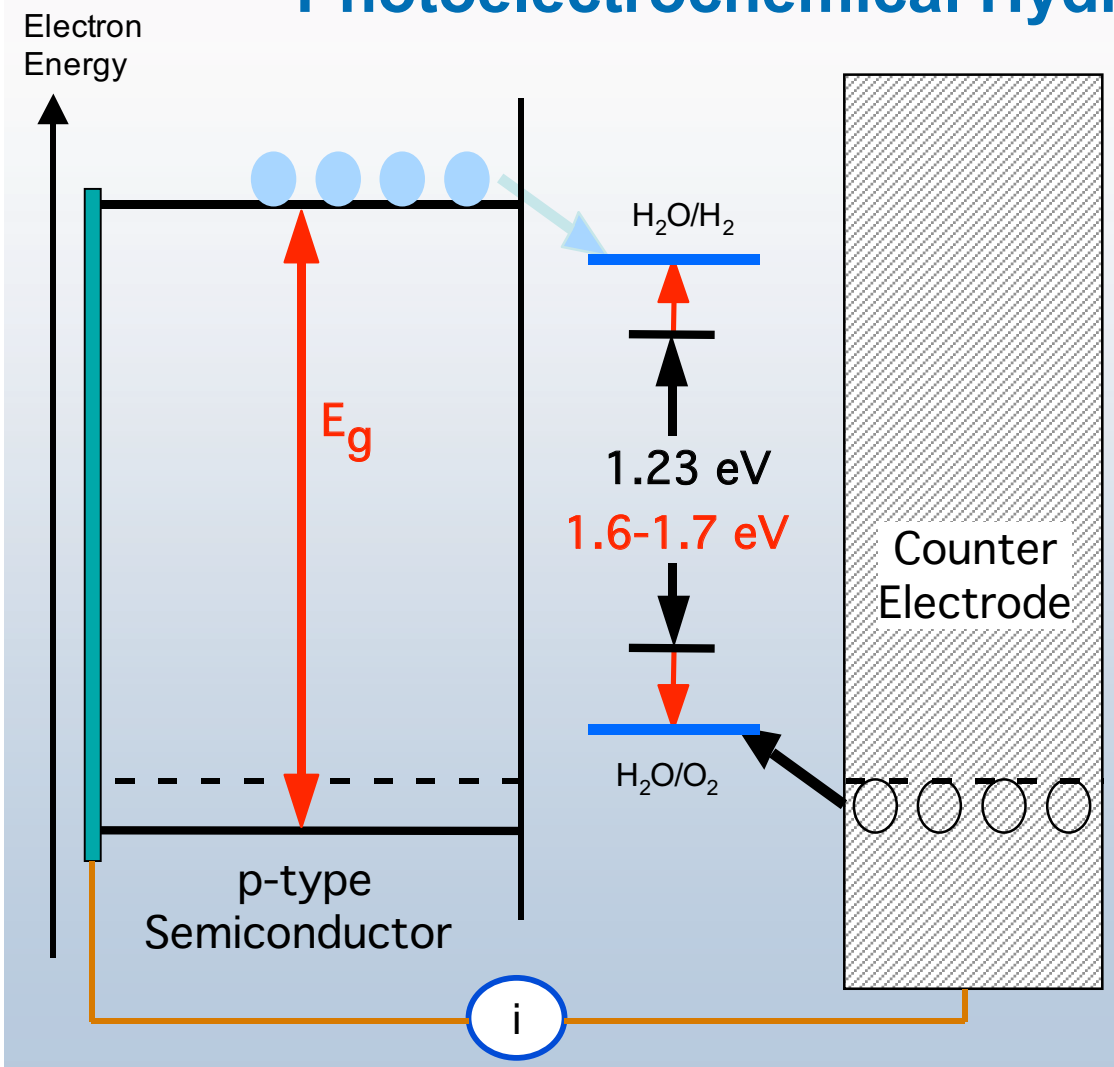
Photoelectrochemical Cell



Light is Converted to Electrical+Chemical Energy

Technical Challenges *(the big three)*

Material Characteristics for Photoelectrochemical Hydrogen Production

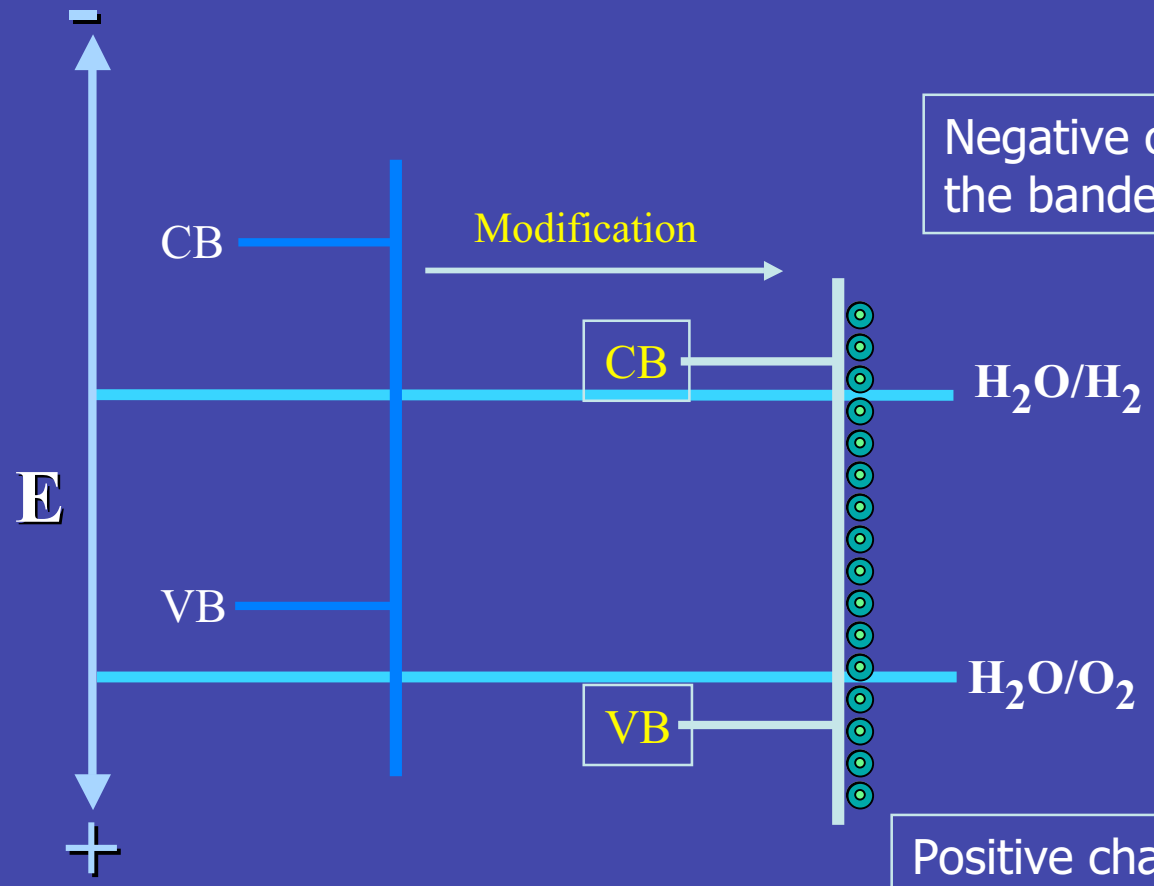


- **Material Durability –** semiconductor must be stable in aqueous solution
- **Efficiency –** the band gap (E_g) must be at least 1.6-1.7 eV, but not over 2.2 eV
- **Energetics –** the band edges must straddle H_2O redox potentials (Grand Challenge)

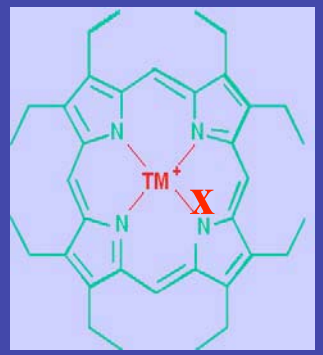
All must be satisfied simultaneously

Band Edge Engineering

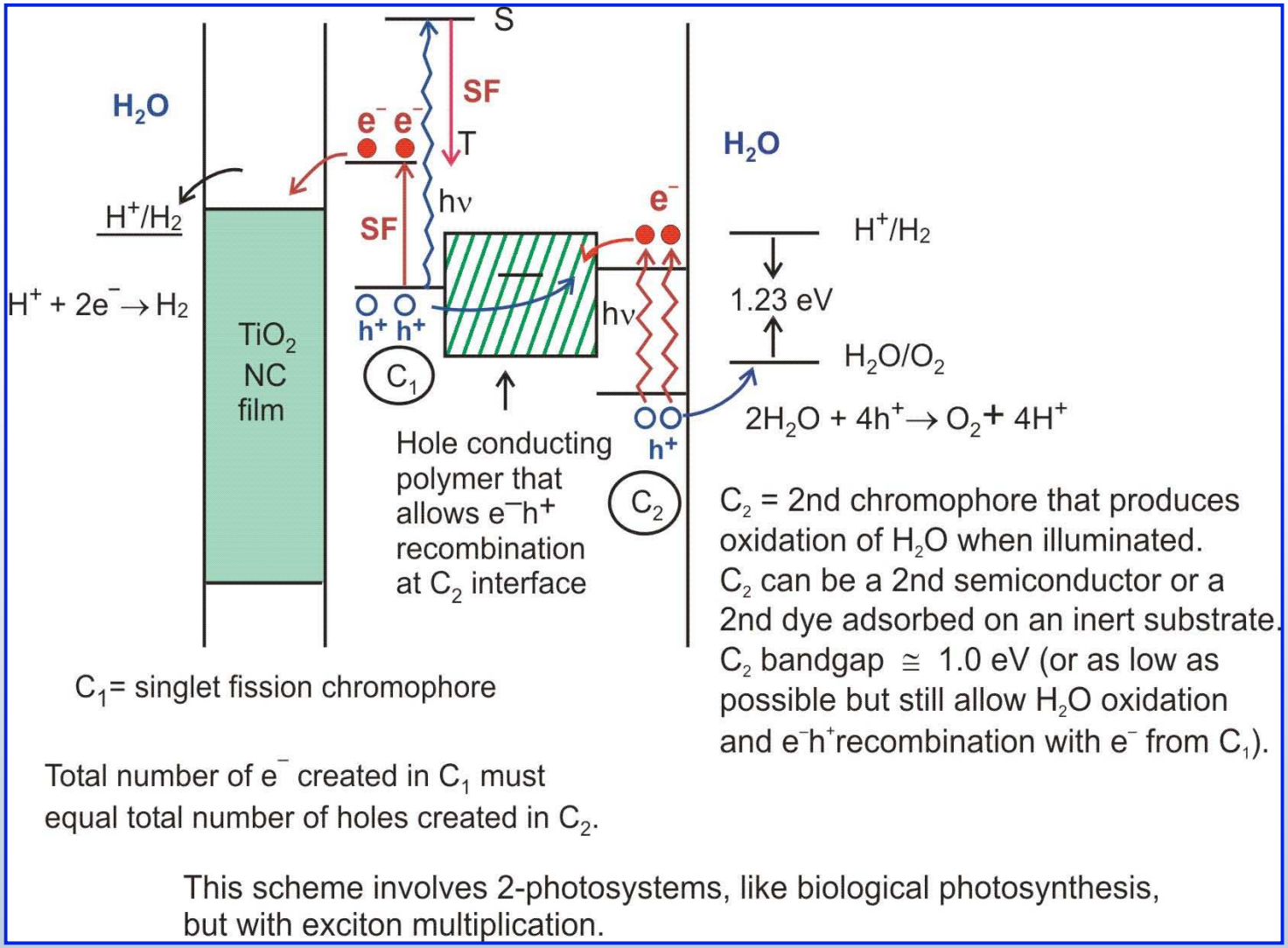
The goal is to shift the band edges by surface modification to provide the proper energetic overlap.



Negative charges move the bandedges negative.

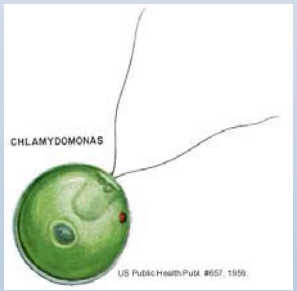


Positive charges move the bandedges positive.



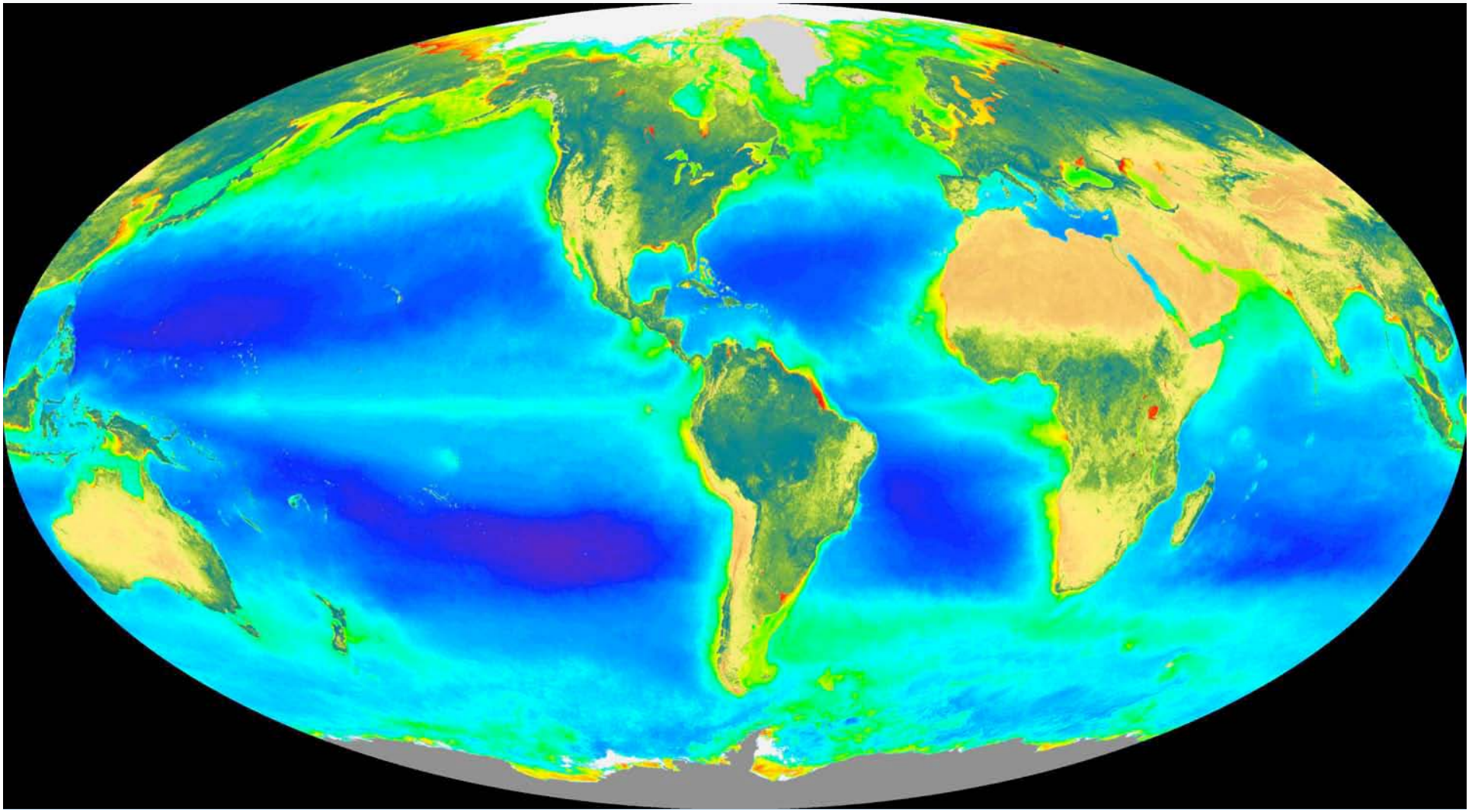
Ultra-high Efficiency Water Splitting Photoconversion Cell Based on Single Exciton Fission

Biological Systems for Hydrogen Photoproduction

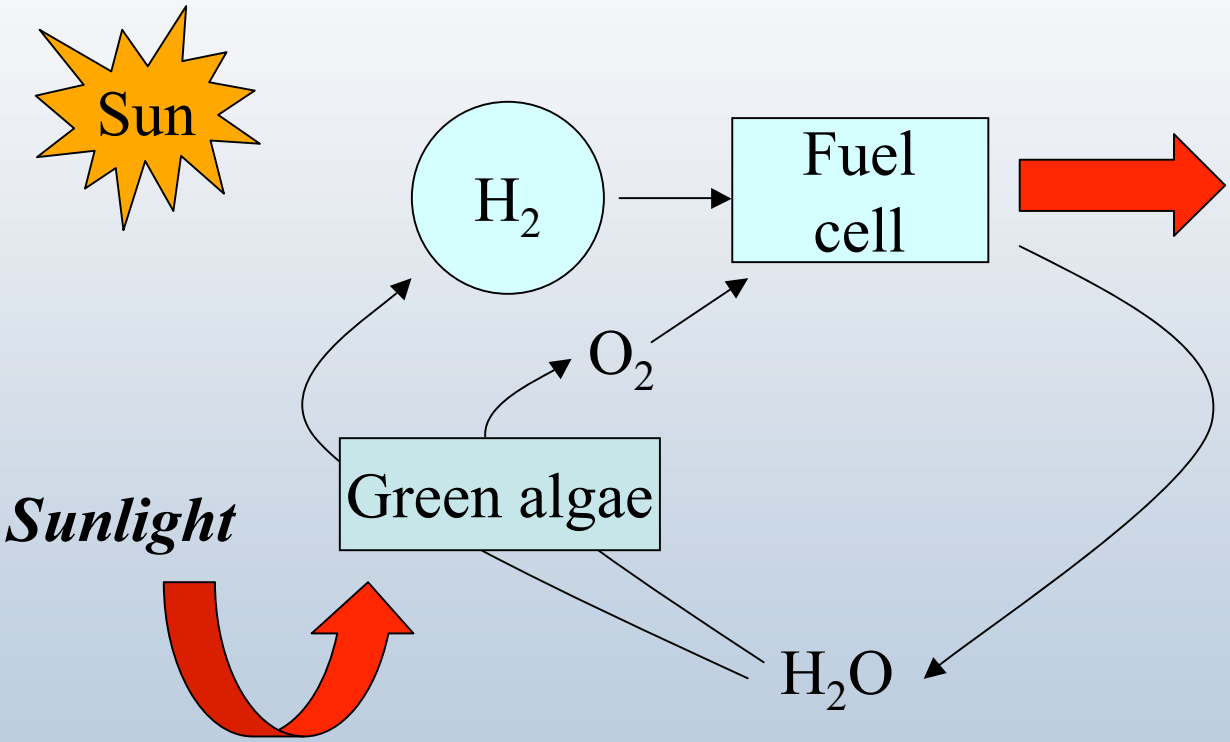


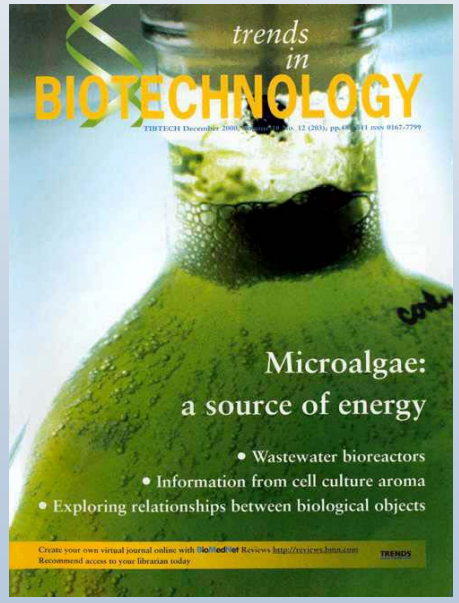
Chlorophyll on Earth!

Plants, cyanobacteria, and algae dominate the biosphere.
They live on sun and water.



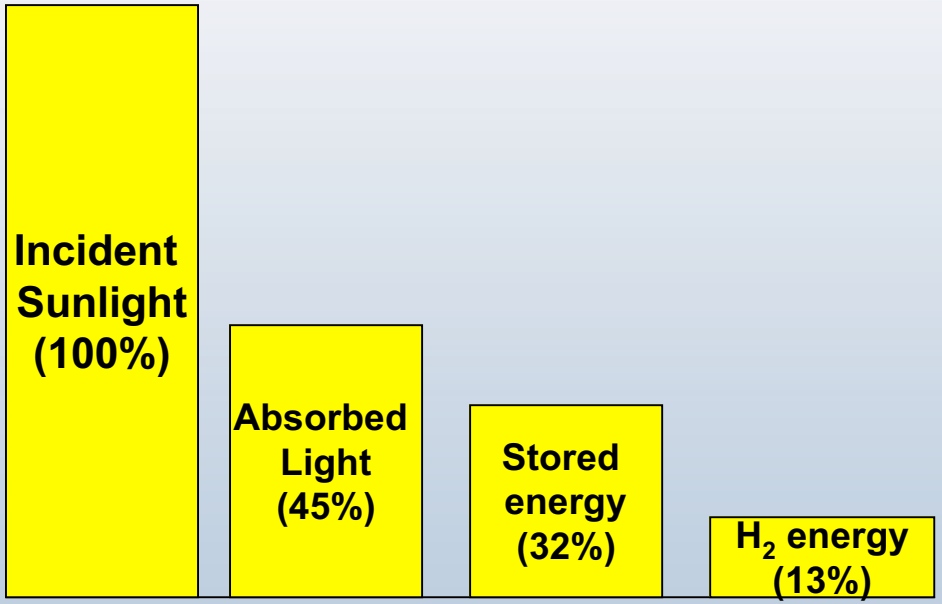
Vision for Renewable Biohydrogen Production/Utilization



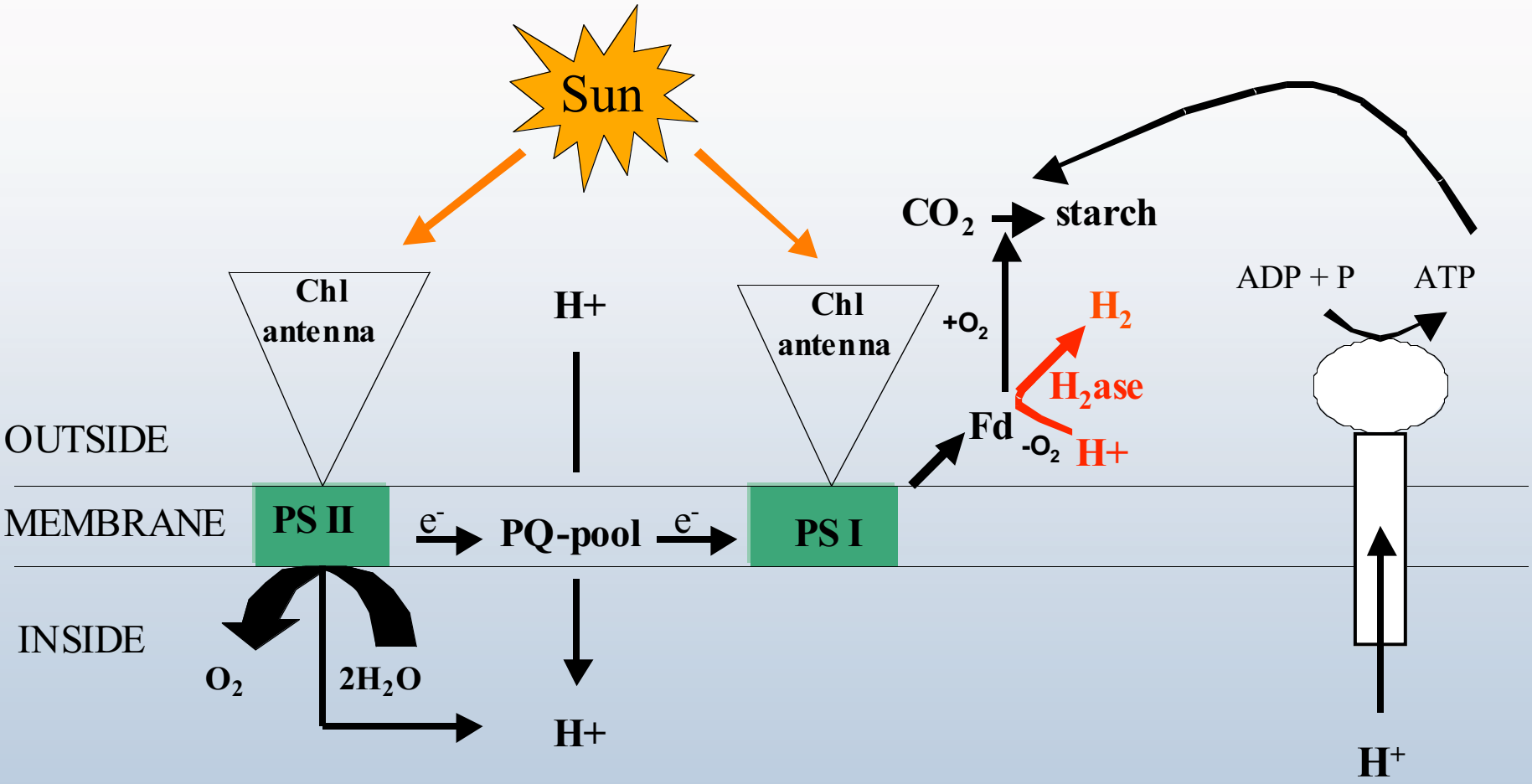


Potential of Photobiological H_2 Production

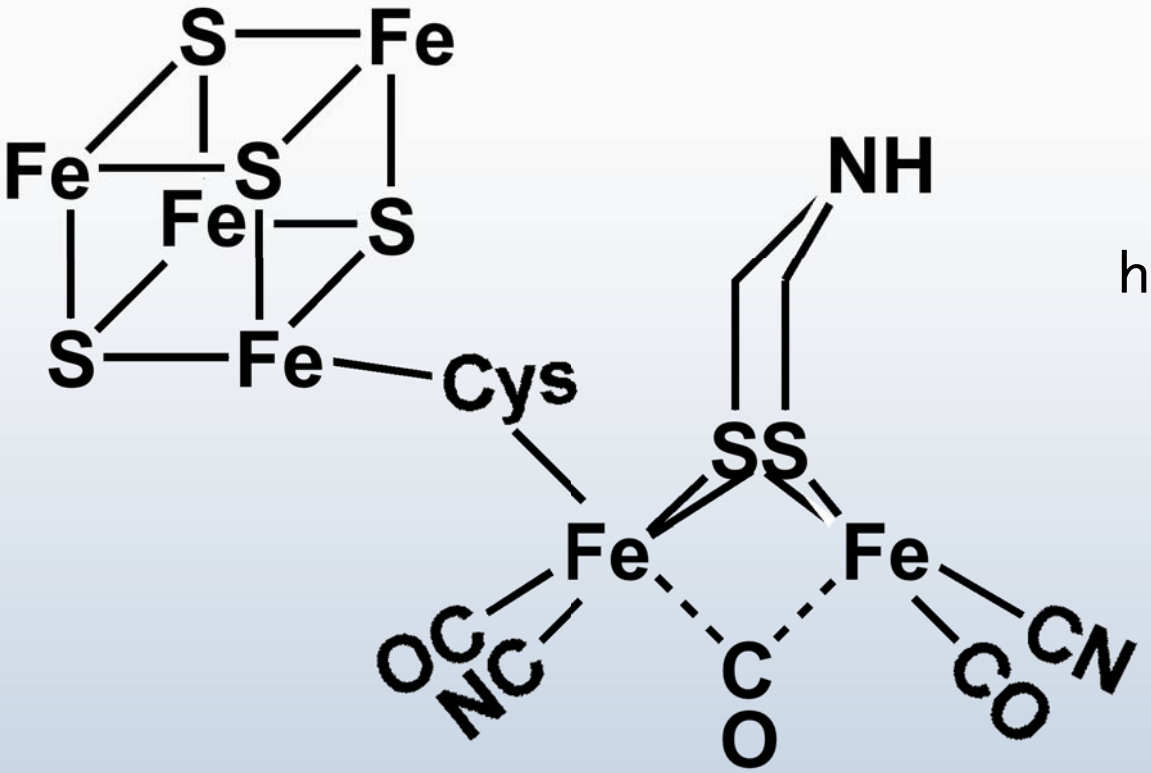
- Light absorbed by the organism's pigments: 45% of incident solar energy;
- Conversion of light into stable reductants and oxidants: 32% of incident solar energy;
- Light utilization for H_2 and O_2 production: 13% of incident solar energy



Problem to be Addressed



Model of [FeFe]-Hydrogenase H-cluster



[FeFe]-hydrogenases are expressed only under strictly anaerobic conditions

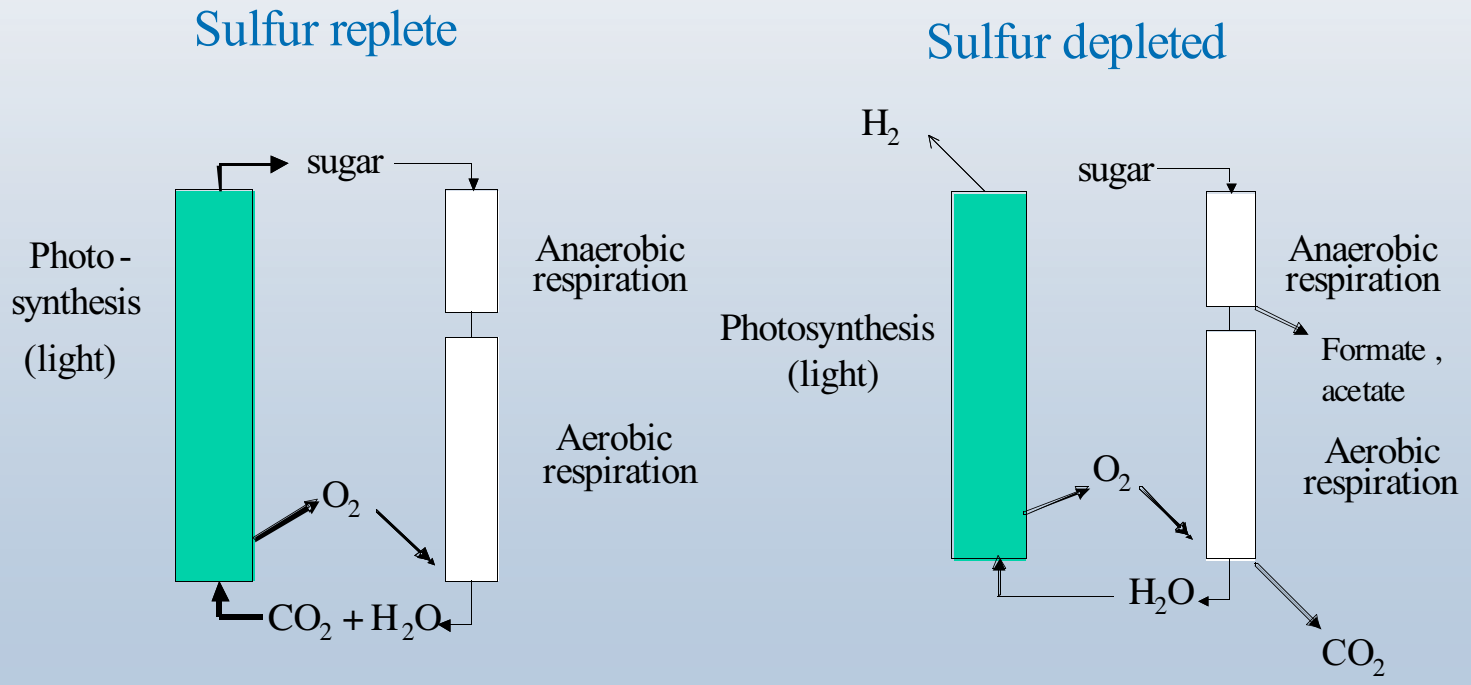
Peters JW, Lanzilotta WN, Lemon BJ, Seefeldt, LC
Science 1998, 282, 1853-1858.
Nicolet Y, Piras C, Legrand P, Hatchikian EC, Fontecilla-Camps JC
Structure and Folding Des. 1999, 7, 13-23.
Nicolet Y, deLacey AL, Vernede X, Fernandez VM, Hatchikian EC, Fontecilla-Camps JC
J. Amer. Chem. Soc. 2001, 123, 1596-1601.

Approaches for Generating an O_2-Tolerant, H_2-Producing System

1. Separate O_2 and H_2-production either temporally (batch system) or physically (continuous 2-reactor system);
2. Engineer an Fe-hydrogenase that functions in the presence of O_2;
3. Understand the mechanisms of hydrogenase gene regulation;
3. Introduce a gene encoding for an O_2-tolerant NiFe hydrogenase from *Rubrivivax gelatinosus* (bacterium) into a cyanobacterium.

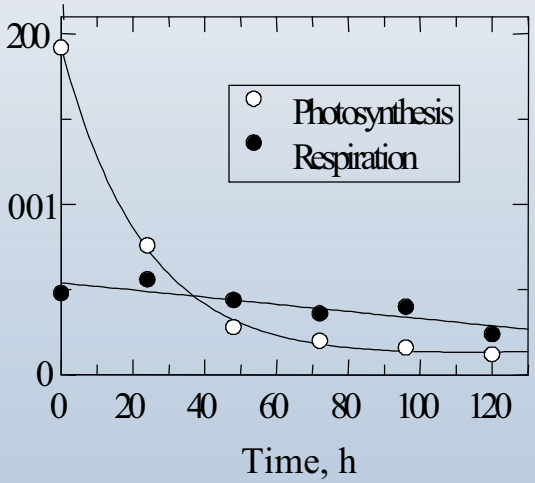
Physiological switch to prevent algal hydrogenase inactivation by O_2

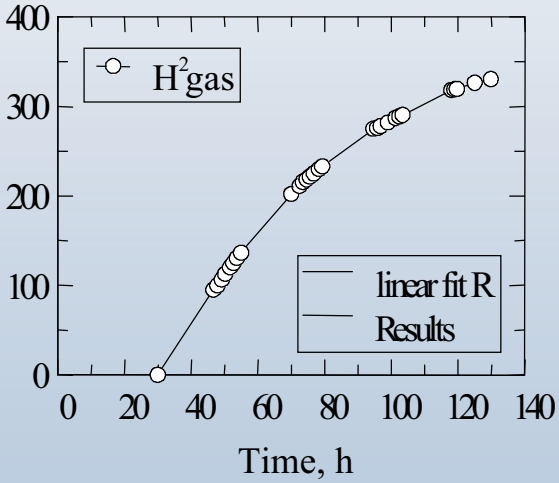
Sulfur deprivation decreases photosynthetic O_2 evolution, shuts off the CO_2 fixation into sugar and induces hydrogenase activity in *Chlamydomonas* (NREL and UCB).



Physiological switch to prevent algal hydrogenase inactivation by O_2

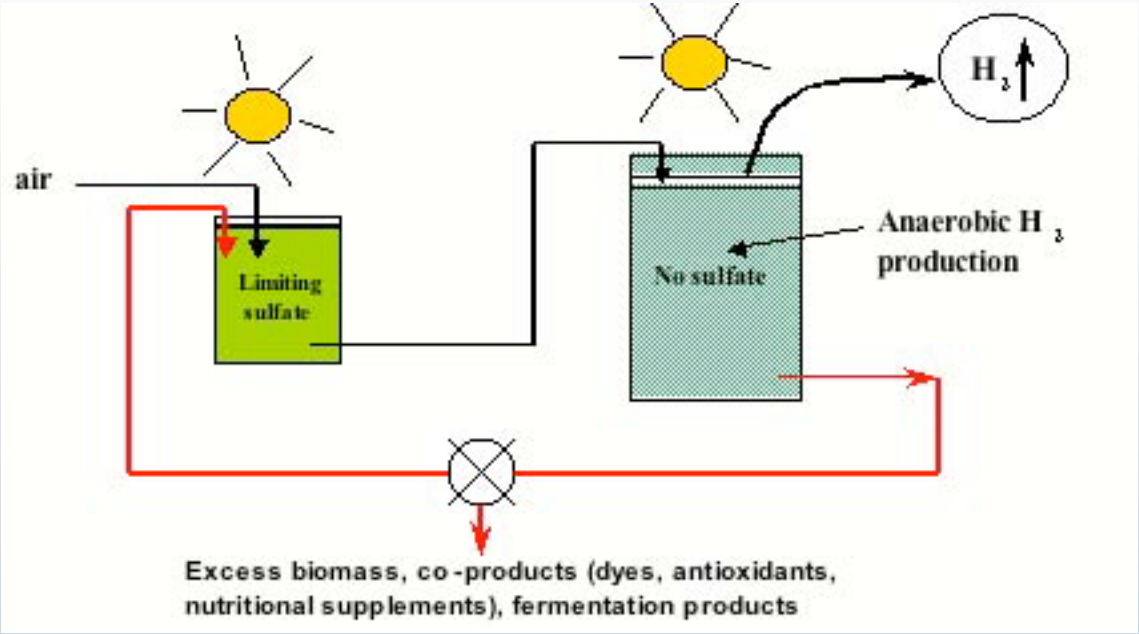
Batch system: Sulfur-deprived cultures gradually inactivate photosynthesis and become anaerobic (1-2 days). They then photoproduce H_2 for a total of 3-4 days. Cycles of +S and –S can be repeated for at least 3 times (Sergey Kosourov).





V_{av} = 30-60 ml L^{-1} d^{-1};
estimated cost for cyclic: $720/kg

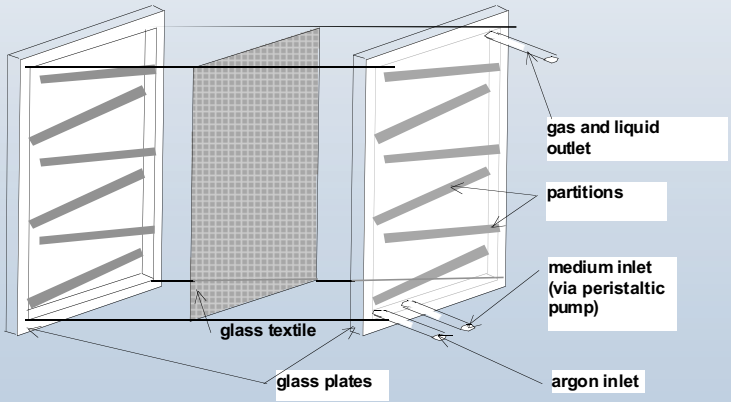
Physical Separation of O_2 Evolution and H_2 Production (continuous system)

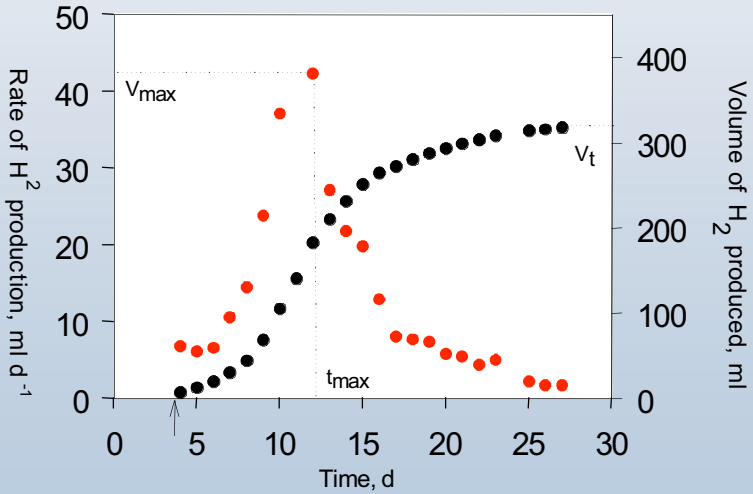


Advantages: H_2 production can be maintained uninterruptedly for up to 6 months; the rates are comparable to average rates of the two phases of the batch system; estimated lowering in costs by a factor of 3-4.

Physiological switch to prevent algal hydrogenase inactivation by O_2

Batch system with immobilized cultures: *Chlamydomonas* cells immobilized onto fiberglass can photoproduce H_2 at higher rates per volume than suspension cultures. H_2 production lasts 7 X longer than with suspension cultures. We are currently investigating the feasibility of using cycles of +S and –S with these cultures (Sasha Fedorov).

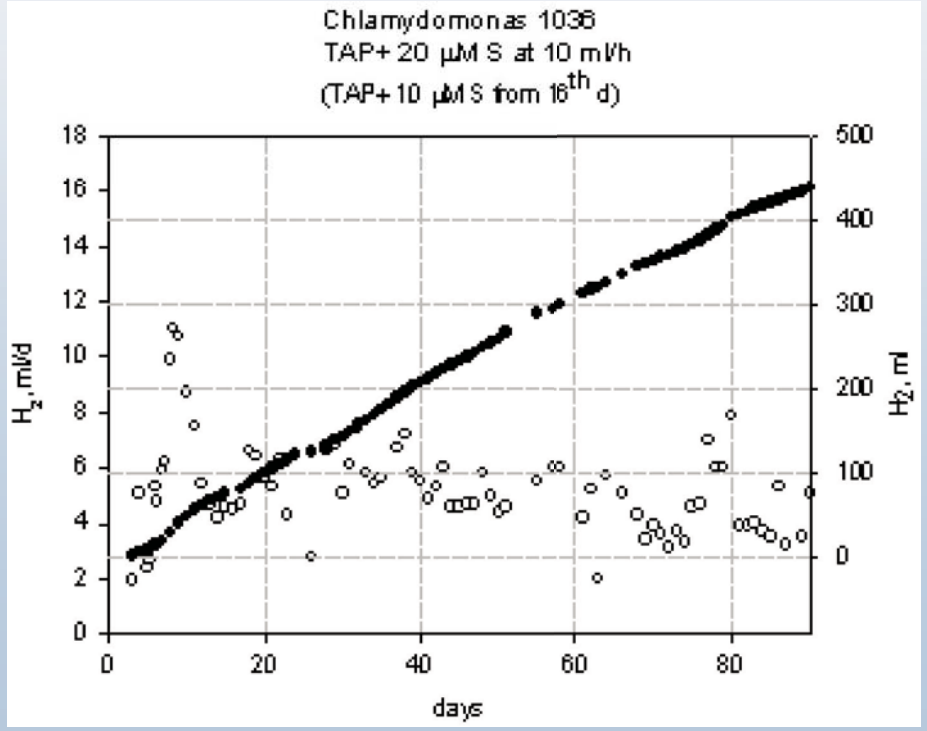




V_{av} = 917 ml L_{matrix}^{-1} d^{-1}; cyclic not investigated yet.

Sulfur Switch to Prevent Algal Hydrogenase Inactivation by O_2

Continuous system with immobilized cultures: *Chlamydomonas* cells were continuously cultivated in the presence of limiting sulfate. The cultures photoproduced H_2 for a total uninterrupted period of 90 days.



V_{av} = 283 ml L_{matrix}^{-1} d^{-1}

Approaches for Generating an O_2-Tolerant, H_2-Producing System

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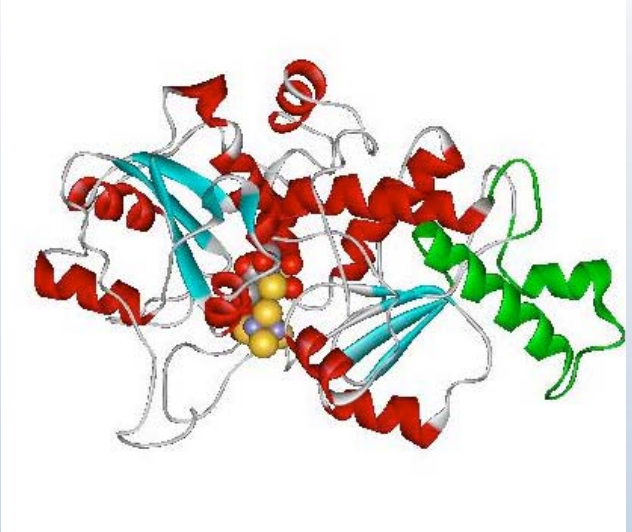
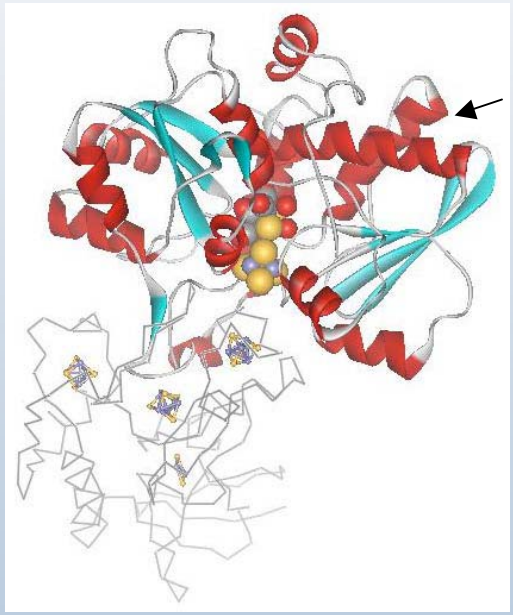
4. Introduce a gene encoding for an O_2-tolerant NiFe hydrogenase from *Rubrivivax gelatinosus* (bacterium) into a cyanobacterium.

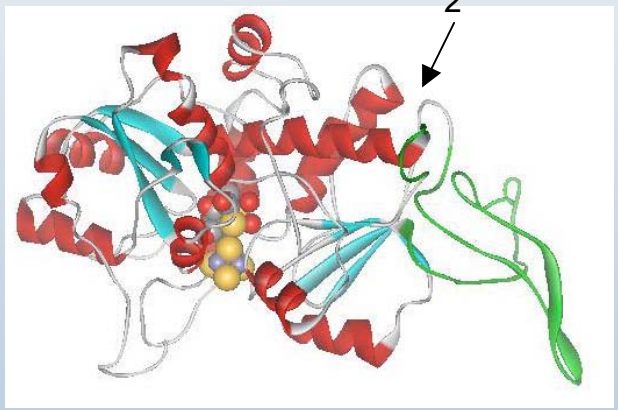


Engineering an O_2-tolerant Hydrogenase

- The catalytic center is located in the middle of the enzyme structure; electrons are delivered to it directly by the electron donor ferredoxin.

Bacterial *(Clostridium Pasteurianum)* CpI hydrogenase (based on X-ray structure)

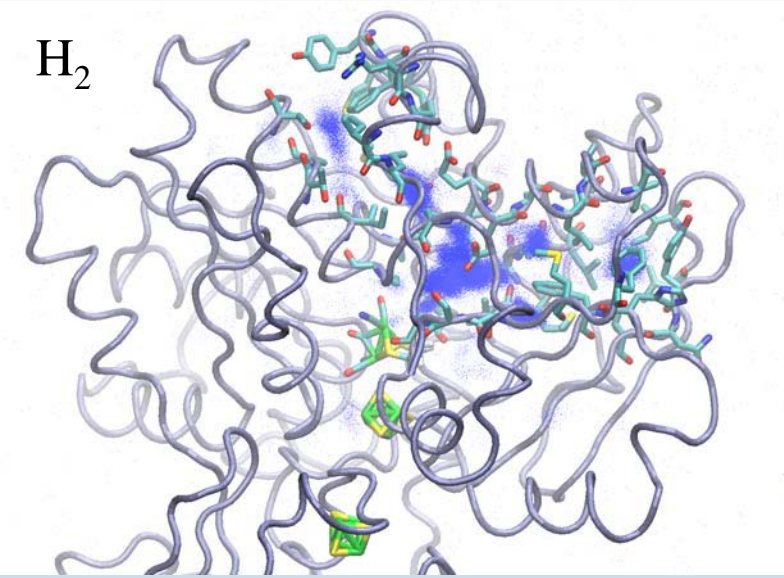


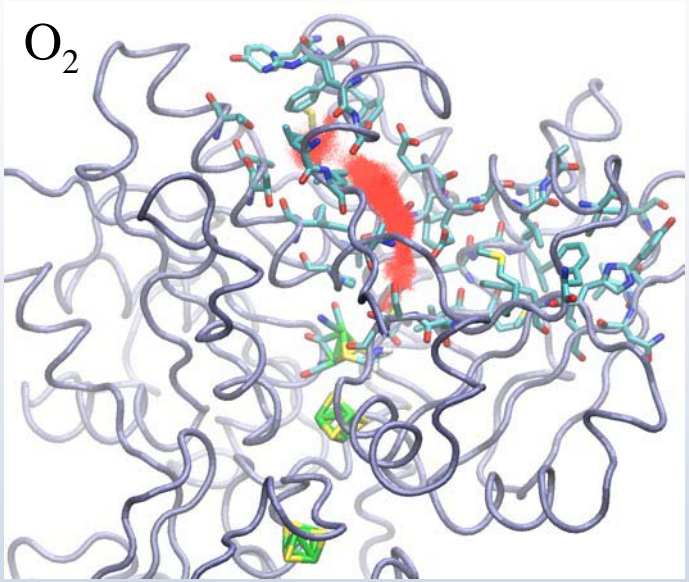


Algal *(Chlamydomonas reinhardtii)* HydA2 hydrogenase (based on homology modeling

We proposed that O_2 inactivation could be prevented by limiting O_2 access to the catalytic site of the enzyme.

Combined Trajectories for H_2 and O_2 Diffusion Simulations



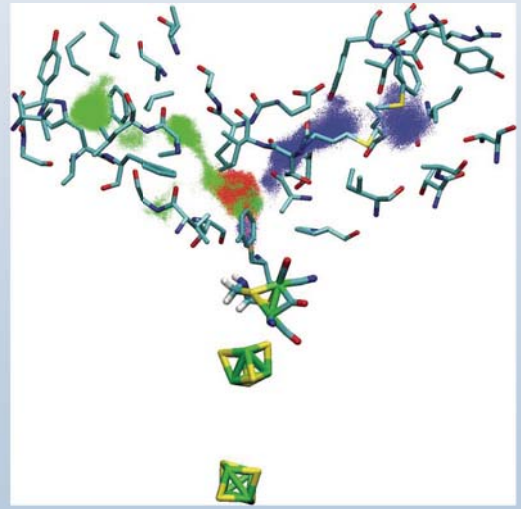


- H_2 diffuses from active site at a greater rate than O_2, and through many more “pathways”
- O_2 diffused through a limited number (1, possibly more) of pathways
- Rate difference and non-biased pathways are due to the difference in size between H_2 and O_2 (masses are set equal)

Engineering an O_2-tolerant Hydrogenase

Introduction of large amino acid residues in the cavity next to the catalytic site (red) resulted in non-assembly of an active enzyme. Mutations done along pathway A (green) resulted in insignificant (< 20%) increase in O_2 tolerance. Future work will address double mutations affecting both pathways simultaneously.

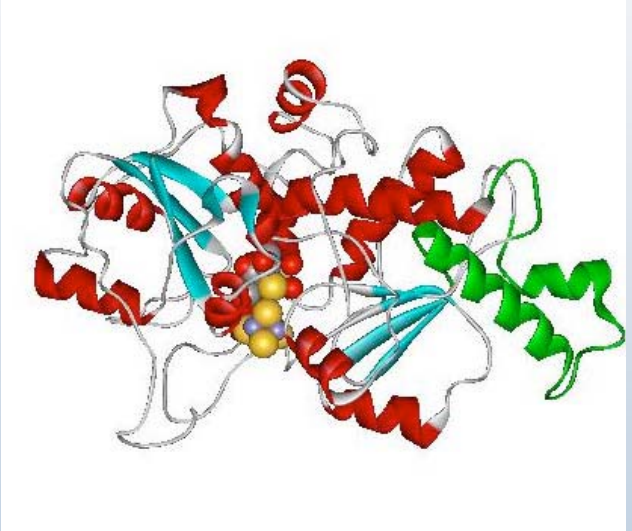
Pathway A



Pathway B

Approaches for Generating an O_2-Tolerant, H_2-Producing System

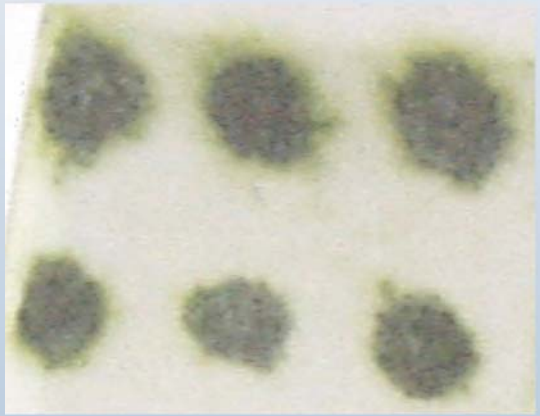
1. Separate O_2 and H_2-production either temporally (batch system) or physically (continuous 2-reactor system);
2. Engineer an Fe-hydrogenase that functions in the presence of O_2;
3. Understand the mechanisms of hydrogenase gene regulation;
3. Introduce a gene encoding for an O_2-tolerant NiFe hydrogenase from *Rubrivivax gelatinosus* (bacterium) into a cyanobacterium.



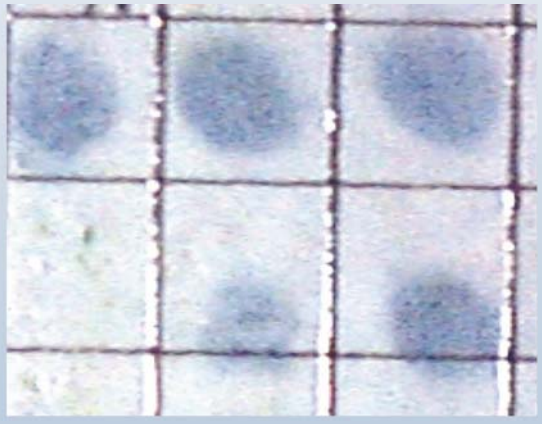
Identification of Factors Involved in Hydrogenase Gene Expression

Chemochromic Screen for H_2-photoproduction

Colonies on TAP plates



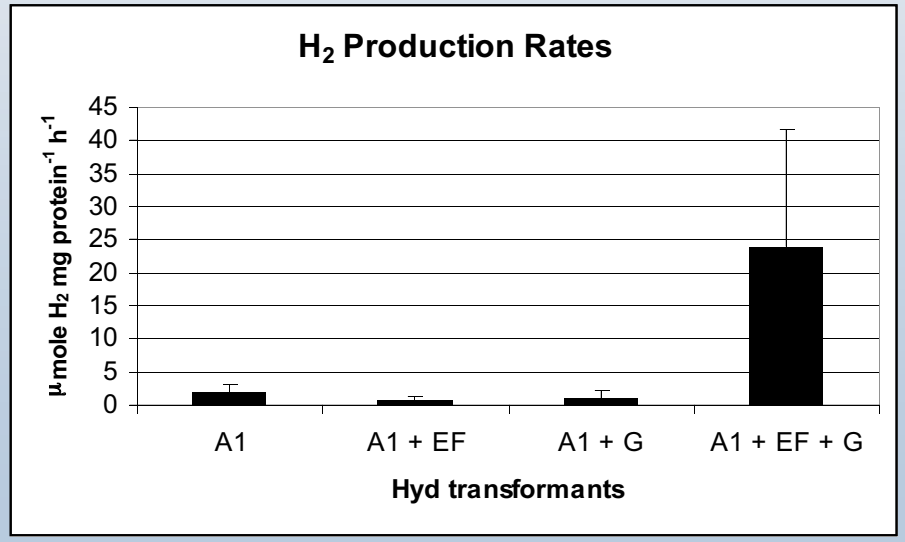
Chemochromic sensor



Sensor is based on the reduction of WO_3 by H_2

Heterologous expression of the Algal Hydrogenase in the Bacterium *E. coli*

- One of the mutants that is unable to produce H_2 is interrupted in a gene (HydEF) required for the assembly of the algal hydrogenase's metallo-catalytic center.
- We demonstrated that another adjacent gene, HydG, is also required for assembly.
- By inserting HydEF, HydG and the hydrogenase HydA1 genes in *E. coli*, we achieved expression of an active algal hydrogenase.



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3. Understand the mechanisms of hydrogenase gene regulation;
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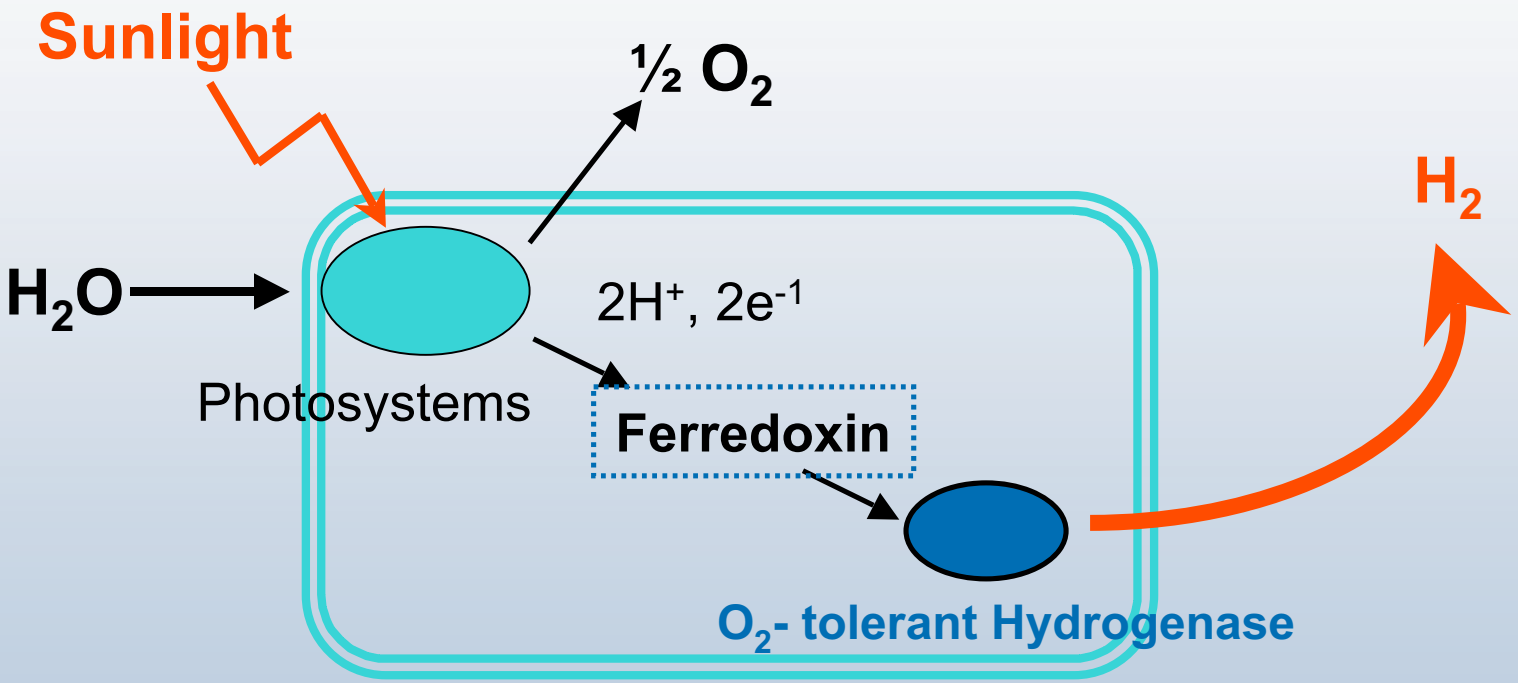
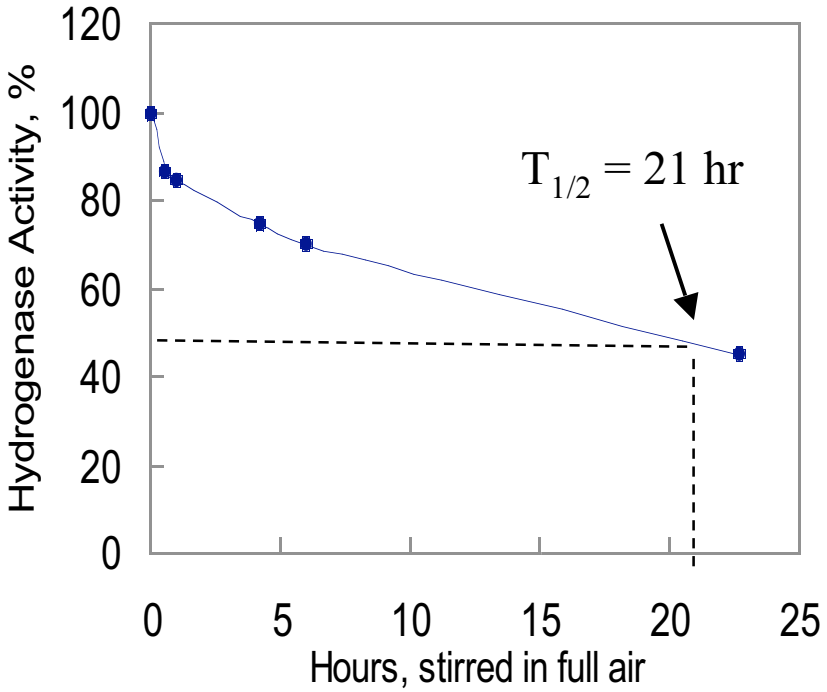
A Cyanobacterial-bacterial Hybrid System



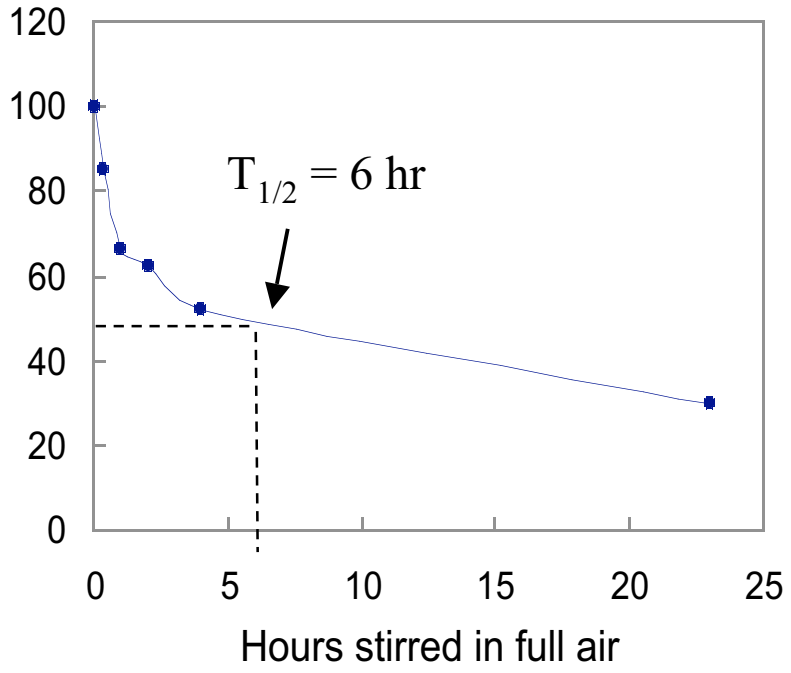
A Cyanobacterial Recombinant Organism

R. gelatinosus Hydrogenase Tolerates O_2

(A) Whole Cell



(B) Purified Hydrogenase



- **Whole cell hydrogenase has a half-life of 21 hr in air**
- **Purified hydrogenase has a half-life of 6 hr in air**

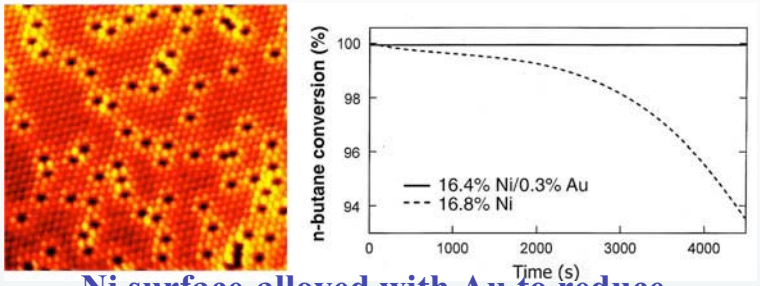
Future Directions

- Scientific feasibility of photobiological H_2 production has been demonstrated.
- It will be a number of years before a commercially viable technology is developed.
- Clear research directions to develop an algal H_2 technology have been identified.
- Cost factors may dictate the addition of coproduct recovery, a system to harvest more of the solar spectrum, and fermentation of excess biomass.

Priority Research Areas in Hydrogen Production

Fossil Fuel Reforming

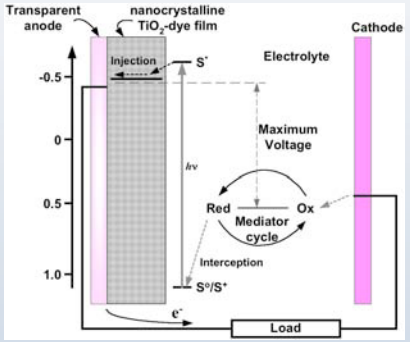
Molecular level understanding of catalytic mechanisms, nanoscale catalyst design, high temperature gas separation

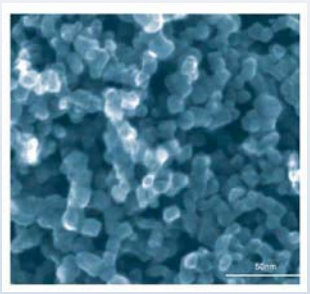


Ni surface-alloyed with Au to reduce carbon poisoning

Solar Photoelectrochemistry/Photocatalysis

Light harvesting, charge transport, chemical assemblies, bandgap engineering, interfacial chemistry, catalysis and photocatalysis, organic semiconductors, theory and modeling, and stability





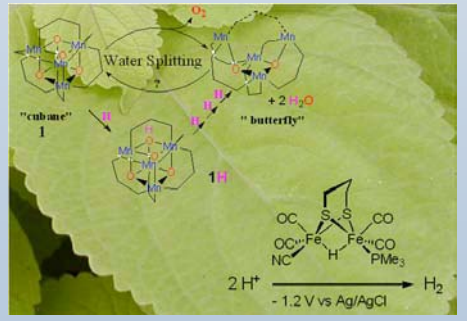
Dye-Sensitized Solar Cells

Bio- and Bio-inspired H_2 Production

Microbes & component redox enzymes, nanostructured 2D & 3D hydrogen/oxygen catalysis, sensing, and energy transduction, engineer robust biological and biomimetic H_2 production systems

Nuclear and Solar Thermal Hydrogen

Thermodynamic data and modeling for thermochemical cycle (TC), high temperature materials: membranes, TC heat exchanger materials, gas separation, improved catalysts

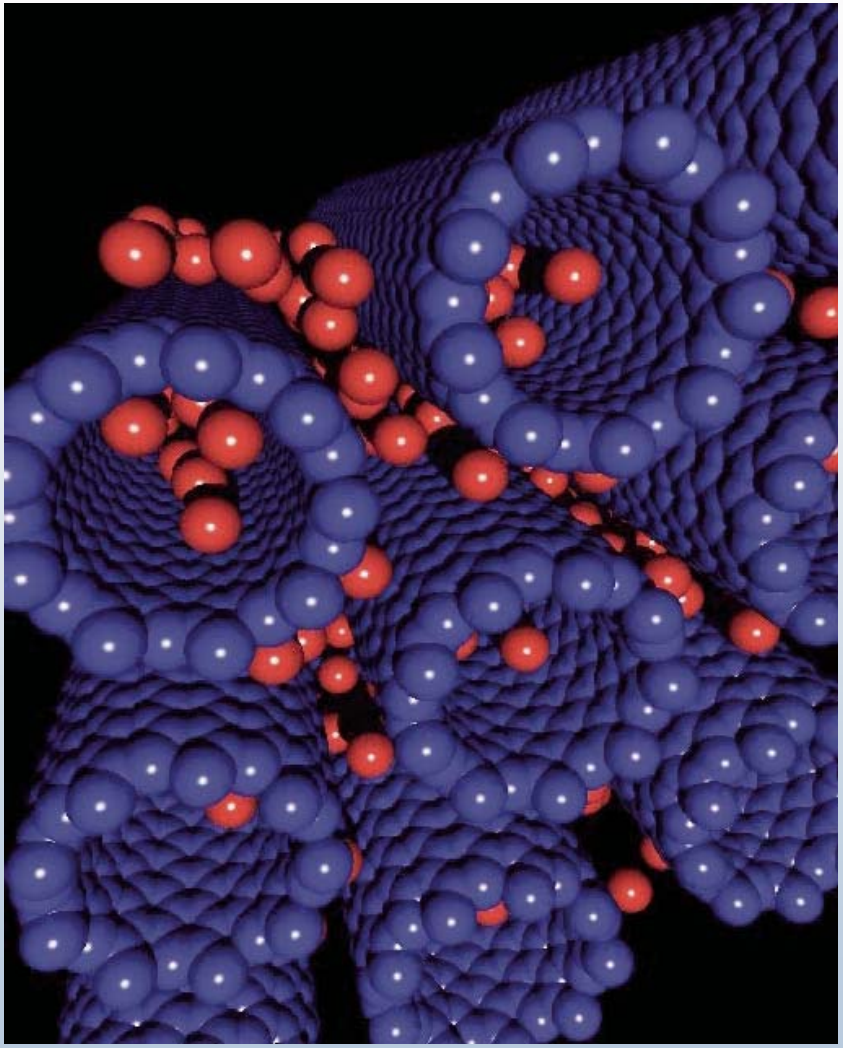


Synthetic Catalysts for Water Oxidation and Hydrogen Activation

Don Addis St. Petersburg Times Feb. 23, 2000 p.13A

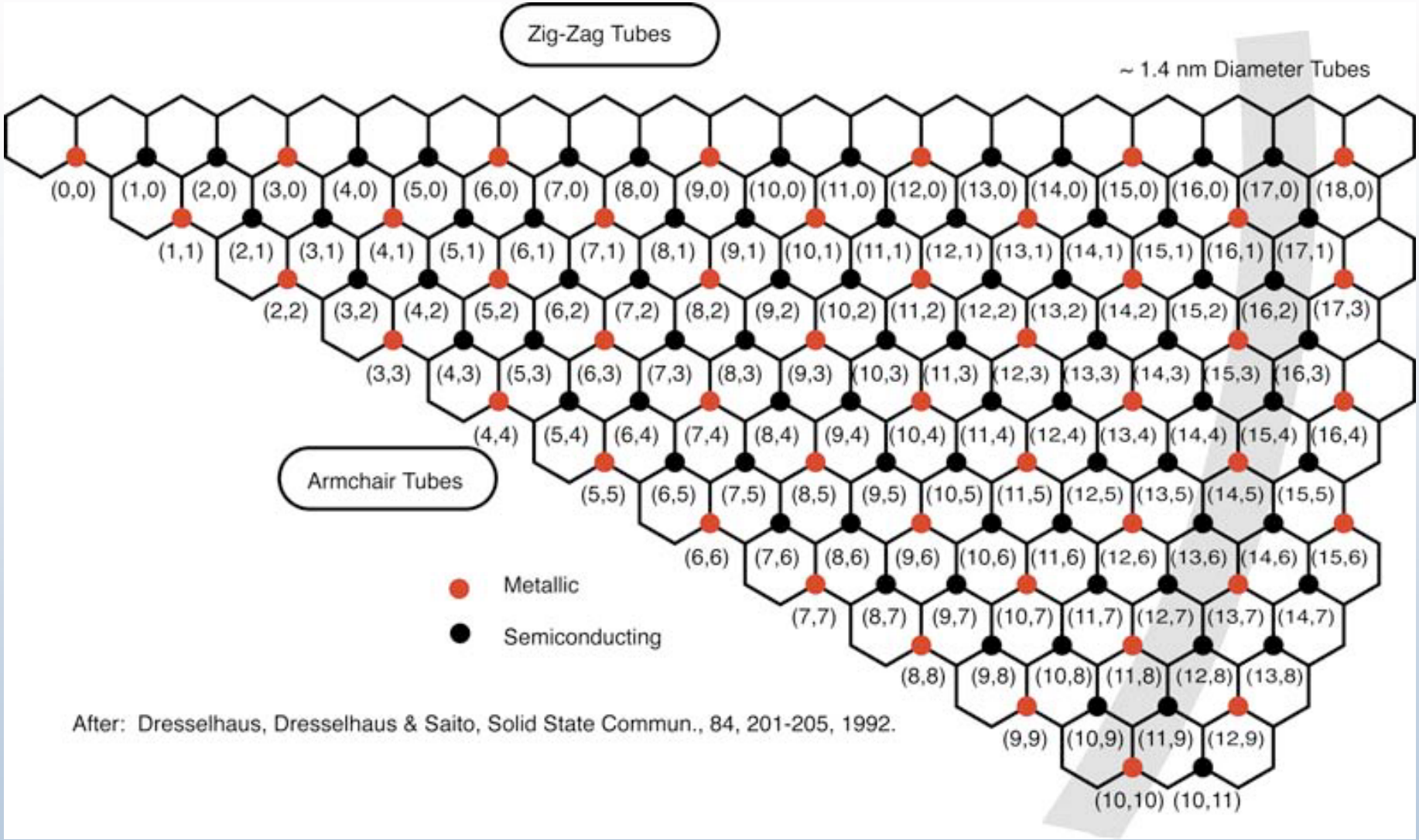
Hydrogen Storage by Carbon Nanotube

Carbon Nanotubes for Hydrogen Storage

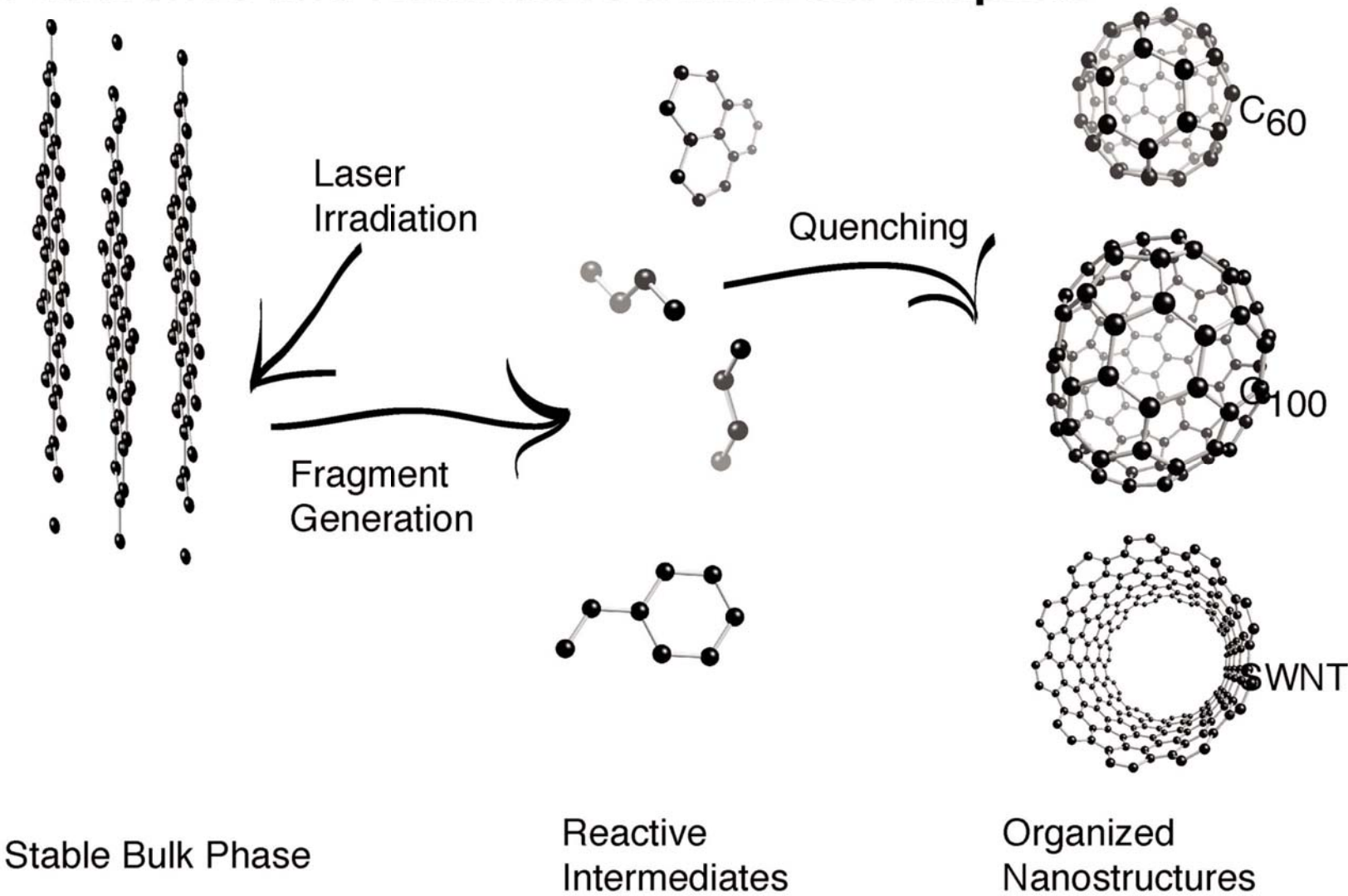


- **The very small size and very high surface area of carbon nanotubes make them interesting for hydrogen storage.**
- **Challenge is to increase the H:C stoichiometry and to strengthen the H—C bonding at 300 K.**

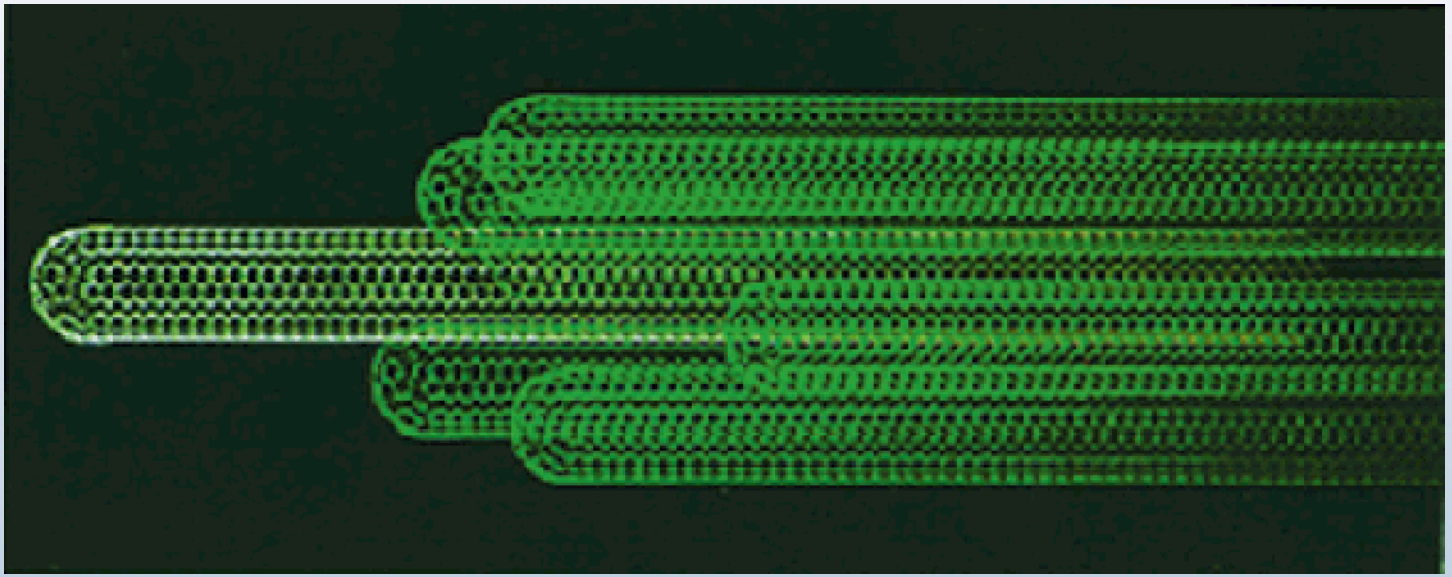
A computational representation of hydrogen adsorption in an optimized array of (10,10) nanotubes at 298 K and 200 Bar. The red spheres represent hydrogen molecules and the blue spheres represent carbon atoms in the nanotubes, showing 3 kinds of binding sites. (K. Johnson et al.)


Zig-Zag Tubes
~ 1.4 nm Diameter Tubes
(0,0) (1,0) (2,0) (3,0) (4,0) (5,0) (6,0) (7,0) (8,0) (9,0) (10,0) (11,0) (12,0) (13,0) (14,0) (15,0) (16,0) (17,0) (18,0)
(1,1) (2,1) (3,1) (4,1) (5,1) (6,1) (7,1) (8,1) (9,1) (10,1) (11,1) (12,1) (13,1) (14,1) (15,1) (16,1) (17,1)
(2,2) (3,2) (4,2) (5,2) (6,2) (7,2) (8,2) (9,2) (10,2) (11,2) (12,2) (13,2) (14,2) (15,2) (16,2) (17,3)
(3,3) (4,3) (5,3) (6,3) (7,3) (8,3) (9,3) (10,3) (11,3) (12,3) (13,3) (14,3) (15,3) (16,3)
(4,4) (5,4) (6,4) (7,4) (8,4) (9,4) (10,4) (11,4) (12,4) (13,4) (14,4) (15,4) (16,4)
Armchair Tubes
(5,5) (6,5) (7,5) (8,5) (9,5) (10,5) (11,5) (12,5) (13,5) (14,5) (15,5)
(6,6) (7,6) (8,6) (9,6) (10,6) (11,6) (12,6) (13,6) (14,6) (15,6)
Metallic
Semiconducting
(7,7) (8,7) (9,7) (10,7) (11,7) (12,7) (13,7) (14,7)
(8,8) (9,8) (10,8) (11,8) (12,8) (13,8)
(9,9) (10,9) (11,9) (12,9)
(10,10) (10,11)
After: Dresselhaus, Dresselhaus & Saito, Solid State Commun., 84, 201-205, 1992.

Fullerenes and Nanotubes from Bulk Graphite



- Carbon Single-walled Nanotubes (SWNT



Some milestones in the synthesis of single-wall nanotubes.

1985 - Kroto et al. discover C_{60}
(leads to 1996 Nobel Prize in Chemistry).

1990 - Kratschmer et al. produce macroscopic quantities of C_{60}

1991 - Iijima discovers multi-walled carbon nanotubes (MWNTs).

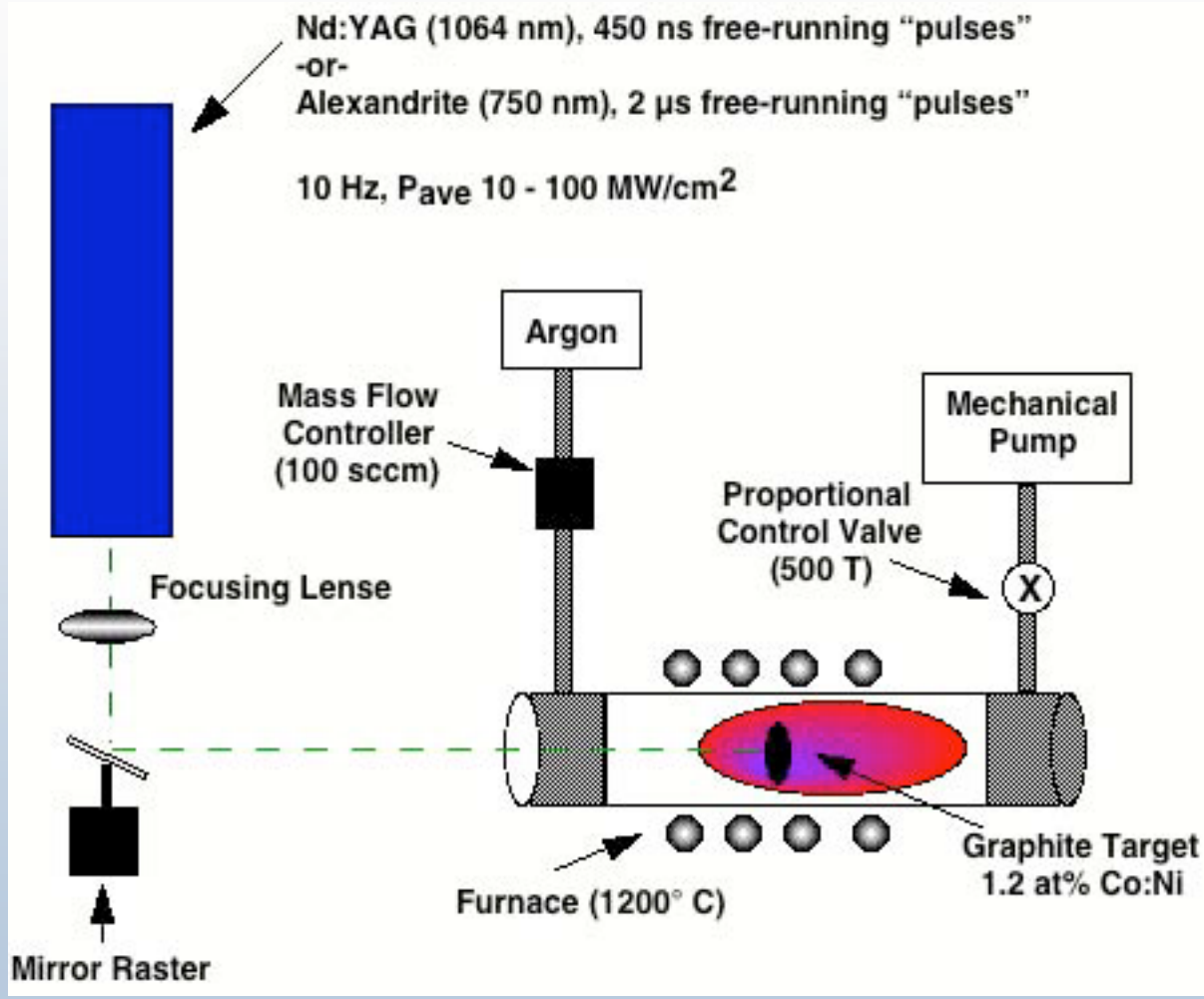
1992 - Ebbesen and Ajayan synthesize gram quantities of MWNTs.

1993 - Iijima & Ichihashi and Bethune et al. discover single-wall nanotubes (SWNTs) by arc-discharge.

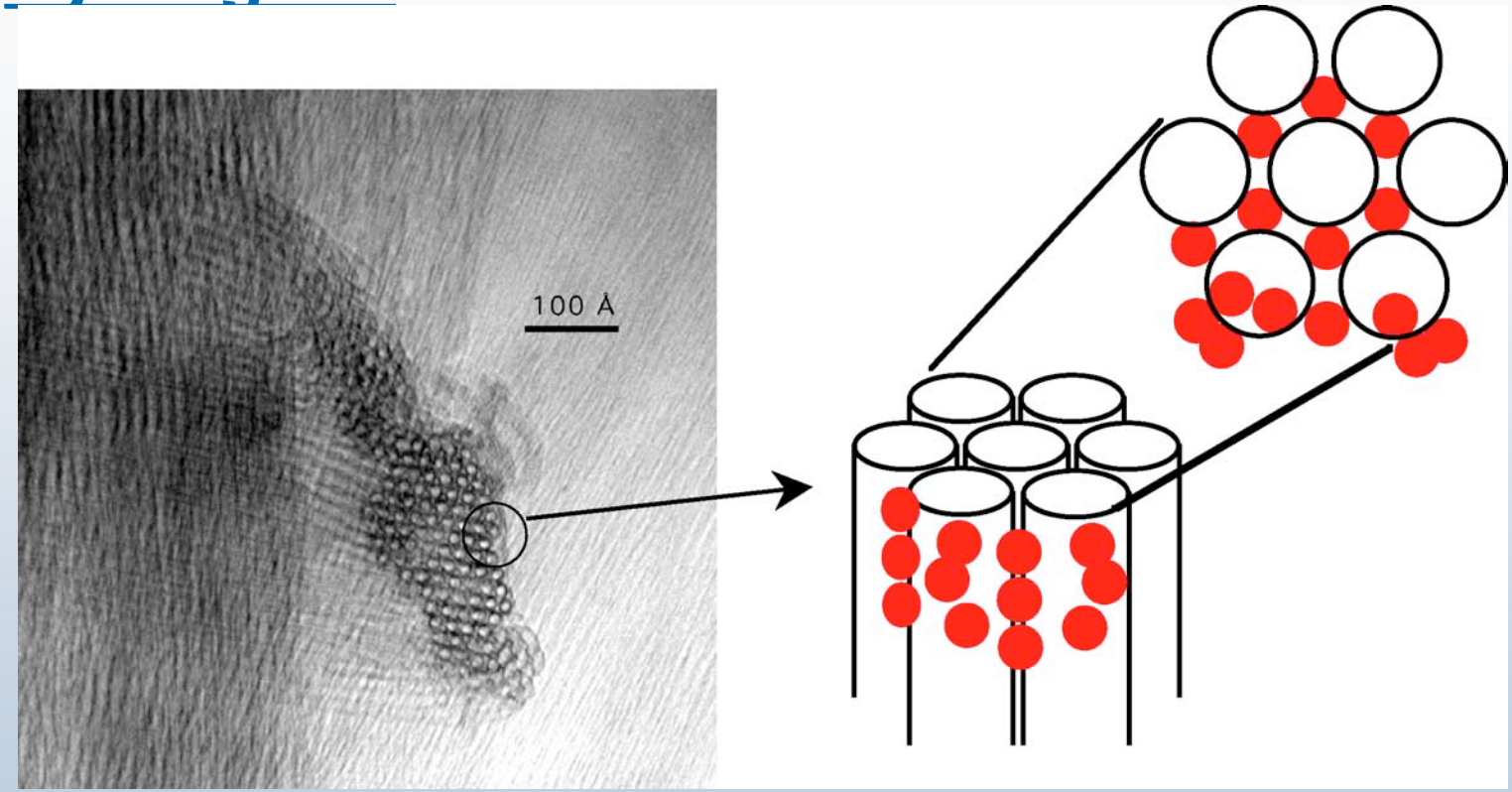
1995 - Guo et al. introduce laser vaporization for production of higher purity SWNT samples.

1996 - Dai et al. decompose CO on metal catalysts to grow SWNTs, introducing chemical vapor deposition.

Laser Synthesis

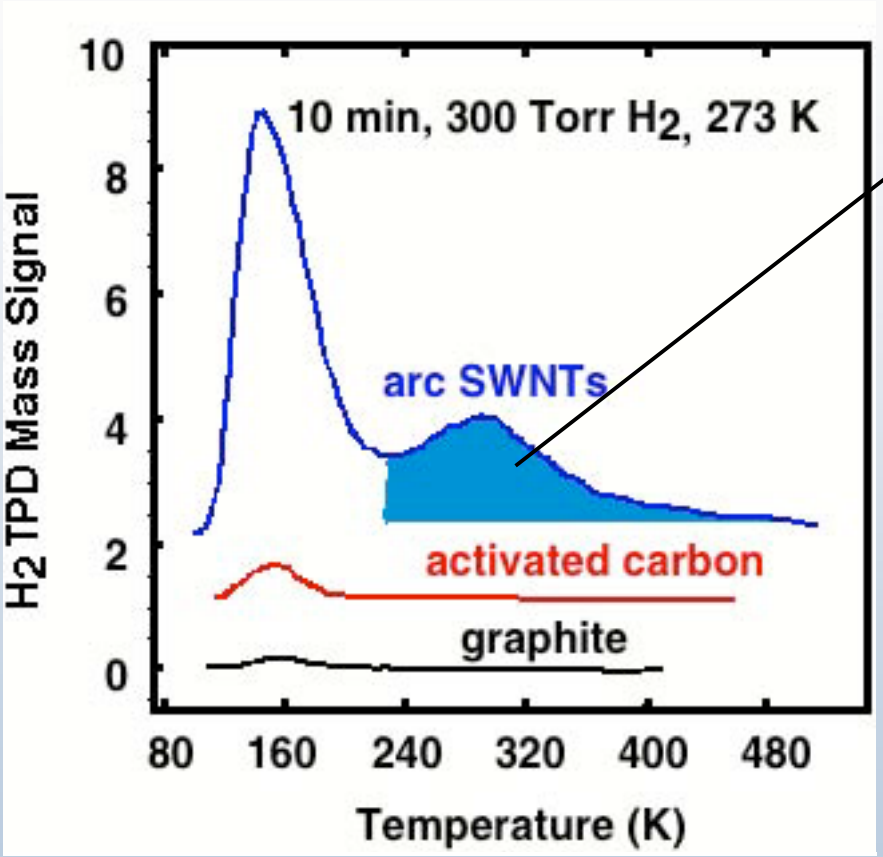


SWNTs: An Ideal Adsorbent for Hydrogen?



M.J. Heben, Proceedings of the 1993 DOE/NREL Hydrogen Program Review, 79-88, 1993.

Hydrogen Adsorption After Degassing

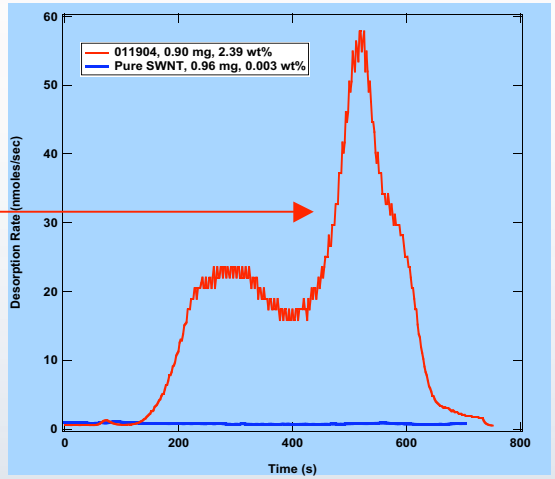


- New high temperature adsorption site
- Integrated H_2 was ~0.01 wt% of the total sample weight
- Storage densities between 5 and 10 wt% on a SWNT basis

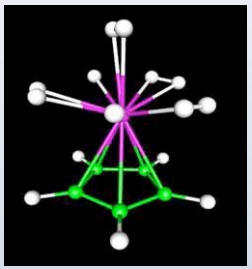
A.C. Dillon, K.M. Jones, T.A.Bekkedahl, C.H. Kiang, D.S. Bethune, & M.J. Heben, Nature (386) 377, 1997.

Storage Accomplishments- Carbon and New Materials

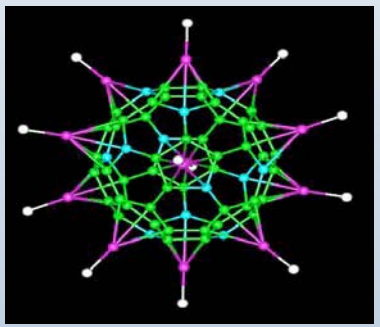
- Doped single-wall nanotubes (SWNTs) synthesized and capacity measured to be ~2.5 - 3 wt.% hydrogen storage
- Expanded work scope beyond SWNTs
- Binding energies calculated and optimum compounds theoretically predicted for potential storage materials

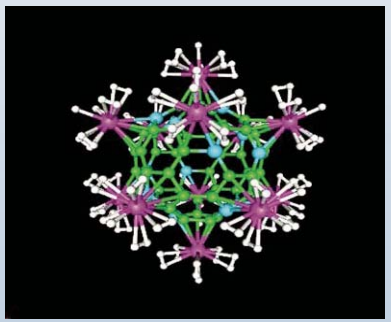


H_2 Desorption



$Cp[ScH_2(H_2)_4]$
0.23 eV



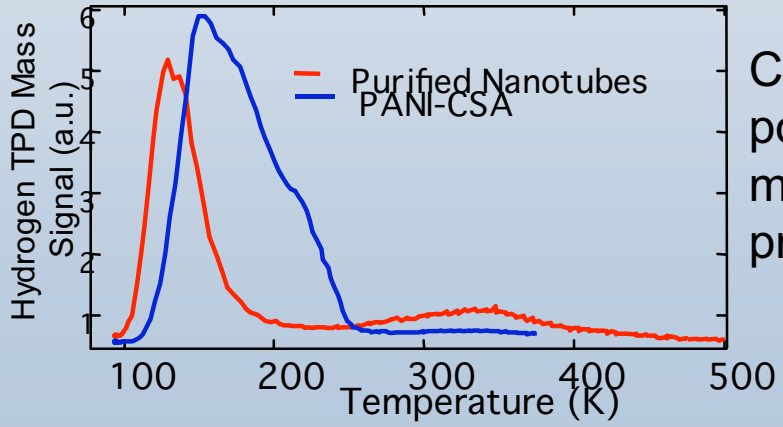


$60H_2$

$C_{48}B_{12}[ScH]_{12} \rightleftharpoons C_{48}B_{12}[ScH(H_2)_5]_{12}$

Potential for 8.8 wt%

NREL: Y. Zhao, et al., PRL 94, 155504 (2005)



Conducting polymers may show promise

MacDiarmid, et al, U.Penn, Heben et al, NREL

SWNT Technologies

- Hydrogen storage
- Gas separation membranes
- Rechargeable Li Ion batteries
- Electrically conducting polymer and ceramic composites
- Ultracapacitors
- High-strength, high-temperature, low-weight composites
- Energy absorbing armor
- Heat dissipation / shielding
- Nanoscale wires & interconnects
- Electromagnetic shielding
- Artificial muscle
- Field emission displays
- Chemical and Biological sensors
- Toxic gas adsorbents

Priority Research Areas in Hydrogen Storage

Metal Hydrides and Complex Hydrides

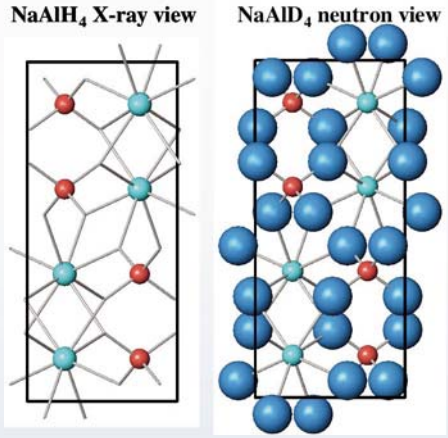
Degradation, thermophysical properties, effects of surfaces, processing, dopants, and catalysts in improving kinetics, nanostructured composites

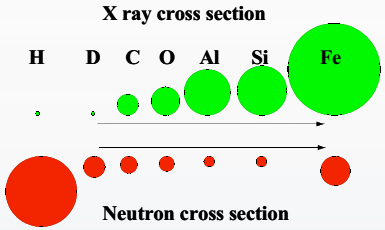
Nanoscale/Novel Materials

Finite size, shape, and curvature effects on electronic states, thermodynamics, and bonding, heterogeneous compositions and structures, catalyzed dissociation and interior storage phase

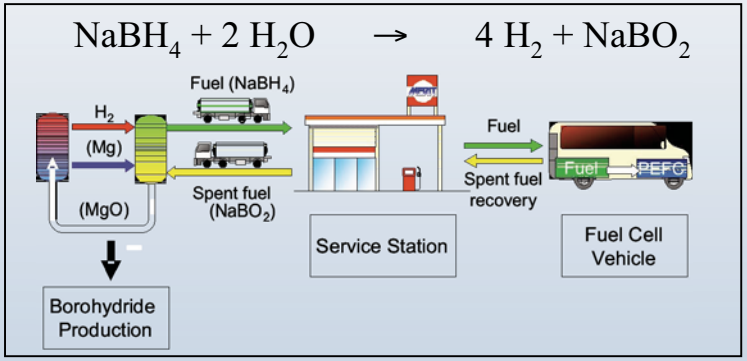
Theory and Modeling

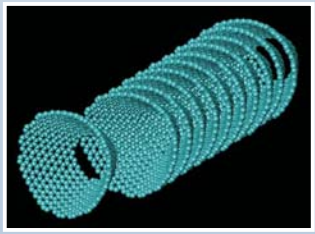
Model systems for benchmarking against calculations at all length scales, integrating disparate time & length scales, first principles methods applicable to condensed phases



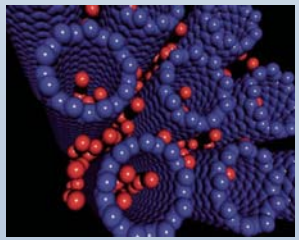


Neutron Imaging of Hydrogen





Cup-Stacked Carbon Nanofiber

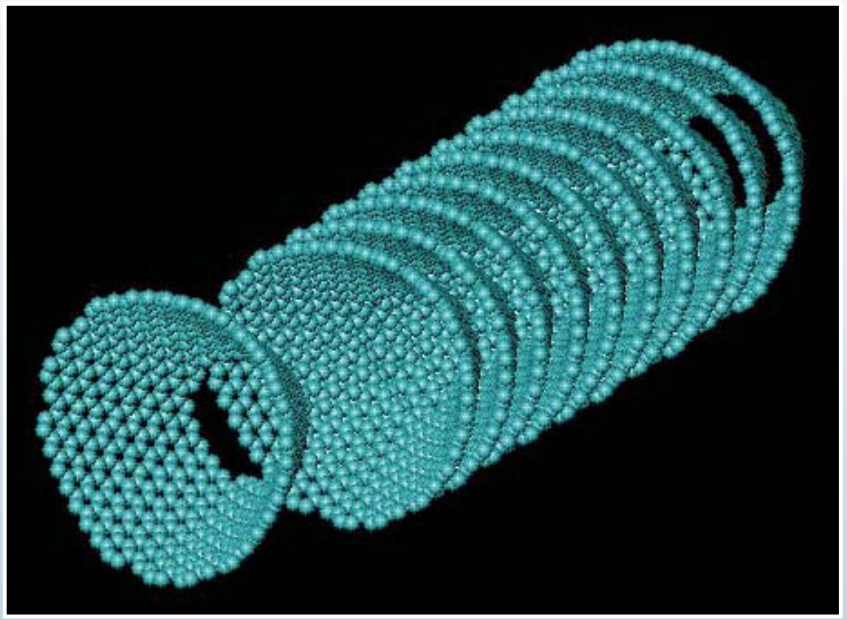


H Adsorption in Nanotube Array

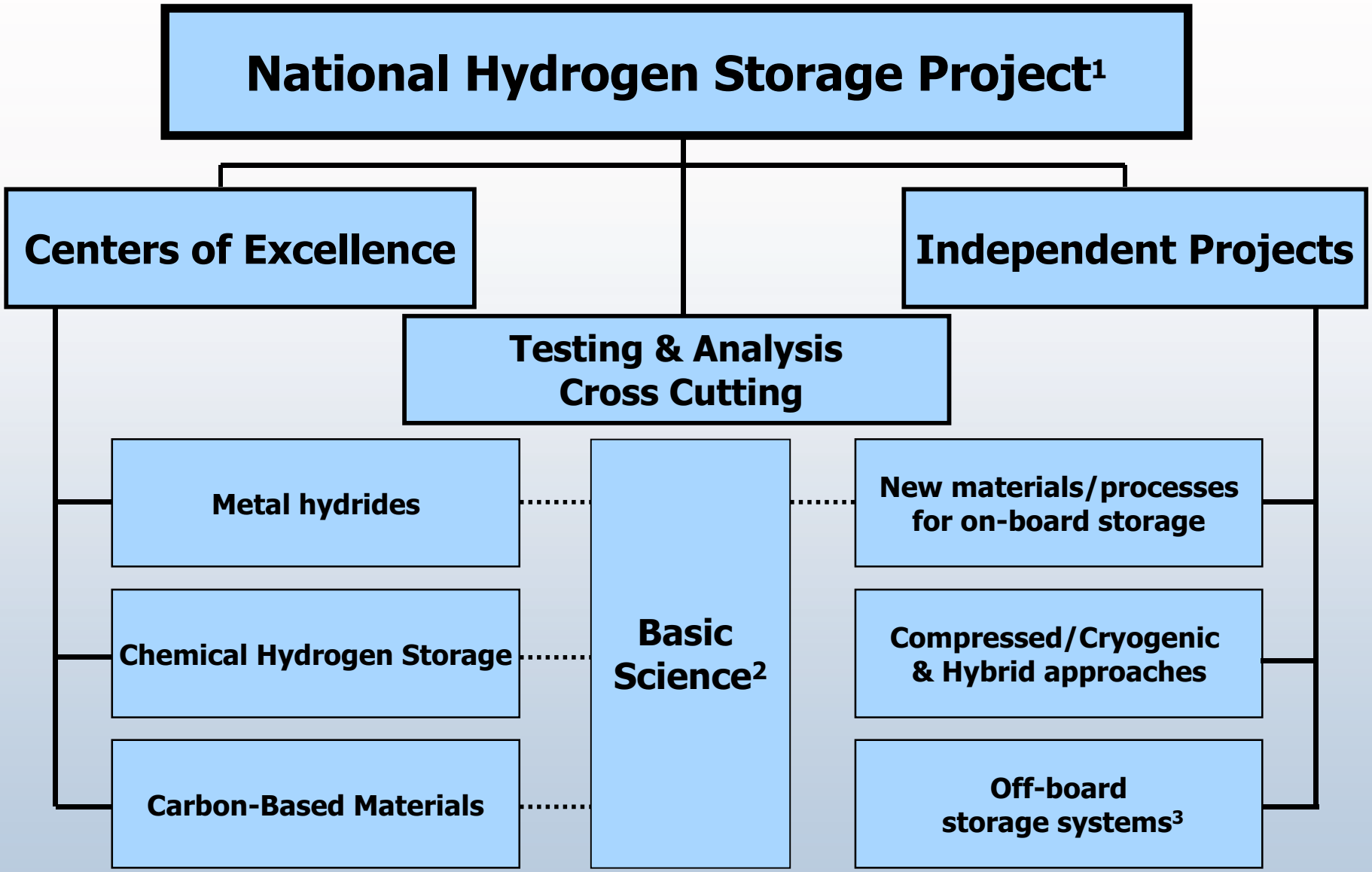
Priority Research Areas in Hydrogen Storage

Nanoscale/Novel Materials

- **Nanoscale materials have high surface areas, novel shapes, with properties much different from their 3D counterparts – especially useful for catalysts and catalyst supports.**
- **Enhanced hydrogen adsorption on high surface area nanostructures may be attained by selective manipulation of surface properties.**
- **Nanostructures also have other opportunities for use for hydrogen storage.**

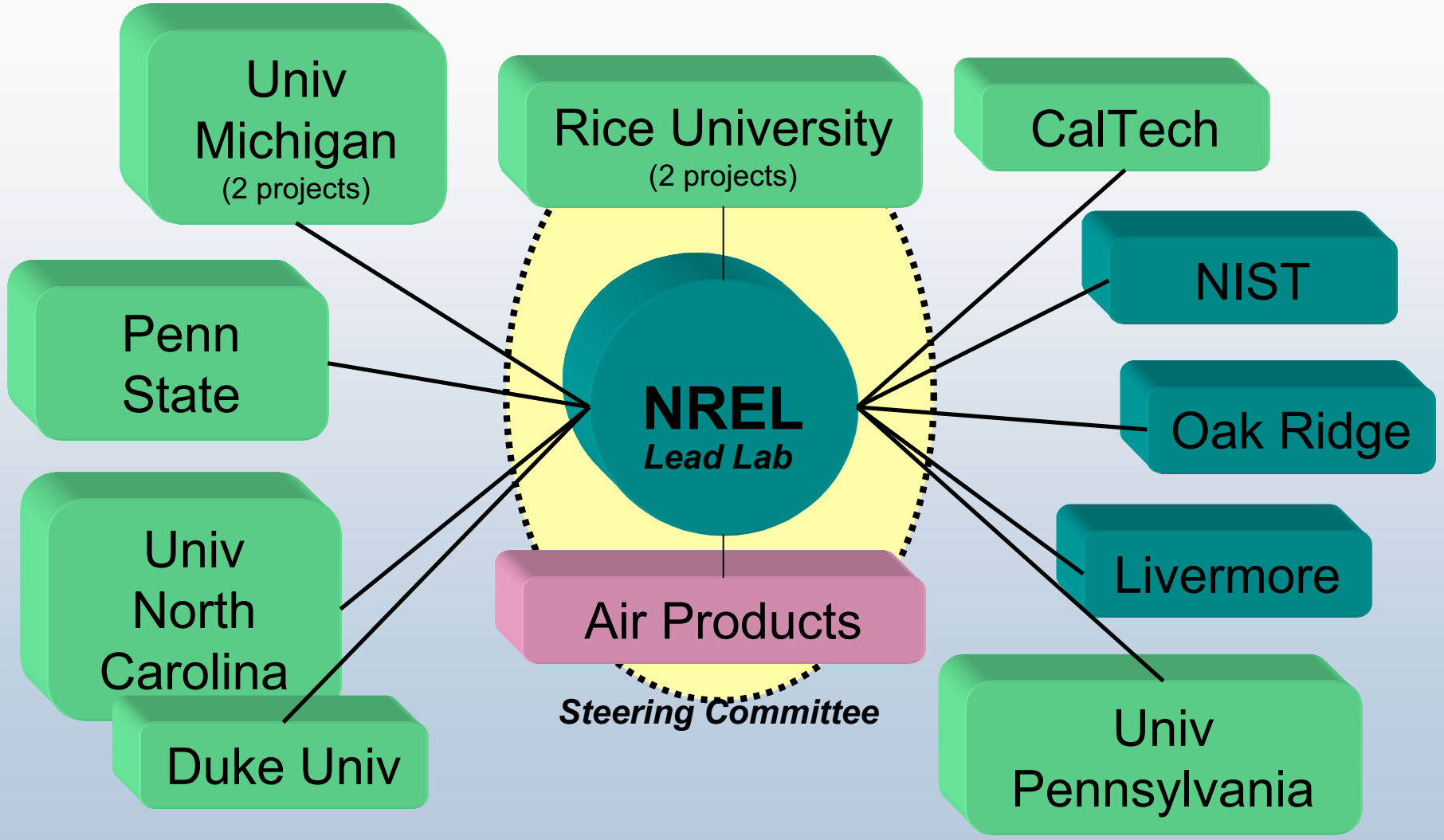


Nanostructures such as cup-stacked carbon nanofibers (less than 10nm diameter) and other high surface area structures are being developed to support tiny nanocatalyst particles (2nm) in the regions between the cups. Results obtained thus far are encouraging for specific applications.

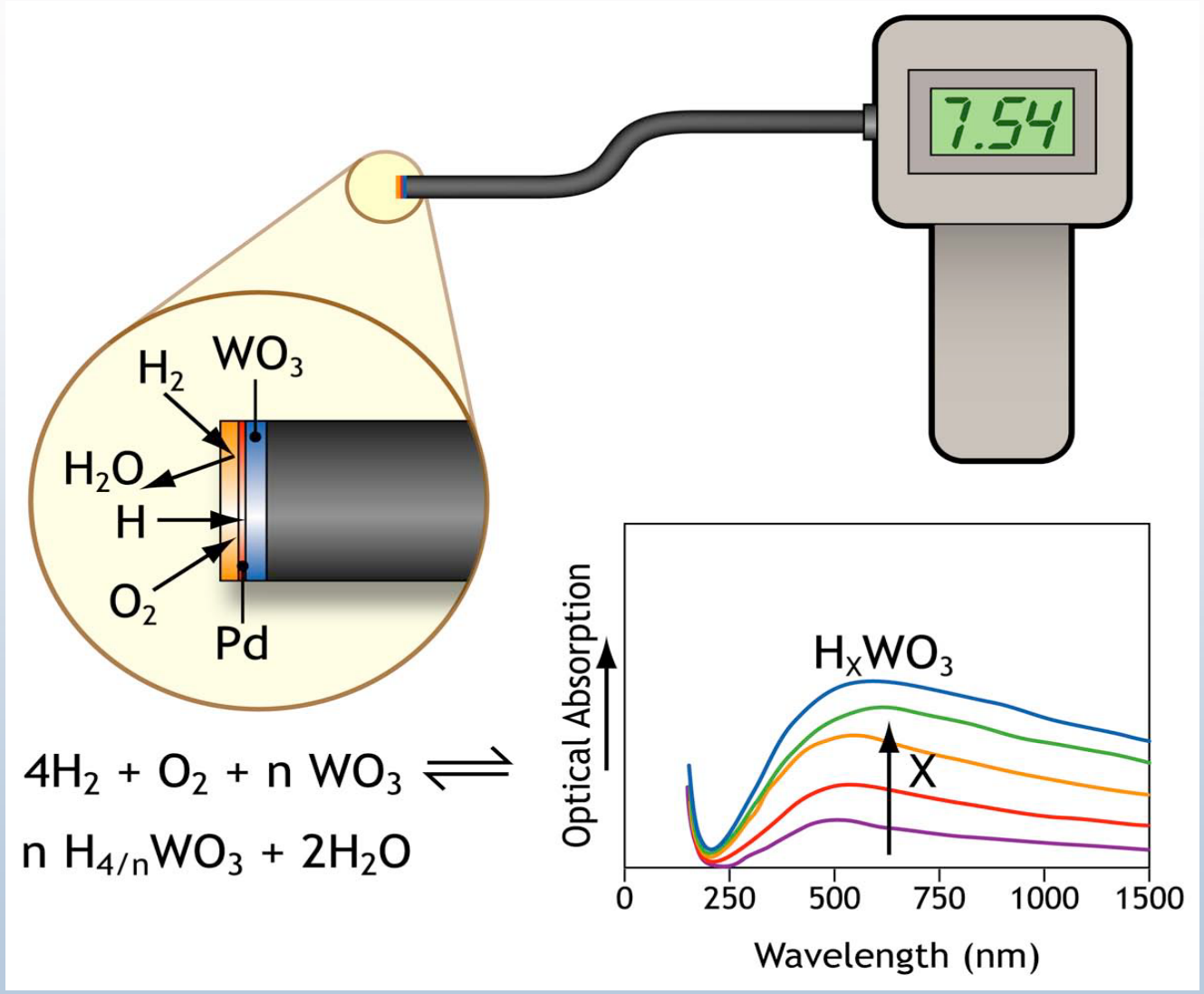


1. Coordinated by DOE Energy Efficiency and Renewable Energy, Office of Hydrogen, Fuel Cells and Infrastructure Technologies
2. Basic science for hydrogen storage conducted through DOE Office of Science, Basic Energy Sciences
3. Coordinated with Delivery program element

DOE Center of Excellence on Carbon-based Hydrogen Storage Materials



Chemochromic Hydrogen Sensors



Conclusions

- Significant advances have been made on photoelectro-chemical and photobiological approaches to generation of hydrogen from water.
- Carbon-based nanostructure materials are promising for hydrogen storage for vehicular applications.
- Major hurdles remain in increasing conversion efficiency and long-term stability while decreasing cost.
- Exciting new research opportunities are being pursued for optimizing materials and device parameters as well as developing new materials and device concepts.

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