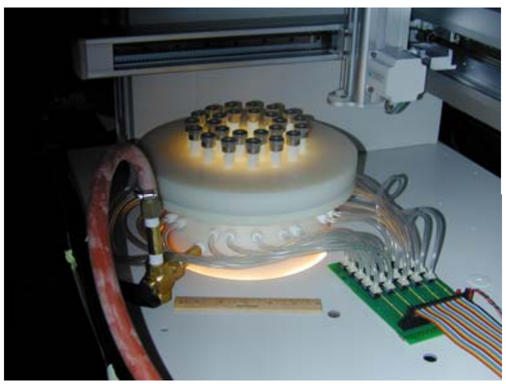
Discovery of Photocatalyst for Hydrogen Production



D. Brent MacQueen

SRI International

May23th, 2005

This presentation does not contain any proprietary or confidential information

Project ID #
PDP33

SRI International

NanoGram corporation

Team

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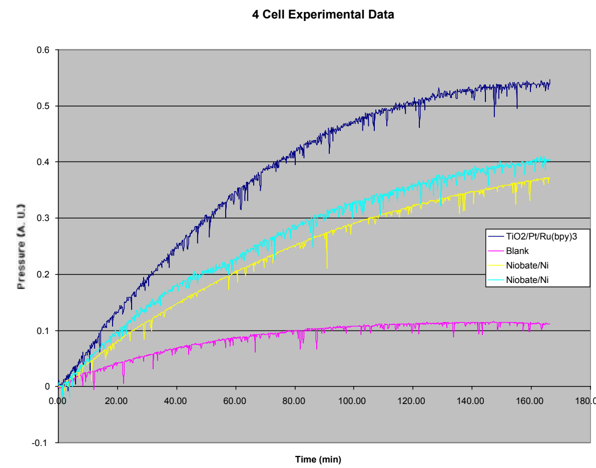
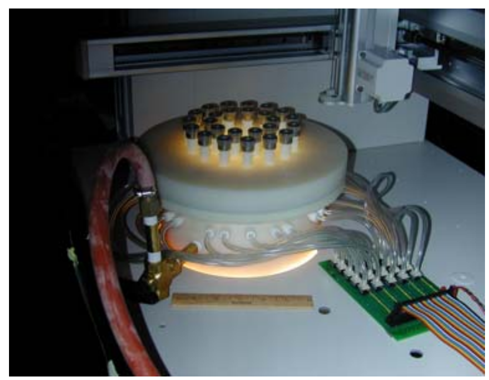
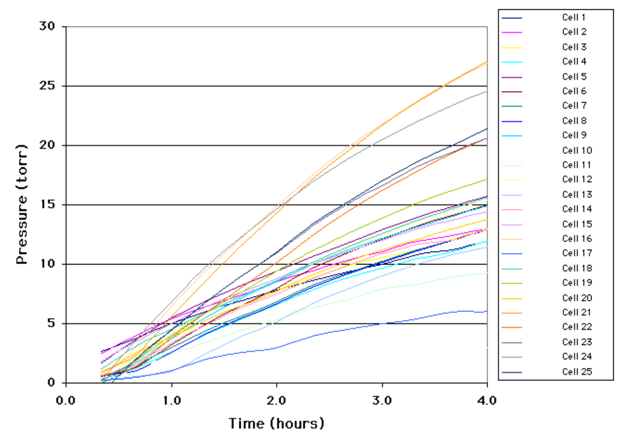
Timothy Jenks, Pres. and CEO
Nobi Kambe, VP and CTO
Ronald Mosso, VP and COO







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Overview

Timeline

- Project start 9/15/2001
- Project end 10/31/2005*
- Percent complete 88%
- **Total project funding**
  - DOE $929,532
  - Contractor $232,383
- **Funding in FY04**
  - DOE $320,000
  - Contractor $80,000
- **Funding for FY05**
  - DOE $360,000
  - Contractor $90,000

Barriers

- Barriers addressed
  - AP Materials Efficiency
  - AQ Materials Durability
  - AR Bulk Materials Synthesis

Partners

- NanoGram, Inc.

Objectives

Key Technical Barriers to PEC Hydrogen Production are Materials and Systems Engineering Related

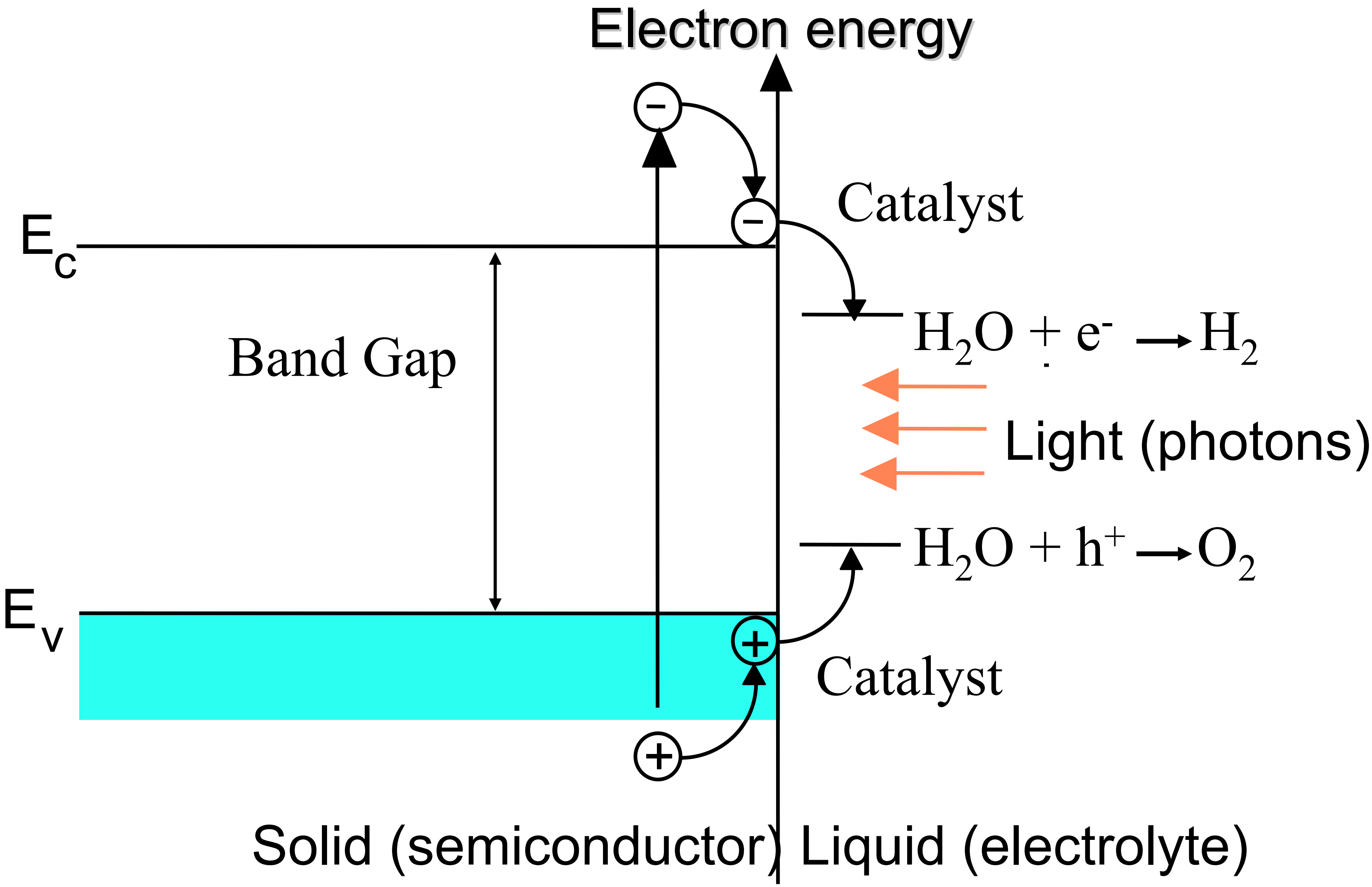
- **Efficiency (band gap and edges), Durability and Cost**

Materials need to be found that address these issues.

This project will assist in the identification of materials that directly address these barriers.

Specifically, the discovery of low cost materials with improved efficiency will be a driver to lower cost PEC hydrogen.

PEC 101



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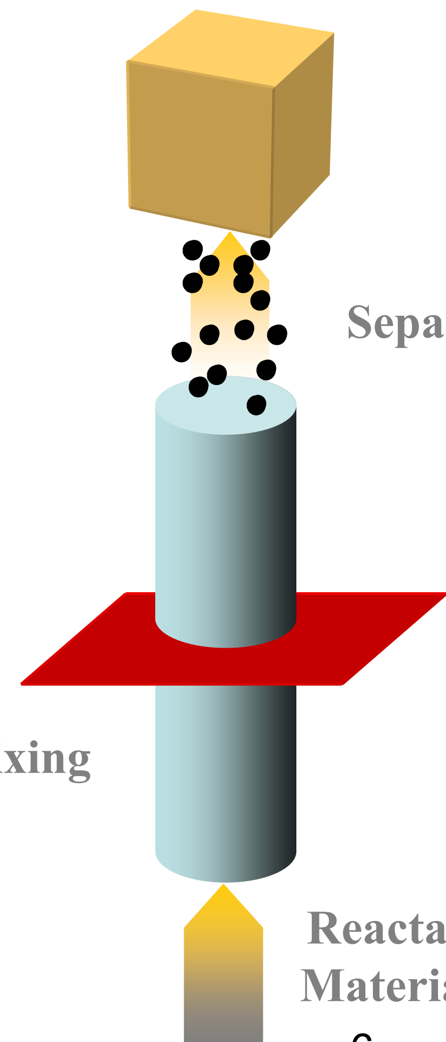
Approach
Nanoparticles Synthesis

- Wide range of precursor forms
  - Gas
  - Vapor
  - Aerosol
- Rapid heating & quench (at order of 10^5 degrees/sec)



- Huge materials portfolio
  - Crystalline inorganics
  - Multi-element compounds
  - Tightly controlled size
  - High chemical purity
  - Oxide, sulfide, nitride, metal, phosphate, carbide, silicate inorganic compounds...
  - Rare earth-doping at high concentration
  - Scalable over 1kg/hr per equipment

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Approach,
High Volume Nanoparticle Synthesis

LRD Reactor

CO_2 Laser

Controller

Scrubber System

Chemical Delivery System

- **Industrial; mass production**
  - **up to 10,000 wafers/year/system**
- **Fifth-generation technology**

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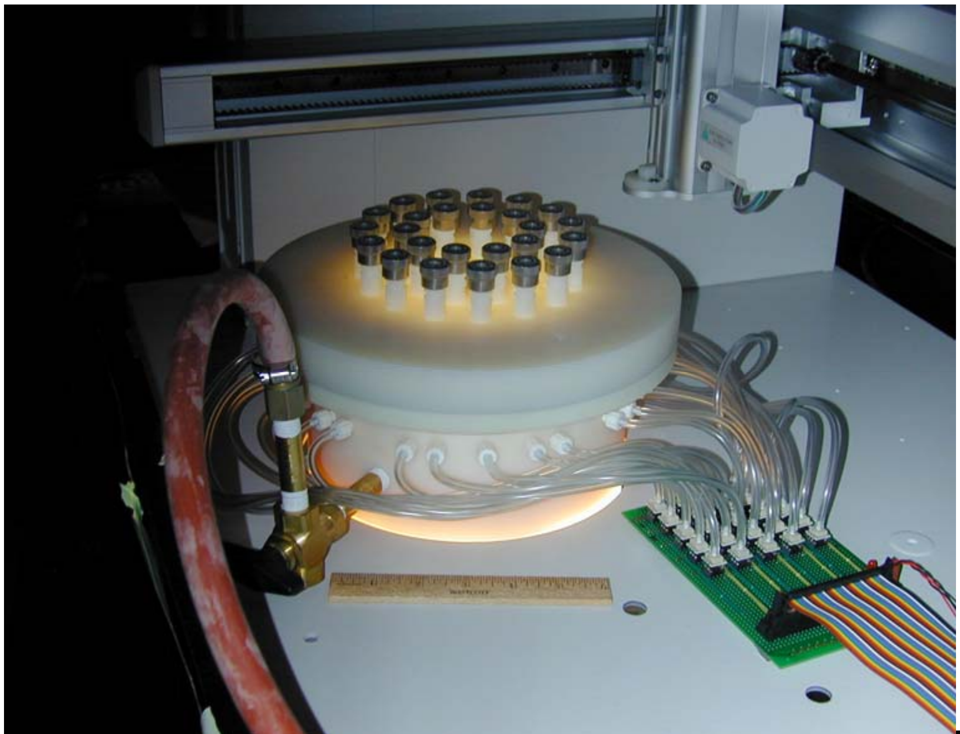
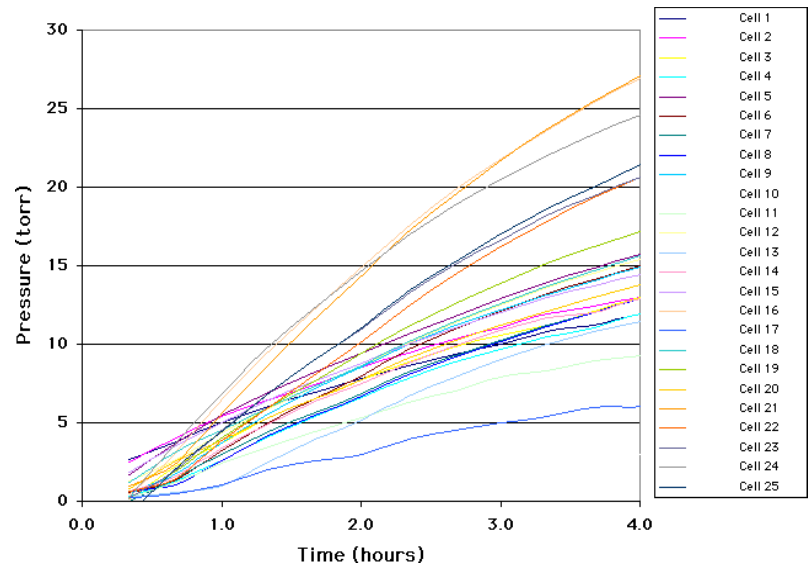
Approach
High Throughput Screening





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Approach,Modeling

Modeling Semiconductor Nano-Heterostructures

Approach

Strain-dependent K.P Hamiltonian

Step 1. Minimizing the strain energy

stable strain configuration

Step 2. Solving the Schrodinger equation

$$H = H_{k.p} + H_{strain}$$

electronic structures and wavefunctions

Reference

*C. Pryor, Phys. Rev. B **57**, 7190 (1998).*

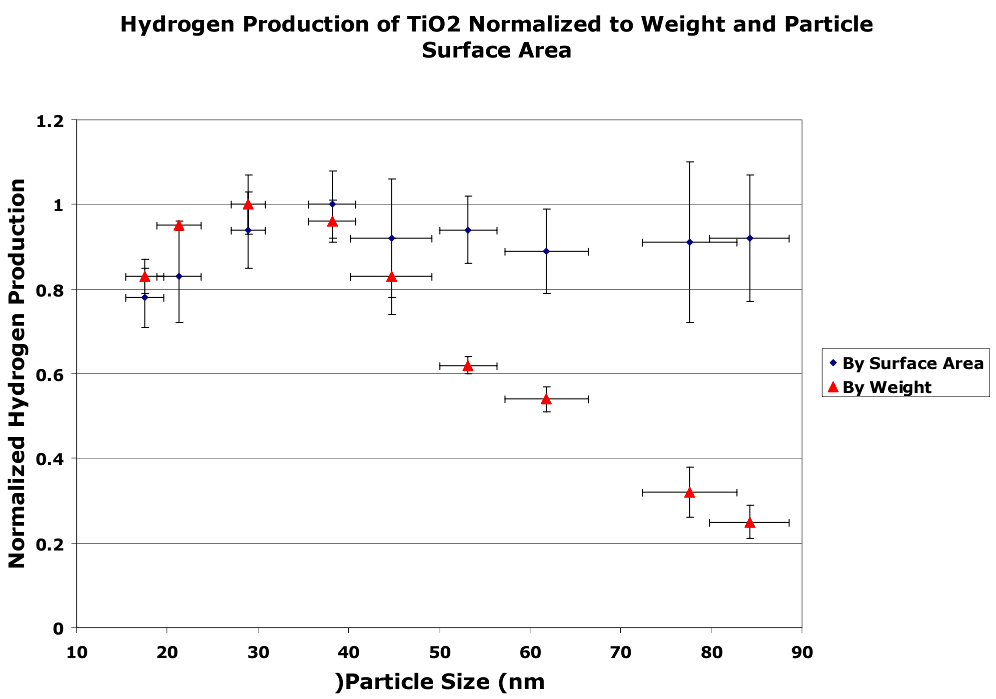
Technical Accomplishments

- TiO_xN_y and TiOS materials show improved efficiency relative to TiO_2
  - Efficiency a factor of 5-10 lower than 2008 target
  - Stability, TiOS <5% loss after 20 hours
  - Longer term, >30 days, in progress
- AgInZnSe alloy
- Constructed Solar Simulator for long term experiments
- Modeling

Results

Nanoparticles > advantage
Is surface area. However in real system use of particle requires separation step which may prevent complete utilization of nanoparticle advantage.

Nanoparticles, in a dispersion, below 30nm begin to lose activity



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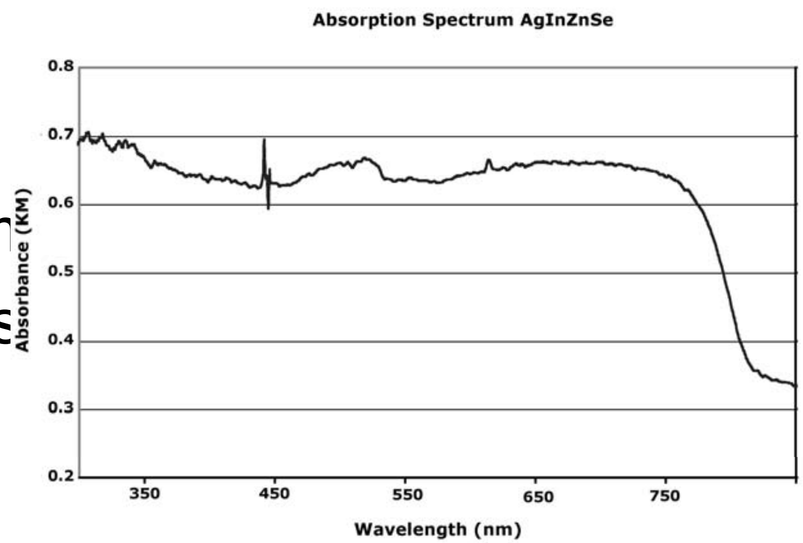
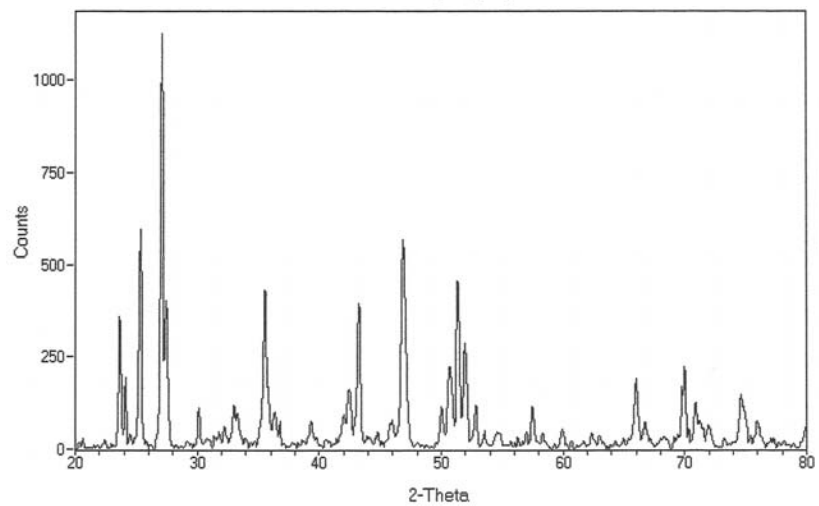
Results

(AgIn)ZnSe

- Utilizes most of Visible spectrum
- < 5% reduction in activity, 20 hrs
- Still factor of 10 below target
- Long term testing in progress





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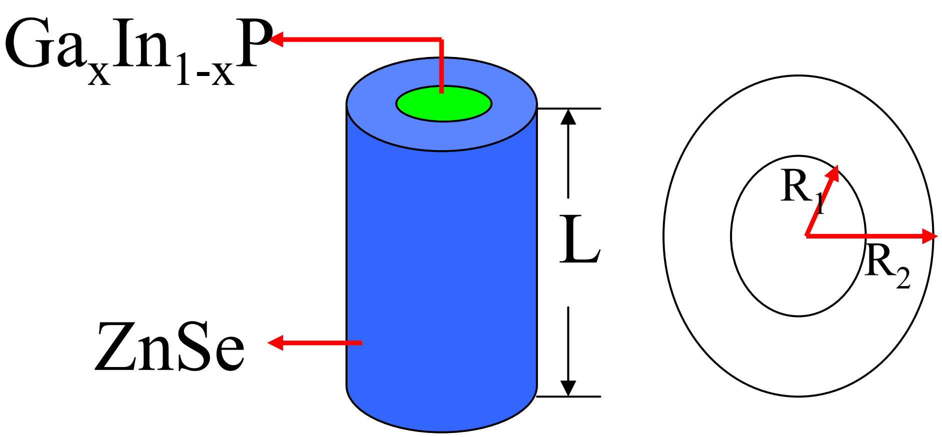
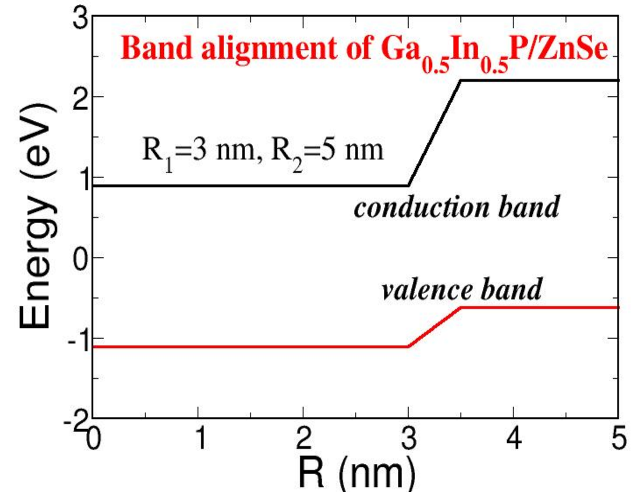
Results, Modeling





- **Separation of electrons and holes in different regions**

  Type-II band alignment ***Holes are localized in shell and electrons in core.***

- **Easy collection of both electrons and holes**
- **Stable in aqueous environment**
- **Tunable of electron and hole band edges**

  Tunable parameters: core radius (R_1), shell thickness (R_2-R_1), length (L), material composition (x)

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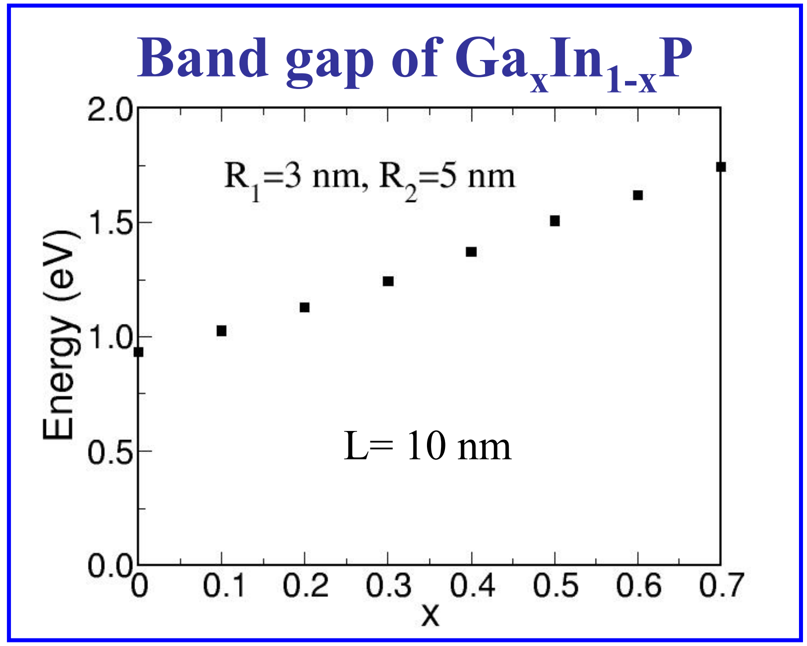
Tuning band edges of $Ga_xIn_{1-x}P/ZnSe$ Nanorods

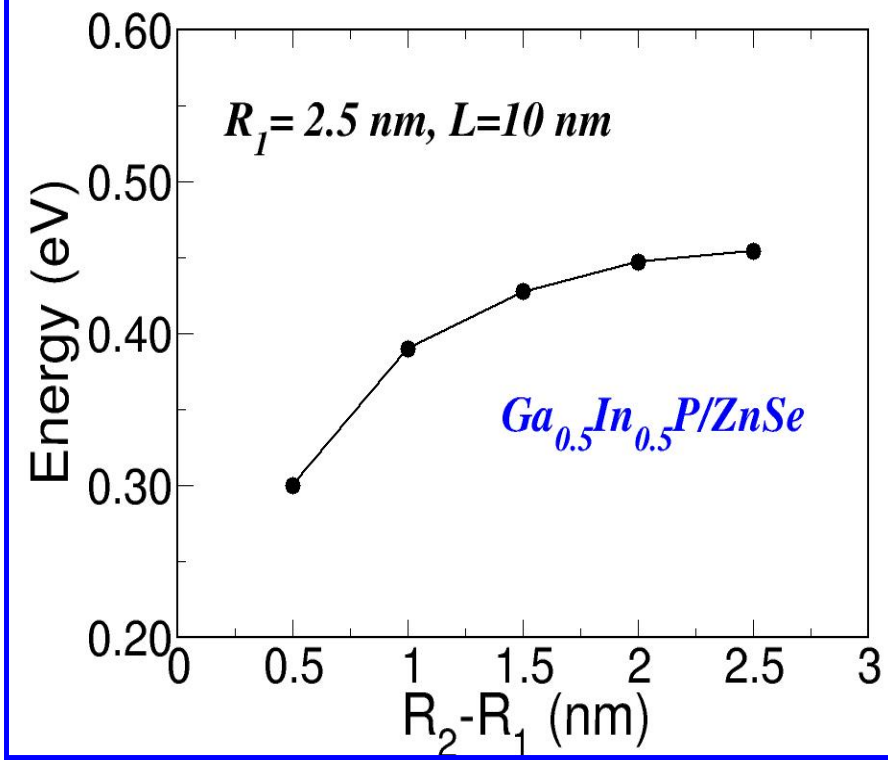
- Composition x
- Quantum confinement

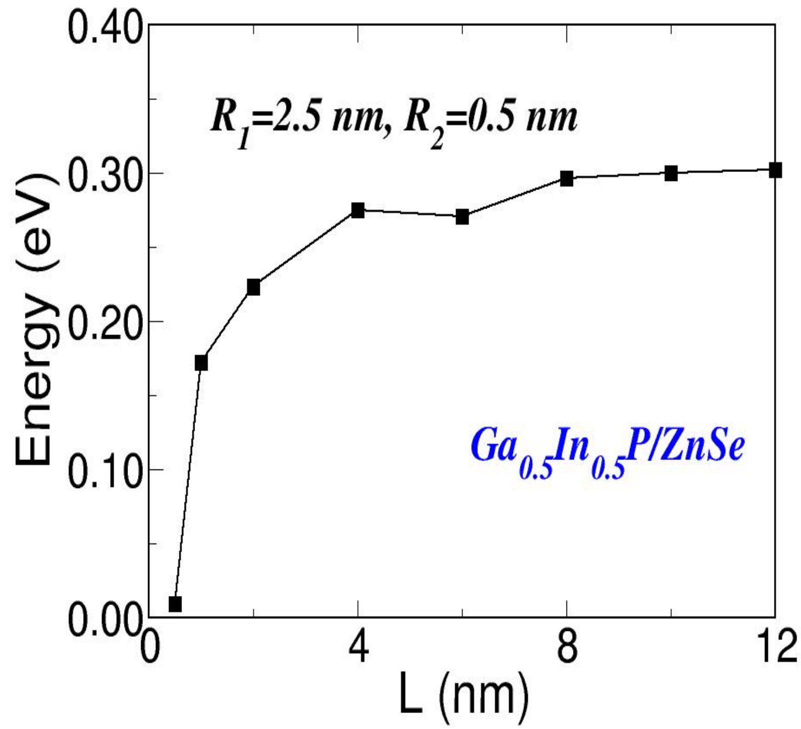
  -- Thickness of shell
  -- Length of rod



Results, Modeling

Energy of HOMO (localized in shell) with respect of band edge of core





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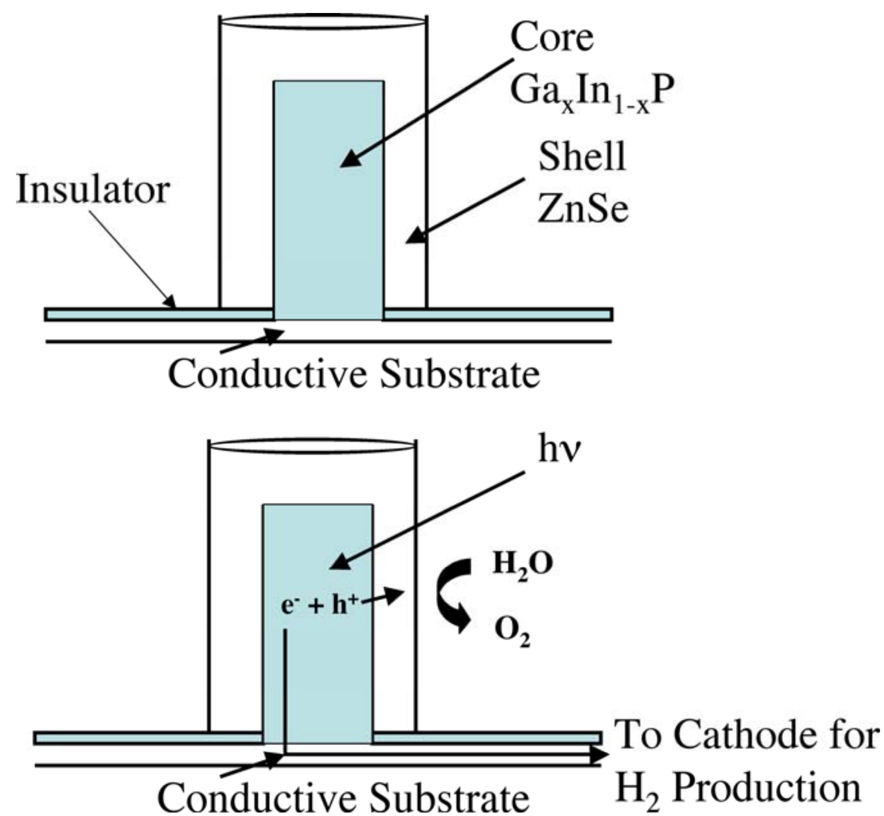
Responses to Previous Year Reviewers' Comments

- No effort to characterize some samples to understand why improvements or failures are achieved
  - Nature of high throughput analysis is to identify candidates for further study, until 2004 none were identified. Idea is to look at improved materials and discard failures
- Methanol oxidation products may interfere with hydrogen analysis
  - Hydrogen evolution is monitored by pressure transducer and actual amount is determined by gas chromatography
- Why is generation of database and modeling emphasized in phase 3?
  - The database discussed is one in which intelligent inquires can be made and requires specific software (Oracle or Microsoft for example). This requires some amount of cooperation between users that has not happened. Data is currently in Excel format.

Future Work

Characterize Mixed Phase Materials

Develop core shell structures



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Supplemental Slides

The following three slides are for the purposes of the reviewers only – they are not to be presented as part of your oral or poster presentation. They will be included in the hardcopies of your presentation that might be made for review purposes.

Publications and Presentations

Publications:

Three in preparation

Presentations:

Discovery of Photocatalyst for Hydrogen Production

NanoSig Clean Energy & Nano Catalyst Conference
August 20th 2004, SRI International

Hydrogen Safety

- Nanoparticle synthesis involves high power laser, high temperatures, potentially reactive gasses and the handling of fine particles
  - Computer controlled system has a range of interlocks for safe operation including start-up, in-process upsets, and during shutdown
  - All nanoparticle production, collection, equipment cleaning is carried out inside a fume hood
  - Reactants, and precursors are contained in exhausted gas cabinet with sensors to detect leakage
  - Respirators are used when handling nanoparticles and nanoparticle-exposed equipment
- Photolysis experiments generate hydrogen and uses light source that generates UV light
  - Sacrificial electron donor used, no Oxygen generated
  - Hydrogen Sensor (Neodym) on pressure transducer board shuts down system if H_2 concentration above 0.2% detected.
  - Light source is fully shielded