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Seminar Scoala Doctorala 2014/2105
Prof.dr.ing. Nicolae VASZILCSIN
Facultatea de Chimie Industrială şi Ingineria Mediului

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Introduction

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Methods

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Results

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Figure #1

CHART or PICTURE

Figure #2

Conclusions

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Bibliography

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Methods

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Figure #1

CHART or PICTURE

Results

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Figure #2

Conclusions

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Bibliography

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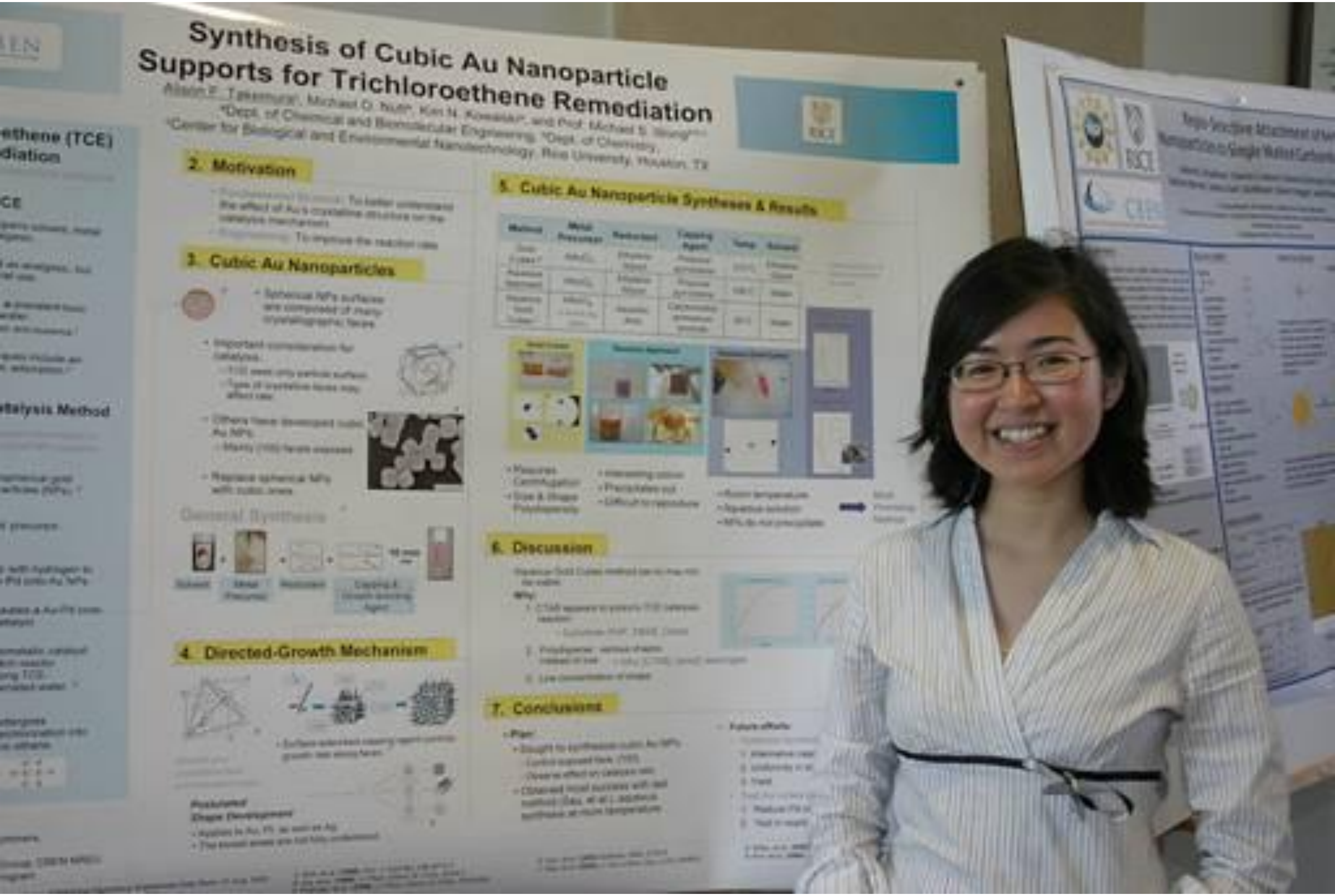
Synthesis of Cubic Au Nanoparticle Supports for Trichloroethene Remediation
2. Motivation
3. Cubic Au Nanoparticles
General Synthesis
4. Directed-Growth Mechanism
5. Cubic Au Nanoparticle Syntheses & Results
6. Discussion
7. Conclusions
Rice University, Houston, TX

Nuclear Nonproliferation Initiative

Strategic Plan

VISION: to be a recognized technical and program leader providing nuclear nonproliferation services to the nation. In so doing, the Laboratory will support the nation's goals for global security and stability; and will further international acceptance of advanced nuclear energy systems.

Step 1 of a Strategic Plan is to create and lay out a Vision of the ideal future and what the company wants to strive for. The Nuclear Nonproliferation Initiative (NNI) Vision above is an example.

Step 2 is to survey the **market "landscape"**. The table above is an element of the nonproliferation "landscape". The three regimes of Nonproliferation in the table are part of a synopsis of the nonproliferation sector. An outline of the sector in broad market details gives a general framework to explain the boundaries of the Plan, then follows a detailed analysis considering the customers and competitors. This includes a breakdown of the customers' needs and spending habits. For instance, the pie graph on the right explains the Nonproliferation Budget for DOE. Next competitors' capabilities, financial resources, and critical infrastructure are assessed. Examples from the NNI Plan are pictured on the right and include a visual illustrating the competitions' areas of specialization and a percentage pie graph showing market share.

Step 3 is to conduct an **organizational assessment**. This includes an evaluation of the business's personnel, infrastructure, financial status, and market infiltration. For example, during our assessment INL's involvement in nearly all the fuel cycle steps pictured below was taken into account.

Step 4 is to identify **Objectives**, outline Strategies, and develop ways to implement these strategies in order to accomplish the Vision all while considering the business vision, market landscape, and organization assessment. The NNI Objectives are below.

OBJECTIVES: to identify shortfalls within the current nuclear nonproliferation market; to build a solid and recognized base of sustainable research and business; to enhance complimentary research; and to promote, develop and maintain relationships.

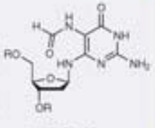
izing Sensitive Oxidative DNA Lesions

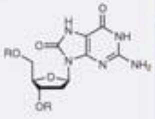
Main Oxidative DNA Lesions 8-oxodG
ydG Exhibit Strongly Different Pairing Properties

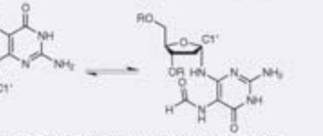
Matthias Ober, Thomas Carell
Philipps-Universität Marburg
Fachbereich Chemie, Hans-Meerwein-Str.,
35032 Marburg, Germany
m.ober@gmx.de, carell@staff.uni-marburg.de

ction

DNA due to oxidation is believed to be
e for the development of cancer and the
of aging. The formamidopyrimidine of
osine (β-FaPydG) and 7,8-dihydro-8-
uanosine (β-8-oxodG) are the two main
nduced by oxidative stress. As a mono-
G readily anomerizes in solution, im-
reospecific incorporation into oligonuc-
g machine assisted DNA synthesis.


β-FaPydG

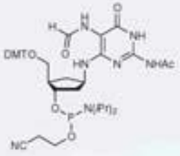

β-8oxodG



e 1: Anomerization equilibrium of FaPydG.

Goals

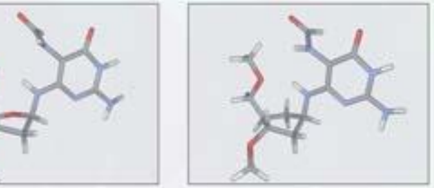
a β-FaPydG building block
ble to anomerization and
steric. The carbocyclic ana-
aPydG (β-*c*FaPydG) fulfills
ents.



The carbocyclic β-*c*FaPydG building block

β-*c*FaPydG into oligonuc-
tomated DNA synthesis.

UV-melting points of DNA
aining β-cFaPydG. Compare with the melting behavior of
s containing 8-oxodG and unmodified dG.


re 1: Geometries of FaPydG (left) and and *c*FaPydG (right).

uctures were obtained by B3LYP/6-31G* density functional
alculation with geometry optimization using Gaussian 98
Rev. A.9)

ces and Acknowledgements

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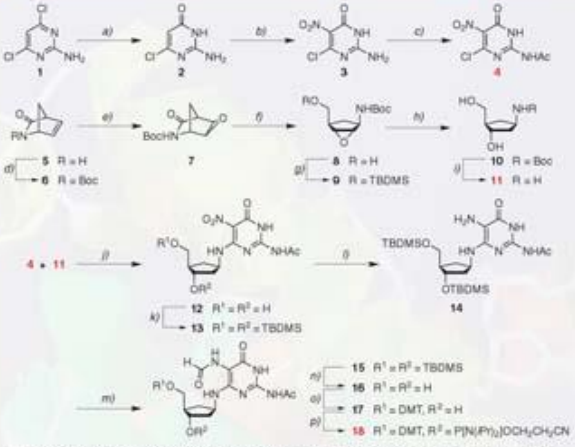
T. Carell, *Chem. Eur. J.* **2002**, *8*, 293.
ez, P. M. Cullis, *Tetrahedron Lett.* **1999**, *40*, 5783.
J. G. Muller, *Chem. Rev.* **1998**, *98*, 1109.
icture was generated from 1EBM.pdb, "Crystal structure of the human 8-
ylase (hOgg1) bound to a substrate oligonucleotide", S. D. Bruner, D. P.

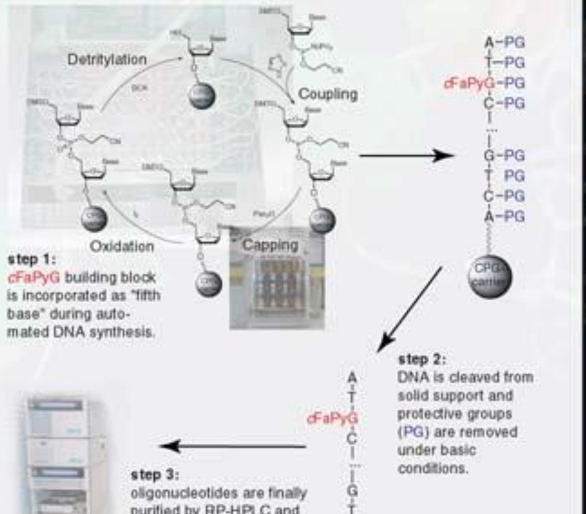
Synthesis of the *c*FaPyG building block



a) 1 N NaOH, 100 °C, 1 h, 99%, *b)* HNO_3·NO_2, H_2SO_4, 0 °C, 15 min, 85%, *c)* Ac_2O, H_2SO_4, 90 °C, 60 min, 80%, *d)* Boc_2O, DMAP, THF, 25 °C, 85%, *e)* *m*CPBA, CH_2Cl_2, 25 °C, 20 h, 72%, *f)* $NaBH_4$, MeOH, 0 °C, 0.5 h, 92%, *g)* TBDMSCl, imidazole, DMF, 25 °C, 3 h, 90%, *h)* Red-Al®, toluene, 25 °C, 3 h, 93%, *i)* H_2O, 100 °C, 14 h, 95%, *j)* DIPEA, DMF, 70 °C, 90 min, 72%, *k)* TBDMSCl, imidazole, DMF, 25 °C, 3 h, 88%, *l)* Pd/C, H_2, EtOH, 25 °C, 3 h *m)* HCOOH, DIPEA, EDC, DMF, 25 °C, 48 h, 65% for two steps, *n)* HF·pyridine, pyridine, EtOAc, 25 °C, 20 h, 97%, *o)* DMTCl, pyridine, DMAP, 0-20 °C, 3 h, 62%, *p)* DIPEA, $ClP[N(iPr)_2]OCH_2CH_2CN$, THF, 0 °C, 30 min, 86%.

Scheme 2: Synthesis of the *c*FaPydG lesion analogue phosphoramidite **18**.

Synthesis of modified oligonucleotides



step 1:
*c*FaPyG building block is incorporated as "fifth base" during automated DNA synthesis.

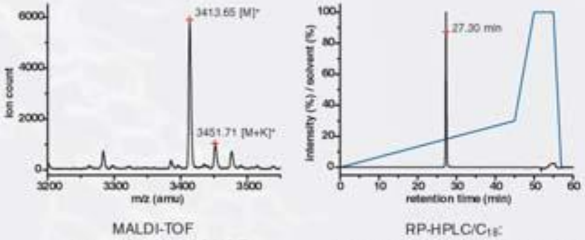
step 2:
DNA is cleaved from solid support and protective groups (PG) are removed under basic conditions.

step 3:
oligonucleotides are finally purified by RP-HPLC and

Analysis

→ The oligonucleotides had, after preparative HPLC purification, a purity of >98%.

→ A HPLC/MS/MS experiment of a total digest of the DNA proofed the incorporation of *c*FaPydG. The fragmentation patterns of the five compounds separated during chromatography were identical to the monomeric and fully deprotected nucleotides dA, dT, dC, dG and *c*FaPydG.



MALDI-TOF
m/z: exp'd 3413.0, f'd 3413.7

RP-HPLC/C_{18}:
H_2O/MeCN 0-24% (0.1 M Et_3N/AcOH)

Figure 2: Analytical data of the oligonucleotide 5'-d(GCGATXTAGCG)-3'; X=*c*FaPyG.

Results

→ Three oligonucleotides containing *c*FaPydG, 8oxodG and dG whithin the same sequence have been synthesized. These strands were hybridized with their four counterstrands containing the four canonical bases as counterbases.

5'-d(GCGATXTAGCG)-3'
3'-d(CGCTAYATCGC)-5'

→ All mismatches strongly destabilized the duplex by at least 14°C. The two lesions generally destabilized the strand. 8-oxodG paired relatively well with dC and dA.

	X = dG	X = 8oxodG	X = cFaPydG
Y = dA	33.9	46.0	16.1 (54.9)
Y = dC	51.8	45.6	36.9
Y = dG	37.9	31.4	16.1 (54.9)
Y = dT	35.8	36.7	45.0

Table 1: UV-melting points of examined DNA duplexes.

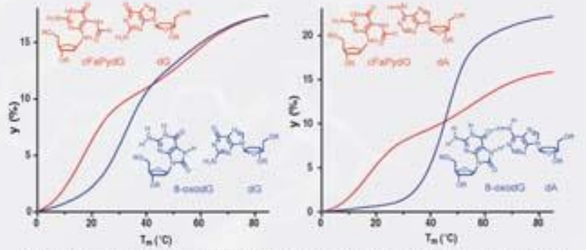
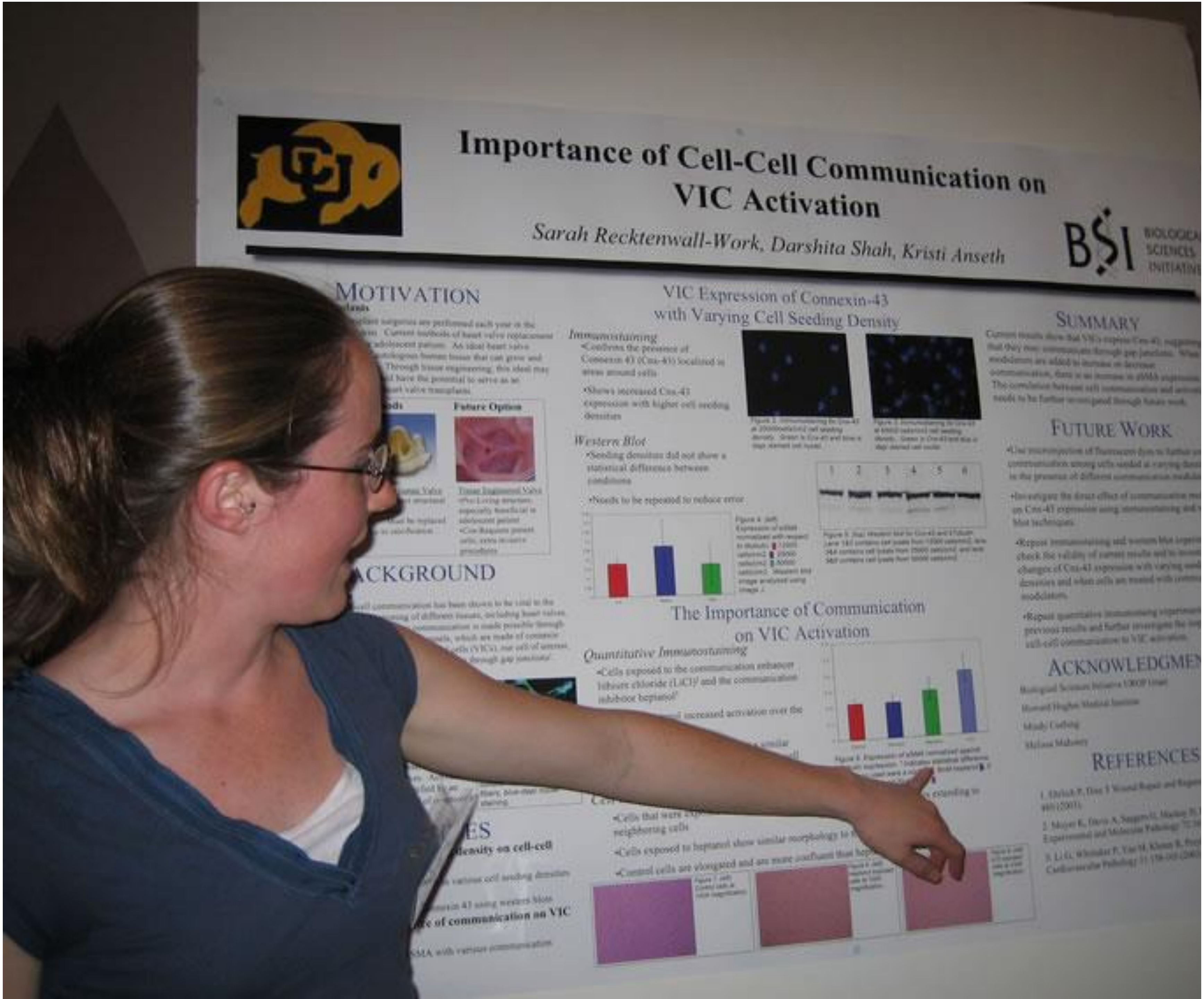


Figure 3: Melting curves of *c*FaPydG and 8-oxodG in complex with purine bases.

# Importance of Cell-Cell Communication on VIC Activation

Sarah Recktenwall-Work, Darshita Shah, Kristi Anseth

MOTIVATION

Future Option

ACKGROUND

VIC Expression of Connexin-43 with Varying Cell Seeding Density

Immunostaining

- Confirms the presence of Connexin 43 (Cnx-43) localized in areas around cells
- Shows increased Cnx-43 expression with higher cell seeding densities

Western Blot

- Seeding densities did not show a statistical difference between conditions
- Needs to be repeated to reduce error

The Importance of Communication on VIC Activation

Quantitative Immunostaining

- Cells exposed to the communication enhancer lithium chloride (LiCl)[1] and the communication inhibitor heptanol[2]

SUMMARY

FUTURE WORK

ACKNOWLEDGME

REFERENCES

HPCL The George Washington University
High Performance Computing Lab

Simulation and Evaluation of On-Chip Interconnect Architectures: 2D mesh, Spidergon, and WK-recursive Network

S. Suboh, M. Bakhouya, T. El-Ghazawi
ECE Department, The George Washington University

THE GEORGE WASHINGTON UNIVERSITY
WASHINGTON DC

ABSTRACT

Network-on-chip has been proposed as an alternative to bus-based system to achieve high performance and scalability. The topology of on-chip interconnect plays a crucial role in System on chip performance, energy, and area requirements

An on-chip interconnects architecture following WK-recursive topology is proposed, WK-recursive interconnect is analyzed and compared with two networks on chip structures: 2D Mesh and Spidergon. Comparison results for performance metrics, such as the throughput, the communication load, and latency are reported. Initial results show that WK-Recursive on-chip interconnect generally outperforms the other architectures in main performance metrics

W-recursive attractive properties

A high degree of regularity, symmetry, efficient communication, and ease of extendibility that suit network on-chip infrastructures

MOTIVATION

A new architecture, based on recursive networks, is investigated. WK-recursive network was initially proposed in [2] as a network for VLSI implementation. Since this topology has many attractive properties, such as a high degree of regularity, symmetry and an efficient communication, WK-Recursive networks have also received considerable attention in parallel computing community [3]. An interesting property that makes this structure most suitable for NoC architectures is the ability to expand the network to any arbitrary size level without reconfiguring the edges. In this paper, WK-recursive network is compared with the most commonly used topologies in NoC: 2D regular mesh and Spidergon networks. Simulation results are reported for different performance metrics.

METHODOLGY AND ANALYSIS

Performance metrics are explored for the considered topologies. A simulator developed in [4] is upgraded and modified to suit different topologies and their related parameters [5]. Recently, NoC architectures have been studied and compared based on different performance metrics such as message delay, throughput and network load [1, 5, 6]. In our study, topologies of 16-nodes of 2D mesh, Spidergon, and WK(4,2) are constructed (see figure 1). WK-Recursive network with amplitude W and level L, is denoted by a WK(W,L) [3].

To analyze and compare the above interconnect topologies, the following performance metrics are considered: latency, flits loss, aggregated throughput, and communication load [4, 5]. The considered values of the injection rate are 70, 100, 130, 160, and 190Mbps. The size of the buffer is fixed to 32 flits. Figure 2 compares the latency of the three topologies when a heavy traffic is applied. In this figure, 2D mesh and Spidergon show higher maximum latency compared to the WK-recursive topology. Figure 3 shows the variation of aggregated throughput under different injection rates. Wk-recursive interconnect outperforms the rest of the topologies when the injection rate is less that 160Mbps because of lower loss rate as depicted in figure 4. Figure 5 shows that WK outperforms the rest of topologies in the communication load.

WK-recursive Network: An overview

The communication between processors was the key issue to many parallel algorithms. It was considered as one of the most important issues for interconnection networks. It is known that the topology plays a crucial role to address this issue. Several topologies, such as Mesh and 2D torus have been proposed in the field of parallel computing and adopted in NoC research [5]. Recently, an attractive topology called WK-recursive network has received considerable attention in parallel community due to its many favorable properties, such as high degree of regularity, symmetry and efficient communication, scalability, and ease of extendibility [4]. This topology has been initially proposed for VLSI implementations [1] and later adapted as a network topology for parallel computing [2, 3, 4].
One of the interesting properties of this structure is its ease of expandability; the WK-recursive network can be constructed hierarchically by grouping basic modules. More precisely, a WK-recursive network with amplitude W and level L, denoted by a WK(W,L), can be recursively constructed . A WK(W, 0) is a vertex with W free edges. A WK(W, 1) is a W-vertex complete graph that is denoted by a KW. Each vertex has one free edge and W-1 edges that are used for connecting to the other vertices. A WK(W, H) consists of W copies of WK(W, H-1) as supervertices and the W supervertices are connected as a KW, where . By induction, WK(W,L) has WL vertices and W free edges. Consequently, for any specified degree W, WK-Recursive networks can be expanded to an arbitrary level L without reconfiguring the edges. The structures of a WK(4, 0), a WK(4, 1), and WK(4, 2) are depicted in figure to the right

RESULTS

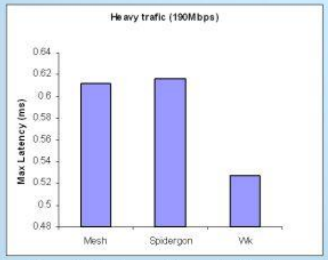


Figure 2. Maximum latency for the three topologies.

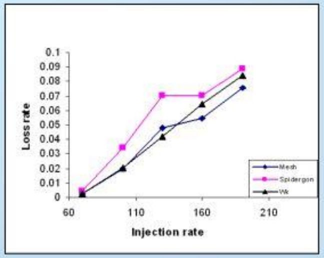


Figure 2. Variation of loss rate vs. injection rate

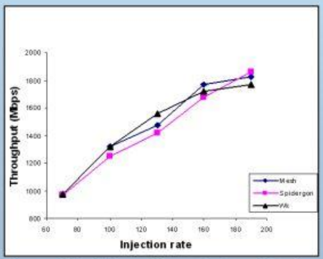


Figure 3. Variation of throughput vs. injection rate

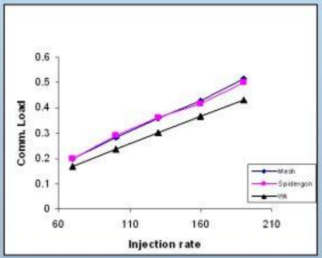
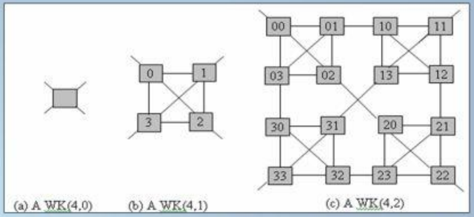


Figure 5. Variation of communication load vs. injection rate



The structures of (a) WK(4, 0), (b) WK(4, 1), and (c) WK(4, 2)

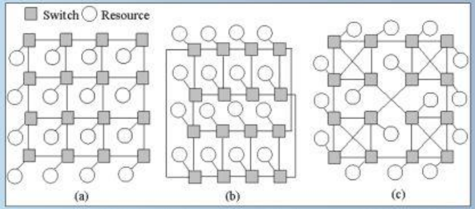


Figure 1. NoC Topologies on the chip layout: (a) 2D Mesh, (b) Spidergon, (c) WK

CONCLUSIONS

We proposed a NoC interconnect based on the WK recursive network. A simulation study aimed at exploring this on-chip interconnect for NoC is conducted. The study compares this topology with two NoC architectures: 2D regular mesh and Spidergon. Selected configurations of these architectures have been constructed and simulated to compare main performance metrics such as packet losses, throughput, message delay, and network load. WK-recursive interconnect generally outperforms Mesh and Spidergon topologies because of its regular and stable behavior at the nodes' level, since it has lower latency and lower communication load. However, when heavy traffic is applied a slight increase in drop probability is appearing. Other important metrics, such as energy consumption and area requirements, is an ongoing work.

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[8] L. Bononi and N. Concer, "Simulation and Analysis of Network on Ship Architectures: Ring, Spidergon, and 2D Mesh", Proceedings of DATE'06, 2006.

Site Characterization Projects for Contaminant Hydrogeology

Jean M. Bahr, University of Wisconsin - Madison

Motivations for the project

- Synthesis of geologic, hydrologic and contaminant data
- Examples of data available for actual sites
- Data uncertainty
- Data gaps
- Multiple and differing interpretations

Background Information Provided to Students

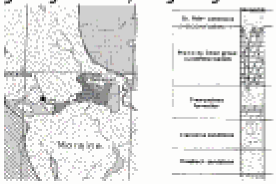
- History of suspected source and/or detected plume

Chips'N Dips (CND) Corporation has been attempting to develop silicon wafers as a snack treat. Unfortunately, during the early stages of product development and manufacturing, housekeeping operations at the plant were rather careless and a number of TCE spills occurred. By the time a groundwater plume was discovered in 1983, it had moved beyond the CND property boundary.

- Critical questions to be answered by student "consultants"

It is now early 2004 and you have been called in as a consultant to assess the effectiveness of the existing remedial measures at the site and to recommend additional measures if necessary.

- Regional geologic and hydrogeologic setting

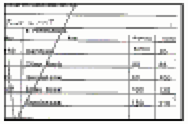


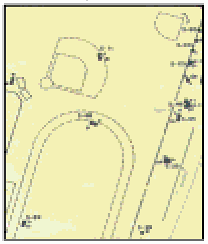
Data Set for Student Interpretation

- Geologic/geotechnical boring logs from monitoring wells (nested if possible) and nearby private or municipal wells
- Base Map

Geotech log

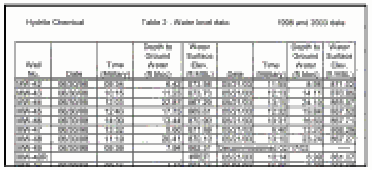
Private well

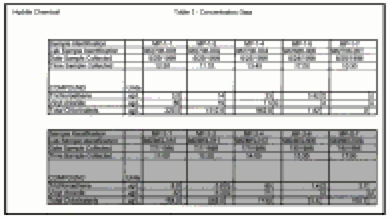




Other Essential Data

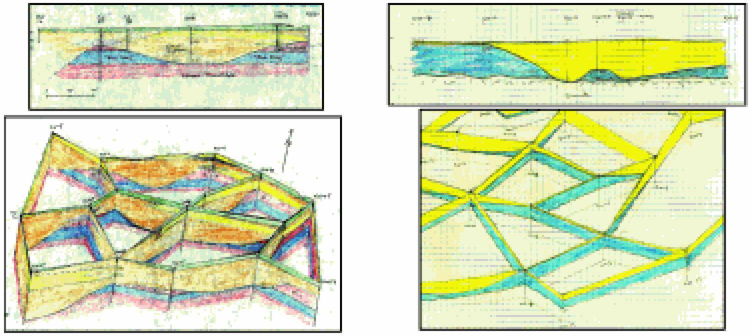
- Water level data (from multiple years or seasons if possible)
- Contaminant concentration data (from sampling rounds at similar times as water levels)





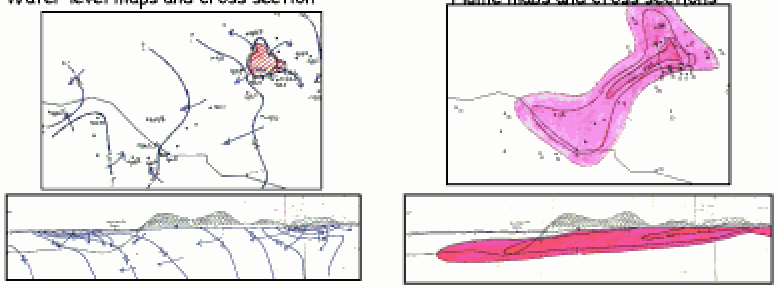
Interpretive Documents

- Geologic/hydrostratigraphic cross sections and a fence diagram

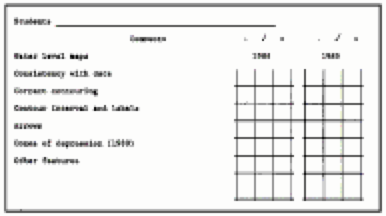


Additional Interpretive Documents

- Water level maps and cross section
- Plume maps and cross sections

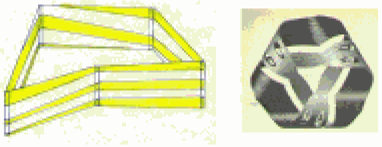


Checklists for assessing and comparing groups



Useful Lessons 1

3D interpretations should be geologically reasonable



What's wrong with this fence diagram?

Useful Lessons 2

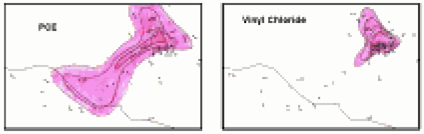
Water level and dissolved plume maps should be generally consistent

Useful Lessons 3

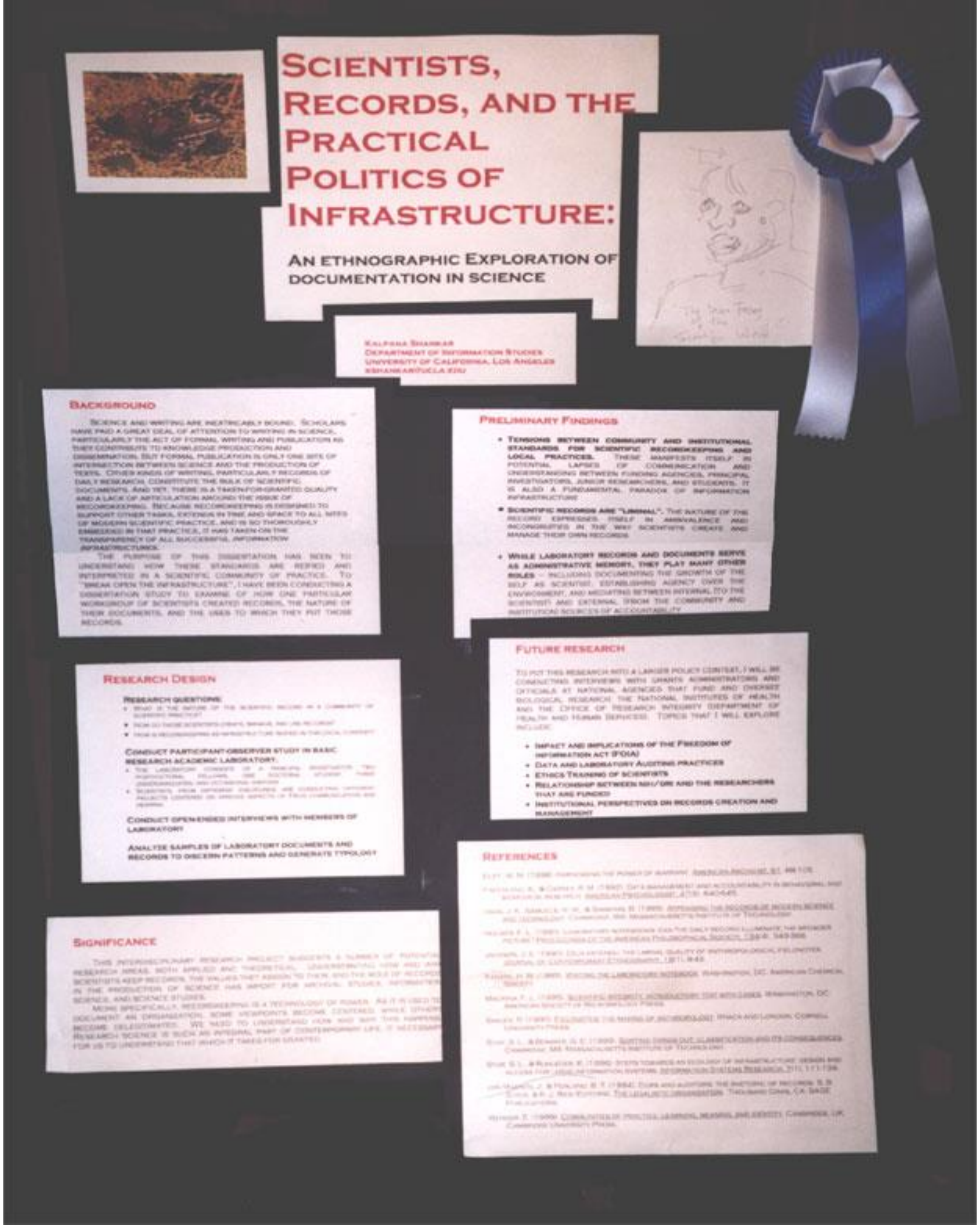
Water levels can change over time

Useful Lessons 4

Retardation and/or Degradation



Note: Examples in this poster are from a number of different site projects that have been



SCIENTISTS, RECORDS, AND THE PRACTICAL POLITICS OF INFRASTRUCTURE:

AN ETHNOGRAPHIC EXPLORATION OF DOCUMENTATION IN SCIENCE

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BACKGROUND

SCIENCE AND WRITING ARE INEXTRICABLY BOUND. SCHOLARS HAVE PAID A GREAT DEAL OF ATTENTION TO WRITING IN SCIENCE, PARTICULARLY THE ACT OF FORMAL WRITING AND PUBLICATION AS THEY CONTRIBUTE TO KNOWLEDGE PRODUCTION AND DISSEMINATION. BUT FORMAL PUBLICATION IS ONLY ONE SITE OF INTERSECTION BETWEEN SCIENCE AND THE PRODUCTION OF TEXTS. OTHER KINDS OF WRITING, PARTICULARLY RECORDS OF DAILY RESEARCH, CONSTITUTE THE BULK OF SCIENTIFIC DOCUMENTS. AND YET, THERE IS A TAKEN-FOR-GRANTED QUALITY AND A LACK OF ARTICULATION AROUND THE ISSUE OF RECORDKEEPING. BECAUSE RECORDKEEPING IS DESIGNED TO SUPPORT OTHER TASKS, EXTENDS IN TIME AND SPACE TO ALL SITES OF MODERN SCIENTIFIC PRACTICE, AND IS SO THOROUGHLY EMBEDDED IN THAT PRACTICE, IT HAS TAKEN ON THE TRANSPARENCY OF ALL SUCCESSFUL INFORMATION INFRASTRUCTURES.

THE PURPOSE OF THIS DISSERTATION HAS BEEN TO UNDERSTAND HOW THESE STANDARDS ARE REIFIED AND INTERPRETED IN A SCIENTIFIC COMMUNITY OF PRACTICE. TO "BREAK OPEN THE INFRASTRUCTURE", I HAVE BEEN CONDUCTING A DISSERTATION STUDY TO EXAMINE OF HOW ONE PARTICULAR WORKGROUP OF SCIENTISTS CREATED RECORDS, THE NATURE OF THEIR DOCUMENTS, AND THE USES TO WHICH THEY PUT THOSE RECORDS.

RESEARCH DESIGN

RESEARCH QUESTIONS:

CONDUCT PARTICIPANT-OBSERVER STUDY IN BASIC RESEARCH ACADEMIC LABORATORY.

CONDUCT OPEN-ENDED INTERVIEWS WITH MEMBERS OF LABORATORY

ANALYZE SAMPLES OF LABORATORY DOCUMENTS AND RECORDS TO DISCERN PATTERNS AND GENERATE TYPOLOGY

SIGNIFICANCE

THIS INTERDISCIPLINARY RESEARCH PROJECT SUGGESTS A NUMBER OF POTENTIA RESEARCH AREAS, BOTH APPLIED AND THEORETICAL. UNDERSTANDING HOW AND WH SCIENTISTS KEEP RECORDS, THE VALUES THEY ASSIGN TO THEM, AND THE ROLE OF RECORD IN THE PRODUCTION OF SCIENCE HAS IMPORT FOR ARCHIVAL STUDIES, INFORMATIO SCIENCE, AND SCIENCE STUDIES.

MORE SPECIFICALLY, RECORDKEEPING IS A TECHNOLOGY OF POWER. AS IT IS USED T DOCUMENT AN ORGANIZATION, SOME VIEWPOINTS BECOME CENTERED, WHILE OTHER BECOME DELEGITIMATED. WE NEED TO UNDERSTAND HOW AND WHY THIS HAPPEN RESEARCH SCIENCE IS SUCH AN INTEGRAL PART OF CONTEMPORARY LIFE, IT NECESSAR FOR US TO UNDERSTAND THAT WHICH IT TAKES FOR GRANTED.

PRELIMINARY FINDINGS

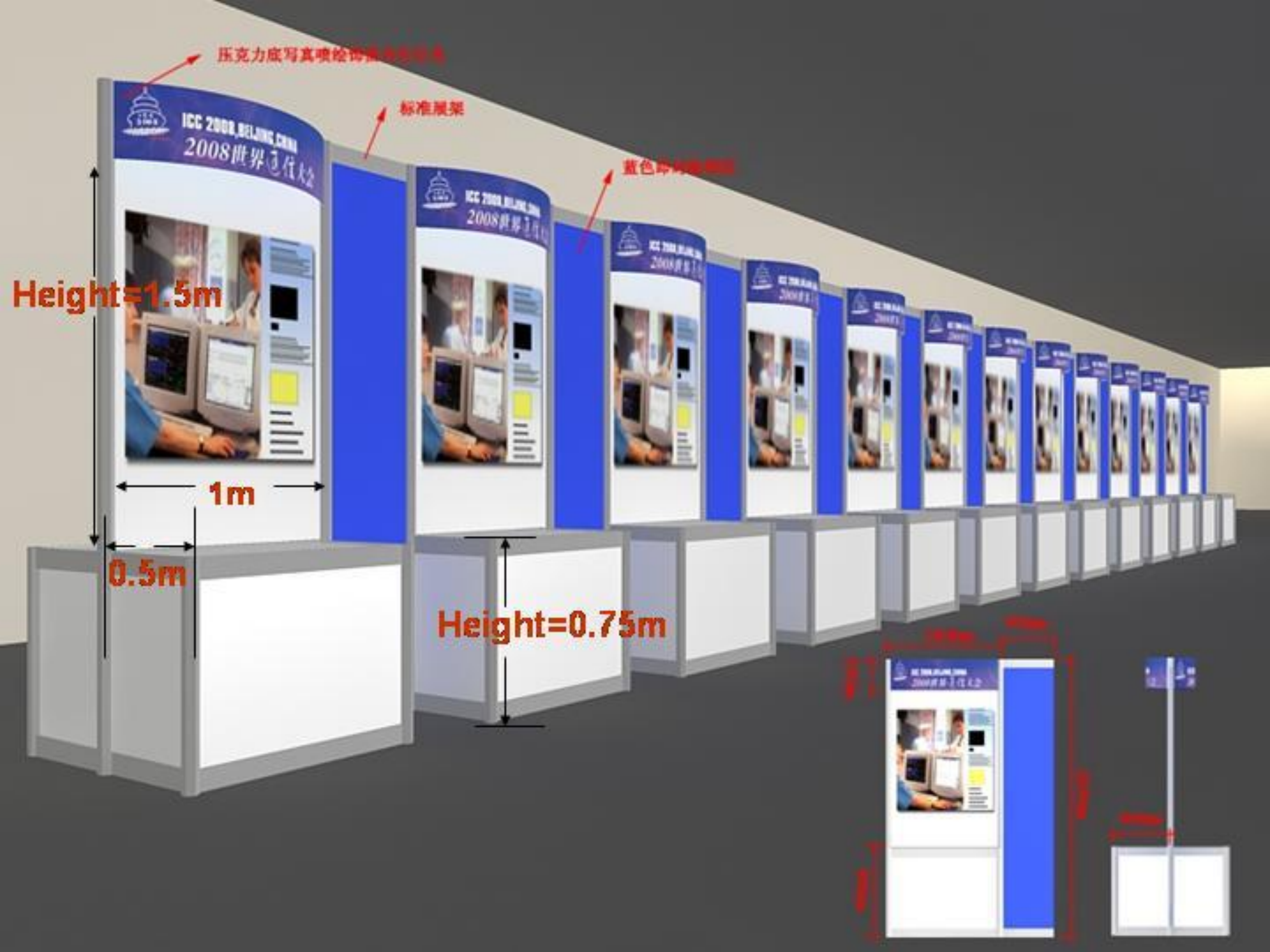
- **TENSIONS BETWEEN COMMUNITY AND INSTITUTIONAL STANDARDS FOR SCIENTIFIC RECORDKEEPING AND LOCAL PRACTICES.** THESE MANIFESTS ITSELF IN POTENTIAL LAPSES OF COMMUNICATION AND UNDERSTANDING BETWEEN FUNDING AGENCIES, PRINCIPAL INVESTIGATORS, JUNIOR RESEARCHERS, AND STUDENTS. IT IS ALSO A FUNDAMENTAL PARADOX OF INFORMATION INFRASTRUCTURE
- **SCIENTIFIC RECORDS ARE "LIMINAL".** THE NATURE OF THE RECORD EXPRESSES ITSELF IN AMBIVALENCE AND INCONGRUITIES IN THE WAY SCIENTISTS CREATE AND MANAGE THEIR OWN RECORDS
- **WHILE LABORATORY RECORDS AND DOCUMENTS SERVE AS ADMINISTRATIVE MEMORY, THEY PLAY MANY OTHER ROLES** – INCLUDING DOCUMENTING THE GROWTH OF THE SELF AS SCIENTIST, ESTABLISHING AGENCY OVER THE ENVIRONMENT, AND MEDIATING BETWEEN INTERNAL (TO THE SCIENTIST) AND EXTERNAL (FROM THE COMMUNITY AND INSTITUTION) SOURCES OF ACCOUNTABILITY

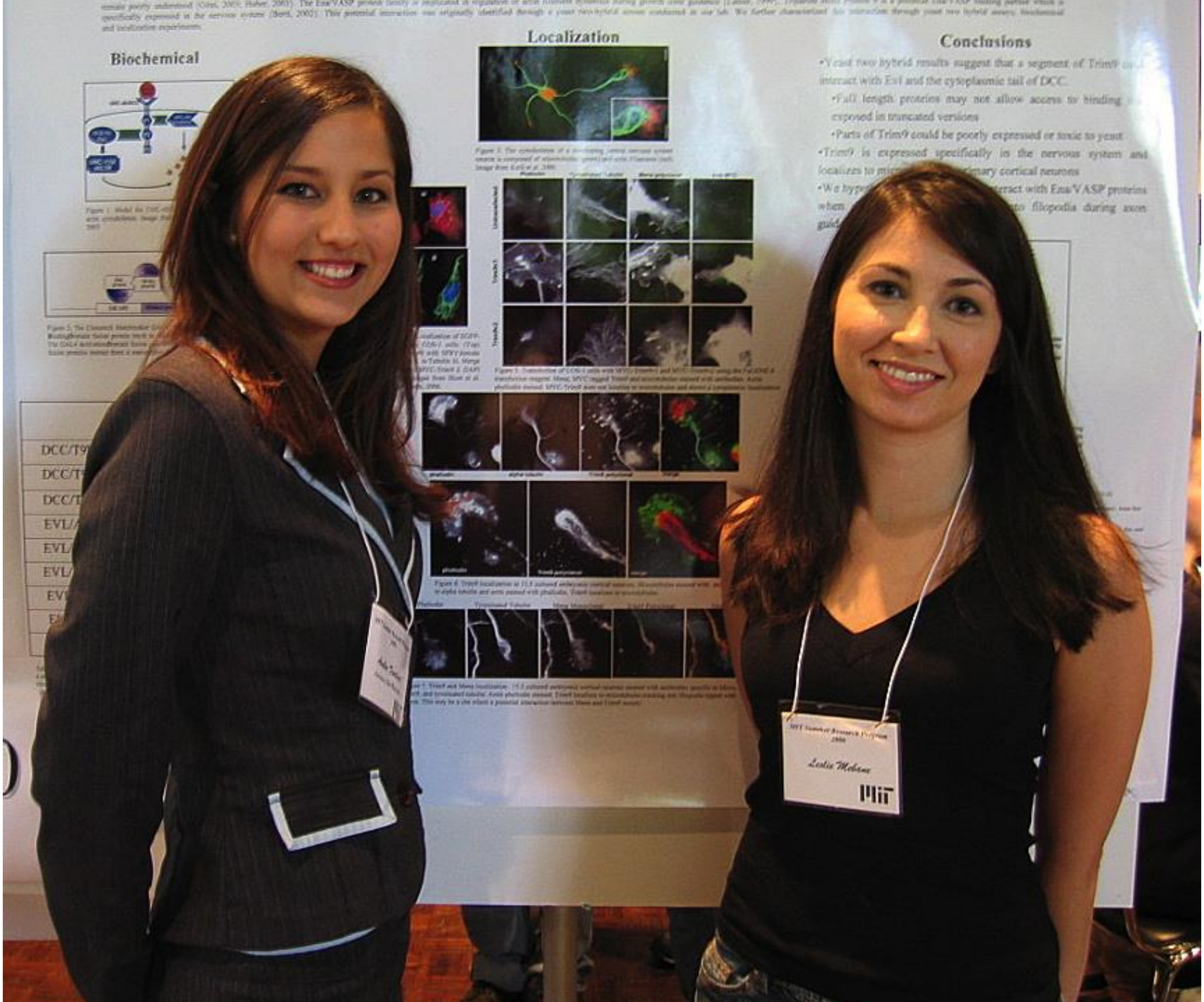
FUTURE RESEARCH

TO PUT THIS RESEARCH INTO A LARGER POLICY CONTEXT, I WILL BE CONDUCTING INTERVIEWS WITH GRANTS ADMINISTRATORS AND OFFICIALS AT NATIONAL AGENCIES THAT FUND AND OVERSEE BIOLOGICAL RESEARCH: THE NATIONAL INSTITUTES OF HEALTH AND THE OFFICE OF RESEARCH INTEGRITY, DEPARTMENT OF HEALTH AND HUMAN SERVICES. TOPICS THAT I WILL EXPLORE INCLUDE:

- **IMPACT AND IMPLICATIONS OF THE FREEDOM OF INFORMATION ACT (FOIA)**
- **DATA AND LABORATORY AUDITING PRACTICES**
- **ETHICS TRAINING OF SCIENTISTS**
- **RELATIONSHIP BETWEEN NIH/ORI AND THE RESEARCHERS THAT ARE FUNDED**
- **INSTITUTIONAL PERSPECTIVES ON RECORDS CREATION AND MANAGEMENT**

REFERENCES


压克力底写真喷绘
标准展架
蓝色
ICC 2008,BEIJING,CHINA
2008世界通信大会
Height=1.5m
1m
0.5m
Height=0.75m


Biochemical
Localization
Conclusions
MIT Summer Research Program 2006
Leslie Mebane
MIT

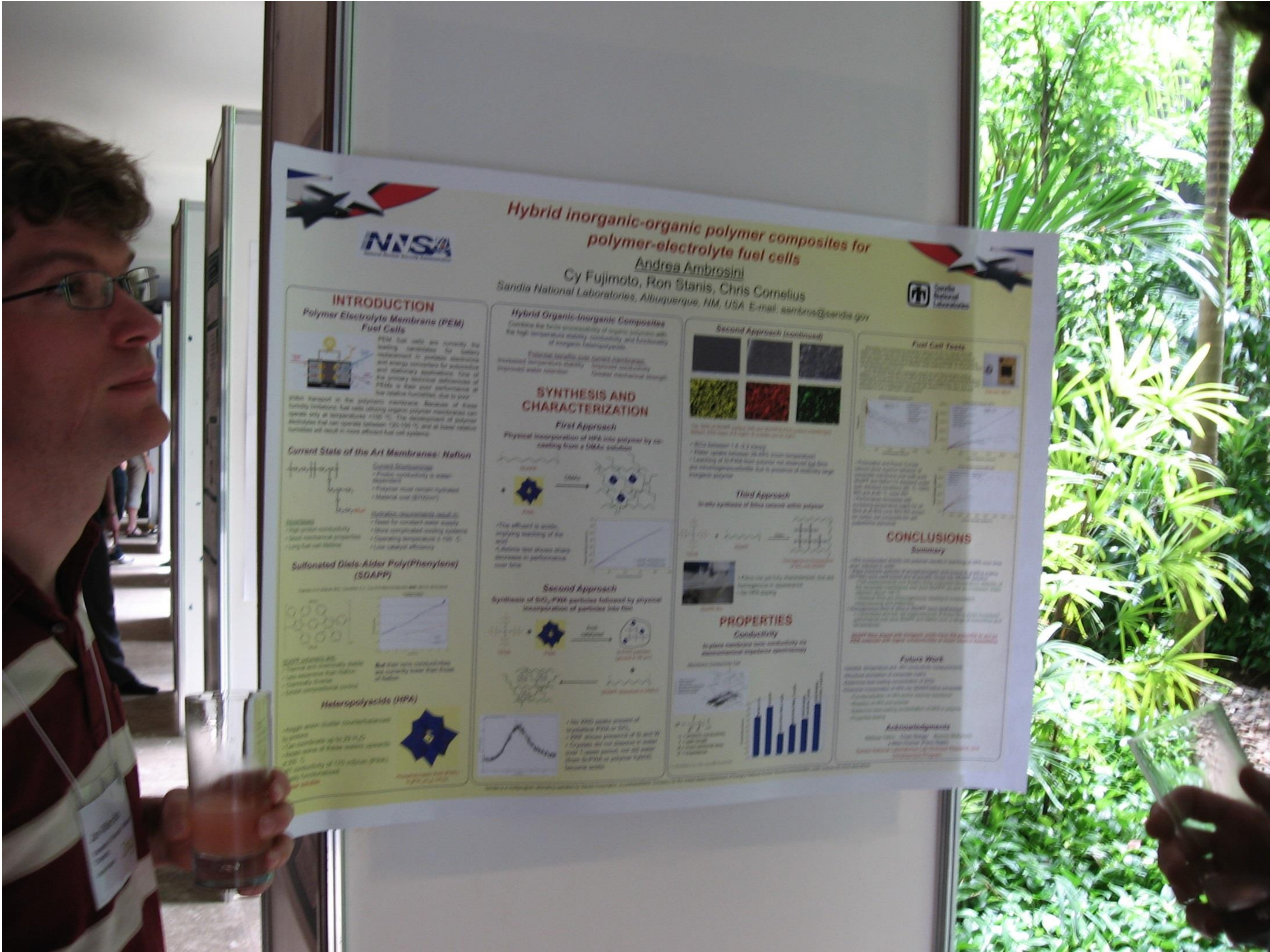

Introduction
Setup
Method
Results

Colaborators

Masaaki ISOBE

- **Dr. Masao ARAI**
  - Senior Researcher, Computational Materials Science Center, NIMS
  - ab-initio band calculation
- **Dr. Mitsuyuki SHIZUYA**
  - Post Doctoral Fellow, Advanced-Nano Materials Laboratory, NIMS
  - Synthesis and physical properties measurements
  - (Present Address: TDK Co., Ltd.)
- **Dr. Eiji TAKAYAMA-MUROMACHI**
  - Managing Director, Advanced-Nano Materials Laboratory, NIMS


16
INCD EMC
Metallic oxide coating of Ti for DSA used in chlorine production for water chlorination
Marius Constantin MIRICA[1], Mirela IORGA[1], Nicolae MIRICA[1], Nicolae VASZILCSIN[2], Ionel BALCU[1], Marinela MICLĂU[1]
Introduction
Results and discussion
1
Quality of prepared substrate surface
2
4
5
6
Conclusions
Marius Mirica
Zing



Hybrid inorganic-organic polymer composites for polymer-electrolyte fuel cells

Andrea Ambrosini
Cy Fujimoto, Ron Stanis, Chris Cornelius
Sandia National Laboratories, Albuquerque, NM, USA E-mail: aambros@sandia.gov

INTRODUCTION

Polymer Electrolyte Membrane (PEM) Fuel Cells

Current State of the Art Membranes: Nafion

Sulfonated Diels-Alder Poly(Phenylene) (SDAPP)

Heteropolyacids (HPA)

Hybrid Organic-Inorganic Composites

SYNTHESIS AND CHARACTERIZATION

First Approach

Physical incorporation of HPA into polymer by co-casting from a DMAc solution

Second Approach

Synthesis of SiO_2/PWA particles followed by physical incorporation of particles into film

Second Approach (continued)

Third Approach

PROPERTIES

Conductivity

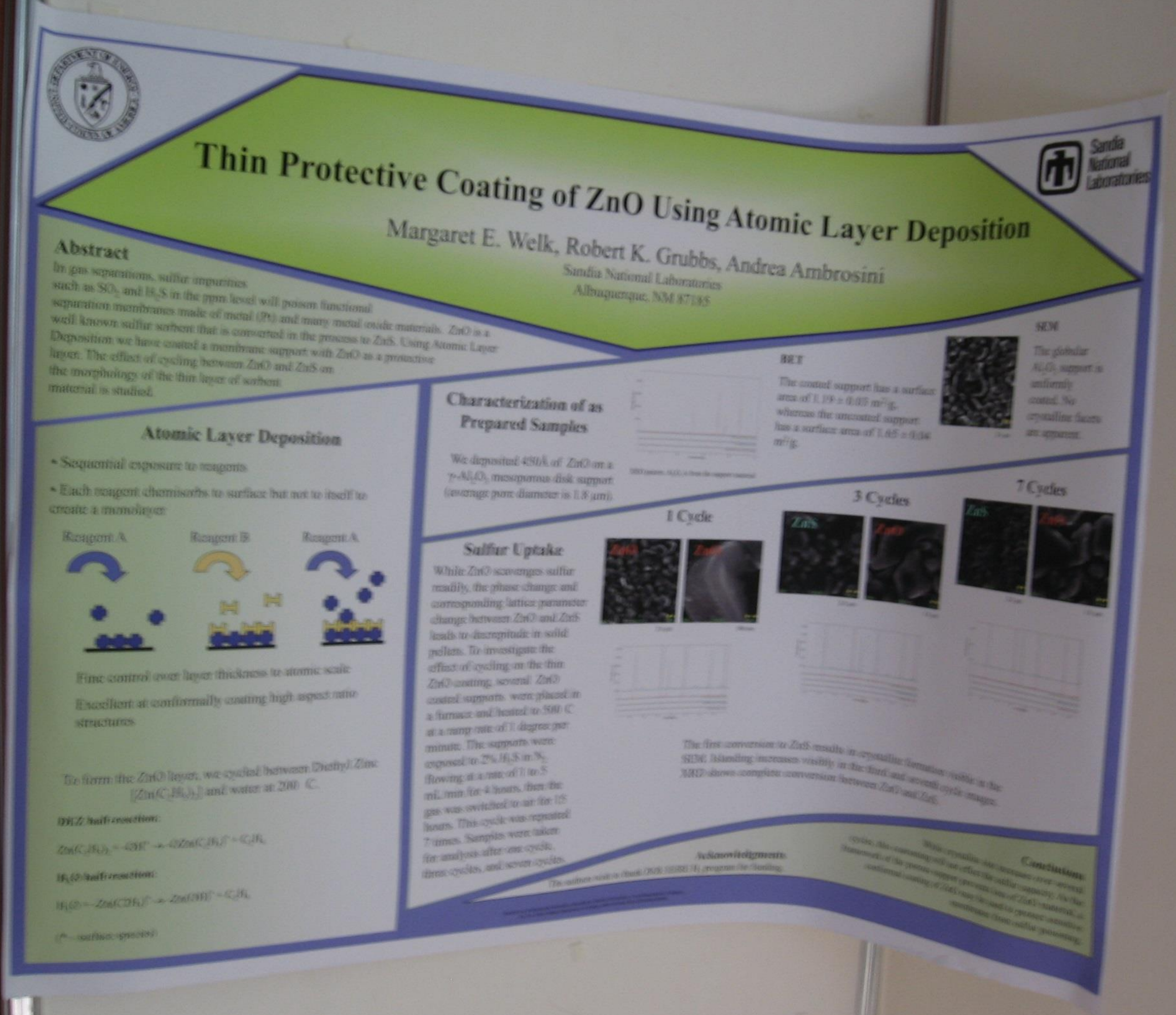
Fuel Cell Tests

CONCLUSIONS

Summary

Future Work

Acknowledgments



Thin Protective Coating of ZnO Using Atomic Layer Deposition

Margaret E. Welk, Robert K. Grubbs, Andrea Ambrosini
Sandia National Laboratories
Albuquerque, NM 87185

Abstract

In gas separations, sulfur impurities such as SO_2 and H_2S in the ppm level will poison functional separation membranes made of metal (Pt) and many metal oxide materials. ZnO is a well known sulfur sorbent that is converted in the process to ZnS. Using Atomic Layer Deposition we have coated a membrane support with ZnO as a protective layer. The effect of cycling between ZnO and ZnS on the morphology of the thin layer of sorbent material is studied.

Atomic Layer Deposition

- Sequential exposure to reagents
- Each reagent chemisorbs to surface but not to itself to create a monolayer

Fine control over layer thickness to atomic scale

Excellent at conformally coating high aspect ratio structures

To form the ZnO layer, we cycled between Diethyl Zinc $[Zn(C_2H_5)_2]$ and water at 200 C.

DEZ half reaction:

$Zn(C_2H_5)_2 + OH^* \rightarrow OZn(C_2H_5)^* + C_2H_6$

H_2O half reaction:

$H_2O + Zn(C_2H_5)^* \rightarrow ZnOH^* + C_2H_6$

(* = surface species)

Characterization of as Prepared Samples

We deposited 450Å of ZnO on a γ-Al_2O_3 mesoporous disk support (average pore diameter is 1.8 µm).

XRD pattern, Al_2O_3 is from the support material

BET

The coated support has a surface area of 1.19 ± 0.05 m^2/g, whereas the uncoated support has a surface area of 1.65 ± 0.04 m^2/g.

SEM

The globular Al_2O_3 support is uniformly coated. No crystalline facets are apparent.

Sulfur Uptake

While ZnO scavenges sulfur readily, the phase change and corresponding lattice parameter change between ZnO and ZnS leads to decrepitude in solid pellets. To investigate the effect of cycling on the thin ZnO coating, several ZnO coated supports were placed in a furnace and heated to 500 C at a ramp rate of 1 degree per minute. The supports were exposed to 2% H_2S in N_2 flowing at a rate of 1 to 5 mL/min for 4 hours, then the gas was switched to air for 15 hours. This cycle was repeated 7 times. Samples were taken for analysis after one cycle, three cycles, and seven cycles.

The first conversion to ZnS results in crystalline formation visible in the SEM. Islanding increases visibly in the third and seventh cycle images. XRD shows complete conversion between ZnO and ZnS.

Conclusion

While crystallite size increases over several cycles, this coarsening will not affect the sulfur capacity. As the framework of the porous support prevents loss of ZnO material, a conformal coating of ZnO may be used to protect sensitive membranes from sulfur poisoning.

Acknowledgments

The authors wish to thank DOE [illegible] program for funding.

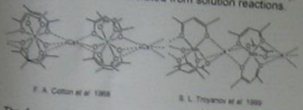
Heterometallic β-Diketonates as Potential Single-Source Precursors for Oxide-Based Materials

Evgeny V. Dikarev*, Haitao Zhang and Bo Li

Department of Chemistry, University at Albany

Introduction

Although heterometallic β-diketonates are promising precursors for multimetallic oxides, they are rare species due, in part, to chelating character of the ligand. Since 1960s, only a limited number of heterometallic β-diketonates have been isolated from solution reactions.



F. A. Cotton et al. 1968 — S. L. Troyanov et al. 1999

The focus of our investigation was to develop a simple, high-yield synthetic approach to heterometallic main group – transition metal β-diketonates.

Synthetic Strategy

We suggested several low-temperature synthetic routes that appear to be general for preparation of heterometallic diketonates incorporating a wide range of *s*-, *p*-, *d*-, and *f*-metals.

Interaction between coordinatively unsaturated homometallic β-diketonates:

$$M(\beta\text{-dik})_n + M(\beta\text{-dik})_m \rightarrow MM(\beta\text{-dik})_{n+m}$$

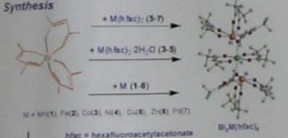
Ligand replacement:

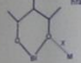
$$M(\beta\text{-dik})_n + M(\beta\text{-dik})_m L \rightarrow MM(\beta\text{-dik})_{n+m} + L$$

Redox reaction:

$$M(\beta\text{-dik})_n + M \rightarrow MM(\beta\text{-dik})_n$$

Heterometallic Bismuth-Transition Metal β-Diketonates



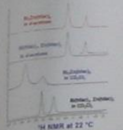


hfac = hexafluoroacetylacetonate

M	Mn	Fe	Co	Ni	Cu	Zn	Pd
X, Å	2.307	2.251	2.232	2.163	2.588	2.264	2.825

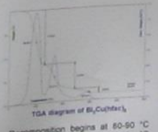
E. V. Dikarev, H. Zhang, B. Li JACS 2005, 127, 6156

Solution and Thermal Behavior of $Bi_2M(hfac)_8$



^1H NMR at 22 °C

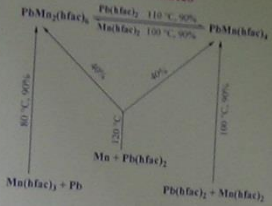
Heterometallic molecules retain their trinuclear structure in noncoordinating solvents and dissociate in coordinating solvents



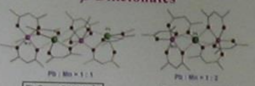
TGA diagram of $Bi_2Cu(hfac)_8$

Decomposition begins at 80-90 °C and completes at 300-350 °C showing no apparent loss of β-diketonates to sublimation.

Controlled Syntheses of Pb–Mn β-Diketonates



Pb(II)-Mn(II) Heterometallic β-Diketonates

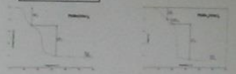


Pb : Mn = 1 : 1

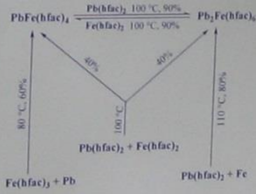
Pb–O	2.34-2.62 Å
Pb···O	2.74-2.93 Å
Mn–O	2.06-2.20 Å
Mn···O	2.21-2.81 Å

Pb : Mn = 1 : 2

Mn–O	2.10-2.24 Å
Pb···O	2.36-2.81 Å



Controlled Syntheses of Pb–Fe β-Diketonates



Pb(II)-Fe(II) Heterometallic β-Diketonates

Pb : Fe = 1 : 1

Pb–O	2.34-2.59 Å
Pb···O	2.81-2.83 Å
Fe–O	2.01-2.03 Å
Fe···O	2.37 Å

Pb : Fe = 2 : 1

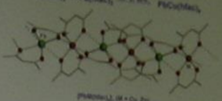
Pb–O	2.37-2.48 Å
Pb···O	2.77-2.91 Å
Fe–O	2.01-2.03 Å
Fe···O	2.23 Å

PbM(hfac)₄ (M = Cu, Zn)

Pb(hfac)₂ + M [illegible] PbM(hfac)₄ (M = Cu, Zn)

Cu(hfac)₂ + Pb [illegible] PbCu(hfac)₄

Cu(hfac)₂ + Pb(hfac)₂ [illegible] PbCu(hfac)₄



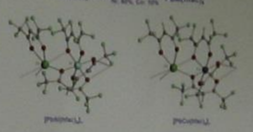
[PbM(hfac)₄]ₙ (M = Cu, Zn)

Pb–O	[illegible]
Pb···O	[illegible]
Cu–O	[illegible]
Cu···O	2.73 Å

Pb–O	[illegible]
Pb···O	[illegible]
Zn–O	[illegible]
Zn···O	2.64 Å

PbM(hfac)₄ (M = Co, Ni)

Pb(hfac)₂ + M [illegible] PbM(hfac)₄



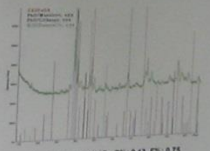
[PbNi(hfac)₄]ₙ

Pb–O	2.34 Å
Pb···O	2.74-2.83 Å
Ni–O	2.02-2.04 Å

[PbCo(hfac)₄]ₙ

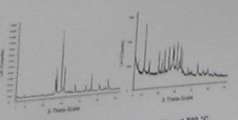
Pb–O	2.32-2.35 Å
Pb···O	2.80-2.84 Å
Co–O	2.03-2.06 Å

Decomposition of $PbNi(hfac)_4$: New Pb-Ni-O Phase



Annealing at 500 °C; C%: 0.43, F%: 0.75

Decomposition of $PbNi(hfac)_4$ at Different Temperatures



Annealing at 400 °C

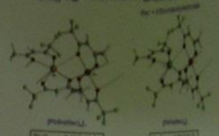
C%: 0.47, F%: 4.35

Annealing at 700 °C

C%: 0.33, F%: < 0.20

MFe(tfac)₄ (M = Pb, Sn)

Fe(tfac)₃ + M [illegible] MFe(tfac)₄ (M = Sn, Pb)

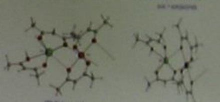


[illegible]	[illegible]
[illegible]	[illegible]
Fe–O	[illegible]
Fe···O	[illegible]

[illegible]	[illegible]
[illegible]	[illegible]
[illegible]	[illegible]
[illegible]	[illegible]

Non-Fluorinated Heterometallic β-Diketonates

M(acac)₂ + Pb [illegible] PbM(acac)₄ (M = Fe, Co)



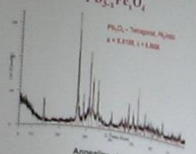
[PbFe(acac)₄]ₙ

Pb–O	2.30-2.50 Å
Pb···O	2.88, 2.93 Å
Fe–O	2.03-2.06 Å
Fe···O	2.24, 2.27 Å

[PbCo(acac)₄]ₙ

Pb–O	[illegible]
Pb···O	[illegible]
Co–O	[illegible]
Co···O	[illegible]

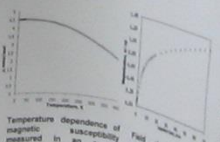
Decomposition of $PbFe(acac)_4$: $Pb_{3-x}Fe_xO_4$



Annealing at 500 °C:

"$Pb_{2.2}Fe_{0.8}O_4$" – Pb%: 68.29, Fe%: 18.28, O%: 13.43

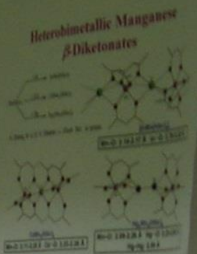
Magnetic Study of $Pb_{3-x}Fe_xO_4$



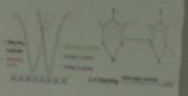
Temperature dependence of magnetic susceptibility measured in an applied magnetic field of 100 Oe.

Field dependence of magnetization measured at 1.8 K.

Heterobimetallic Manganese β-Diketonates



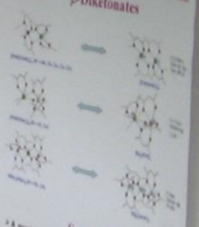
IR Study of Heterometallic β-Diketonates



Infrared C-H Stretching Frequencies (cm^{-1})

Compound	Solid state	[illegible] solution	[illegible] solution
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]		[illegible]
[illegible]	[illegible]		[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]

Structural Analogy with Homometallic β-Diketonates

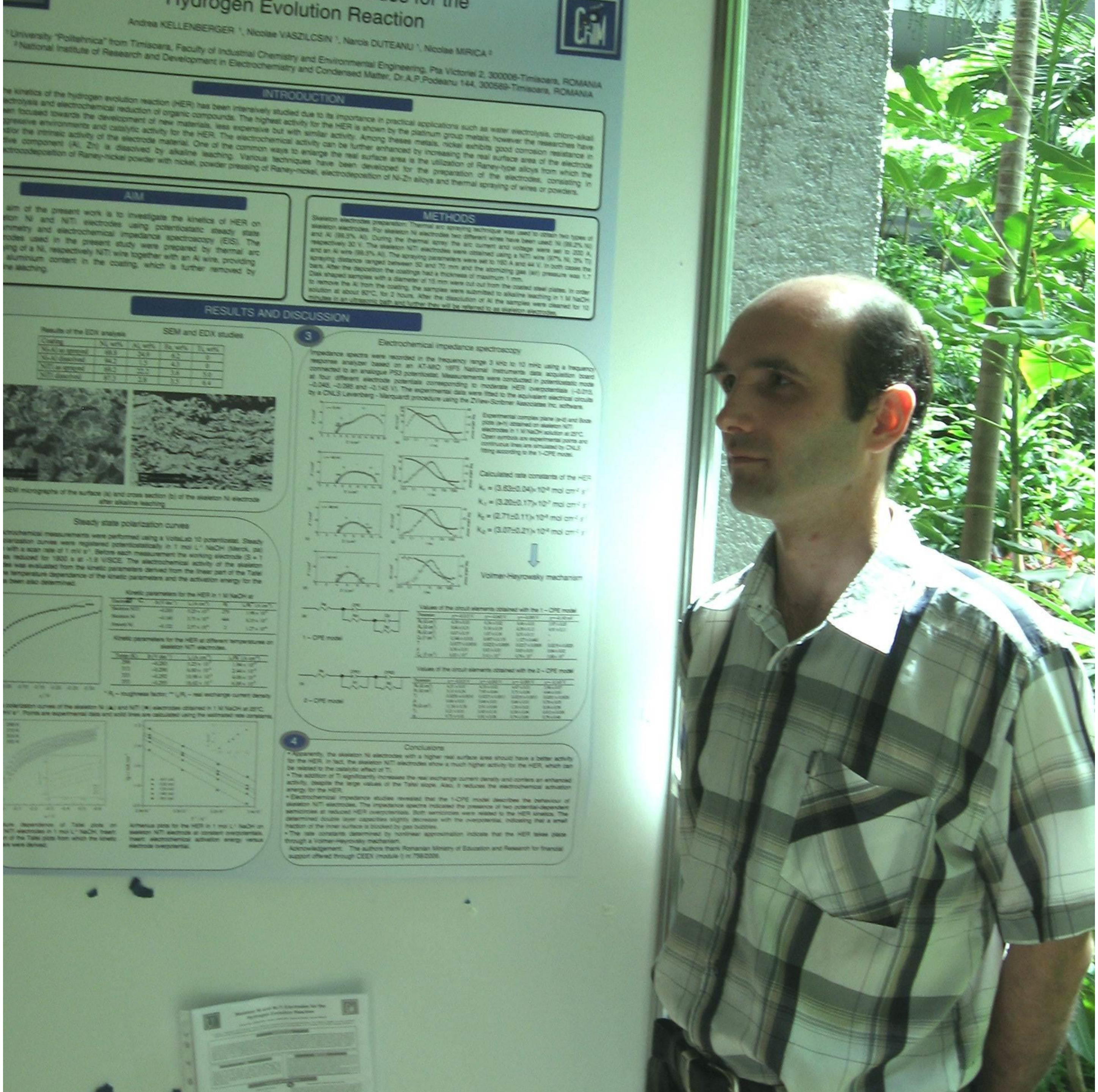


Summary

- A number of heterometallic β-diketonates incorporating main group and transition metals have been isolated by novel low-temperature solid-state synthetic routes.
- Heterometallic β-diketonates are built on Lewis acid-base interactions and bear the structural analogy to their homometallic polynuclear counterparts.
- Heterometallic β-diketonates retain their structures in non-coordinating solvents and dissociate into homometallic species in coordinating solvents.
- Heterometallic β-diketonates with different metal ratio can be obtained using different starting materials.
- Thermal decomposition of heterometallic β-diketonates results in new bimetallic oxides that cannot be obtained from high-temperature synthesis.

Acknowledgement

University at Albany Research Foundation,
ACS-PRF, NSF-CHE



Hydrogen Evolution Reaction

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INTRODUCTION

he kinetics of the hydrogen evolution reaction (HER) has been intensively studied due to its importance in practical applications such as water electrolysis, chloro-alkali ectrolysis and electrochemical reduction of organic compounds. The highest activity for the HER is shown by the platinum group metals; however the researches have een focused towards the development of new materials, less expensive but with similar activity. Among theses metals, nickel exhibits good corrosion resistance in ggressive environments and catalytic activity for the HER. The electrochemical activity can be further enhanced by increasing the real surface area of the electrode nd/or the intrinsic activity of the electrode material. One of the common ways to enlarge the real surface area is the utilization of Raney-type alloys from which the ctive component (Al, Zn) is dissolved by alkaline leaching. Various techniques have been developed for the preparation of the electrodes, consisting in lectrocodeposition of Raney-nickel powder with nickel, powder pressing of Raney-nickel, electrodeposition of Ni-Zn alloys and thermal spraying of wires or powders.

AIM

aim of the present work is to investigate the kinetics of HER on eton Ni and NiTi electrodes using potentiostatic steady state mmetry and electrochemical impedance spectroscopy (EIS). The rodes used in the present study were prepared by thermal arc ing of a Ni, respectively NiTi wire together with an Al wire, providing aluminium content in the coating, which is further removed by he leaching.

METHODS

RESULTS AND DISCUSSION

SEM and EDX studies

Results of the EDX analysis

SEM micrographs of the surface (a) and cross section (b) of the skeleton Ni electrode after alkaline leaching

Steady state polarization curves

3

Electrochemical impedance spectroscopy

Calculated rate constants of the HER

Volmer-Heyrovsky mechanism

4

Conclusions


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