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14. ABSTRACT This is the final report of the ARO project of gathering and sharing information on the role of stress and elastic strain fields on catalysis: "Stress Controlled Catalysis via Engineered Nanostructures." For this effort a workshop was organized and held at Brown University Providence, Rhode Island during June, 1 - 2, 2015. It was planned and organized with help from the co-investigators of the ARO MURI program at Brown University and California State University -Northridge: Shouheng Sun, Andrew Peterson, Sharvan Kumar, Gang Lu and William Curtin. In addition, the workshop leveraged the opportunities of reviewing past and ongoing research on stress effects in					
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## Report Title

Final Report: A Proposal for Workshop Support for current award "Stress Controlled Catalysis via Engineered Nanostructures"

### ABSTRACT

This is the final report of the ARO project of gathering and sharing information on the role of stress and elastic strain fields on catalysis: "Stress Controlled Catalysis via Engineered Nanostructures." For this effort a workshop was organized and held at Brown University Providence, Rhode Island during June, 1 - 2, 2015. It was planned and organized with help from the co-investigators of the ARO MURI program at Brown University and California State University –Northridge: Shouheng Sun, Andrew Peterson, Sharvan Kumar, Gang Lu and William Curtin. In addition, the workshop leveraged the opportunities of reviewing past and ongoing research on stress effects in catalysis in the U.S. and European institutions. Approximately fifty-nine (59) researchers participated in the workshop, including graduate students, postdoctoral researchers and faculty, from multiple disciplines including solid mechanics, materials science, physics, chemistry, chemical engineering and electrochemistry. As an outcome, a number of new ideas on possible future directions on the role of elastic strain on catalysis and ways to exploit the coupling were shared across traditional disciplinary boundaries. The workshop was held successfully and has reached a conclusion that there is potential for exploiting the stress effect to design improved catalysts, possibly with less previous materials. It is also noted that hybrid approaches of experiments coupled with or guided by large scale computation is highly effective in discovering rich phenomena at multiple length and time scales. Some suggestions on future directions for research in this area are described in this report.

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**Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:**

**(a) Papers published in peer-reviewed journals (N/A for none)**

<u>Received</u>	<u>Paper</u>
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**TOTAL:**

**Number of Papers published in peer-reviewed journals:**

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**(b) Papers published in non-peer-reviewed journals (N/A for none)**

<u>Received</u>	<u>Paper</u>
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**TOTAL:**

**Number of Papers published in non peer-reviewed journals:**

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**(c) Presentations**

Number of Presentations: 0.00

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**Non Peer-Reviewed Conference Proceeding publications (other than abstracts):**

Received      Paper

**TOTAL:**

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

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**Peer-Reviewed Conference Proceeding publications (other than abstracts):**

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**TOTAL:**

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

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**(d) Manuscripts**

Received      Paper

**TOTAL:**

Number of Manuscripts:

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**Books**

Received      Book

**TOTAL:**

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

### Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: .....

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:.....

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:.....

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The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense .....

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### Names of Personnel receiving masters degrees

NAME

**Total Number:**

### Names of personnel receiving PHDs

NAME

**Total Number:**

### Names of other research staff

NAME

PERCENT SUPPORTED

**FTE Equivalent:**

**Total Number:**

### Sub Contractors (DD882)

### Inventions (DD882)

### Scientific Progress

### Technology Transfer

## **Final Report**

### **ARO Workshop on**

### **“Stress Controlled Catalysis via Engineered Nanostructures”**

held at Brown University, Providence, Rhode Island from June 1 to June 2 of 2015

PI: Pradeep R. Guduru, Brown University. Pradeep\_Guduru@Brown.edu

### **Supported by**

Army Research Office

Program Manager

Dr. David Stepp

ARO Award # W911NF-15-1-0260

## **Summary**

This is the final report of the ARO project of gathering and sharing information on the role of stress and elastic strain fields on catalysis: “Stress Controlled Catalysis via Engineered Nanostructures.” For this effort a workshop was organized and held at Brown University Providence, Rhode Island during June, 1 - 2, 2015. It was planned and organized with help from the co-investigators of the ARO MURI program at Brown University and California State University –Northridge: Shouheng Sun, Andrew Peterson, Sharvan Kumar, Gang Lu and William Curtin. In addition, the workshop leveraged the opportunities of reviewing past and ongoing research on stress effects in catalysis in the U.S. and European institutions. Approximately fifty-nine (59) researchers participated in the workshop, including graduate students, postdoctoral researchers and faculty, from multiple disciplines including solid mechanics, materials science, physics, chemistry, chemical engineering and electrochemistry. As an outcome, a number of new ideas on possible future directions on the role of elastic strain on catalysis and ways to exploit the coupling were shared across traditional disciplinary boundaries. The workshop was held successfully and has reached a conclusion that there is potential for exploiting the stress effect to design improved catalysts, possibly with less previous materials. It is also noted that hybrid approaches of experiments coupled with or guided by large scale computation is highly effective in discovering rich phenomena at multiple length and time scales. Some suggestions on future directions for research in this area are described in this report.

## Introduction

The full sequence of elementary steps in a general heterogeneous catalytic reaction involves adsorption of reacting species, dissociation and association of chemical species, transport on the surface, and desorption of the product species. The role of elastic strain in tuning catalytic reaction rates has evolved rapidly in the last decade and has initiated a significant re-evaluation of catalyst design. Specifically, researchers have examined the role of misfit strain arising when a catalytically-active metal overlayer is epitaxially deposited on another metal substrate or when a shell metal is deposited around a core metal to form a core/shell nanoparticle. In general, the misfit strain changes the width of the d-band through changes to the d-orbital interactions that are quite sensitive to interatomic spacing  $r$ , scaling as  $r^{-5}$ . Changes in the d-band width modify reactivity of the strained surface by shifting the mean d-band energy (the “d-band center”) relative to the Fermi energy, which influences the bonding and anti-bonding states of adsorbates and reactants on the metal surface. The relationship between the adsorption energy and the d-band center was confirmed, for example, by calculations for CO adsorption on various surfaces, which in combination with the adsorbate scaling relations and the Brønsted-Evans-Polyani (BEP) relationship relates the d-band center to reaction rates and the Volcano Plot. Early experimental evidence of strain effects on phenomena related to catalysis was reported by Gsell *et al.* [Gsell *et al. Science* 1998, 280, 717-720.] who demonstrated that O adsorption on Ru could be enhanced under tensile strain. The lattice strain effect on HER activity was also studied by Wolfschmidt using a Pt-modified Au(111) catalyst; it was shown that monatomically high Pt islands on Au(111) have enhanced catalytic activity [Holger *et al. J. Phys.: Condens. Matter* 2008, 20, 374127]. Other experimental work has involved core-shell nanostructures, which introduce mismatch strains in the outer shell, and also de-alloying to control misfit or surface strains. Strasser *et al. [Nature Chem.* 2010, 2, 454-460] studied PtCu@Cu core@shell system formed by de-alloying Pt-Cu nanoparticles and attributed increases in catalytic activity for oxygen reduction reaction (ORR) to the elastic strain in the de-alloyed shell. In their study, the strain in the shell was estimated indirectly through anomalous X-ray diffraction of nano-particles before and after de-alloying. The saturation of activity with the estimated elastic strain and the absence of the expected Volcano plot were attributed to possible relaxation mechanisms in the shell. However, there are a few studies that separate the strain-effect from the ligand effect. Recently, Smetanin *et al. [Phys. Chem. Chem. Phys.* 2011, 13, 17313-17322] reported changes in the potential and current for predominantly capacitive processes in 20-nm-thick Au films supported on polyimide substrates subjected to cyclic loading under galvanostatic and potentiostatic conditions, which were explained in terms of the influence of strain on the surface capacitive processes. Deng *et al. [J. Catal.* 2014, 309, 351-361] studied the influence of elastic strain in thin films of Au and Pt on polyimide on their electrocatalytic activity for HER. More recently, Yang *et al. [Phys. Chem. Chem. Phys.* 2015, 17, 1746-1754] demonstrated a similar effect for ORR on a Pd-based metallic glass catalyst film under both tensile and compressive strains, which have opposite effect on catalytic activity. Sethuraman *et al. [J. Phys. Chem. C* 2015, 119, 19042-19052] subjected thin Pt films on single crystal Si substrates to external straining while the films were participating in ORR through cyclic voltammetry (CV). They showed that a tensile strain of 0.7% resulted in ~15 mV reduction in the overpotential.

As illustrated above, there is a growing body of investigations and interest in understanding the stress-catalysis coupling and in exploiting such an effect for improved catalyst design. This area offers a variety of rich research problems at the frontiers of solid mechanics and catalytic chemistry, which are yet to be explored completely. Potential benefits from design of new catalysts, particularly for important reactions in the realm of energy conversion and storage, are enormous. The objective of this workshop is to assemble the active researchers in this area, have them present their latest work and lay a roadmap of promising future directions.

The workshop was organized on Brown University campus in Providence, Rhode Island during June 1-2, 2015. The invitees came not only from various US Universities, but also from Europe. The session titles and the topics of their presentations are listed as follows:

Session I: Experimental Investigations of Stress Effects in Catalysis

On the Coupling between Chemistry and Mechanics at Surfaces, Jörg Weissmüller, University of Hamburg

Surface and Growth Stress at Solid Electrodes, Gery Stafford, National Institute of Standards and Technology

The influence of stress on electrochemical reactions on metal surfaces, Pradeep R. Guduru, Brown University

Elastic Strain Effects on Electrochemical Catalysis of Pd-based Metallic Glass Films, Sharvan Kumar, Brown University

Nanostructured catalysts and interfaces for electrochemical energy storage and conversion, Peter Strasser, University of Berlin

Insight into catalyst and battery degradation from operando X-ray scattering, Mike Toney, Stanford Synchrotron Radiation Lightsource

Session II: Theoretical and Computational Investigations of Stress Effects in Catalysis

Theory of stress effects on catalysis at close-packed surfaces, Andrew Peterson, Brown University

Theory of stress effects on catalysis at surface steps, William Curtin, École polytechnique fédérale de Lausanne

Multiscale Quantum Mechanics/Molecular Mechanics Method, Gang Lu, California State University Northridge

Oxygen reduction reaction at the atomic scale, Jan Rossmeisl, Technical University of Denmark

Modeling Material-Environment Chemistry and the Effects of stress, electrochemistry and the nanoscale, Christopher Taylor, Det Norske Veritas

Ab-initio and multiscale studies of catalytic processes on metal and semiconductor surfaces, Eftimos Kaxiras, Harvard University

Session III: Stress effects in catalysis – Applications

Catalysis on core/shell nanoparticles: fabrication and optimization, Shouheng Sun, Brown University

Maximizing the Mass Activity of Pt-Based Catalysts toward Oxygen Reduction, Younan Xia, Georgia Tech

Strained platinum and its alloy nanoparticle electrocatalysts, Hong Yang, University of Illinois, Urbana Champaign

Theory and design of core/shell nanoparticles, Gang Lu, California State University Northridge

Design tool for rapid core/shell nanoparticle optimization, William Curtin, Brown University/École polytechnique fédérale de Lausanne

In addition to the invited presentations listed above, there was also a poster session on June 1, 2015. The outcome of the workshop is described in the following sections. The workshop program and participants are presented in the Appendices A and B respectively.



## Findings

This workshop brought together a multidisciplinary team of scientists spanning chemistry, physics, materials science and applied mechanics, to present and review the scientific foundation, current state of the art and future directions for controlling catalytic reactions using applied stress. Through a combination of controlled experiments and multiscale modeling, it is clearly demonstrated that stress can translate catalysts along the “volcano” plot for a given reaction. For example, for the oxygen reduction reaction (ORR), it is shown that experimentally accessible elastic strain of  $\sim 0.5\%$  can change the overpotential of Pt by approximately 10%; compressive strain is shown to decrease the overpotential (increased catalytic activity) and tensile strain has the opposite effect. Experimental results were generally in good agreement with computational predictions, which gives confidence in the predictions of the latter for more complicated strain states that have not yet been accessed experimentally. Thus, it is shown that a judicious combination of experiments and multiscale modeling is an effective approach in advancing the new ideas and problems discussed at the workshop. Some specific results and advances presented at the workshop include: (i) Clear demonstration of the strain effect on the hydrogen evolution reaction (HER) in three catalysts that span the volcano plot (Ni, Pt, Cu). Experiments and computations show good agreement with each other. (ii) Clear demonstration of the stress effect on ORR (oxygen reduction reaction) catalysis on Pt, Pd and Pd based bulk metallic glass through *in situ* uniaxial compression/tension experiments on thin film samples while they are participating in the electrocatalytic reaction. (iii) DFT calculations of catalytic activity of strained Pt and Pd surfaces towards ORR, which predicted that 2-3% biaxial compressive strain moves them towards the peak of the “volcano plot,” resulting in enhanced catalytic activity. These calculations are in qualitative agreement with the experiments described above. (iv) A recent novel advance in computational capability is the development of quantum mechanics/molecular mechanics (QM/MM) multiscale framework that allows investigation of catalytic activity of active sites on large nano-particles, which was not possible before. The QM/MM multiscale method has been applied successfully to the design of core-shell catalyst nanoparticles. Previous models of core-shell particles have been limited to particle sizes of less than 1 nm due to computational cost. The QM/MM method now enables a systematic study of surface strain effects in larger particles and provides a theoretical guidance on materials selection and optimal dimensions. The investigations of Pt-Cu core shell particles for ORR reactions not only explain the performance of catalyst particles reported in literature, but also predict optimal particle size and shell thickness. (v) It has been demonstrated that due to Pt surface compression, intermetallic FePt is a robust catalyst system for the oxygen reduction reaction and hydrogen evolution. The right controls on surface compression and its effect on catalytic enhancement was further supported by the QM-MM calculations. (vi) Through a combination of prediction made by the QM/MM method and novel synthesis processes, it has been demonstrated that intermetallic particle induced surface compression in FePd system leads to excellent catalyst for oxygen reduction reaction with catalytic performance comparable to Pt. (vii) Guided by QM/MM method, a new core/shell NiAu/Au nanoparticles has been developed as new non-Pt catalyst for hydrogen evolution reaction in acid with Pt-like activity. (viii) It has been demonstrated that for catalytic reactions occurring at step edges, mechanical effects (i.e. change in strain energy due to interaction between the strain induced by adsorption and the prevailing stress field) can dominate the electronic structure effect. This is an exciting new finding and it predicts that stress can potentially have a large effect on methanation on Ni. This is an open problem for experimental investigation.

## **Recommendations for further/future research**

The above advances discussed at the workshop present a few exciting avenues for future:

- (i) Investigation of the class of reactions in which step edges serve as active sites in such a way that external strain can have a much larger effect on catalytic activity than what has been demonstrated so far. Computations predict that methanation on Ni is one such reaction. Investigating and classifying all such reactions that are of importance in energy technologies (and other technologically important reactions) is a fruitful avenue of research.
- (ii) Developing experimental techniques and tools to complement the above predictions must be pursued.
- (iii) Most of the experimental investigations of externally applied stress effect on catalysis have been done in uniaxial loading configuration. Computations predict a significantly larger effect for biaxially strained catalyst films. This remains to be a fruitful investigation to pursue.
- (iv) More extensive use of the QM/MM multiscale modeling tool to predict new classes of core-shell nanoparticle catalysts for several technologically important reactions and a complementary experimental investigation to validate (or otherwise) the computational findings.

## **Acknowledgments**

We gratefully appreciate the ARO funding for the workshop, ARO grant # W911NF-15-1-0260.

## Appendix A: Workshop Program

### ARO Workshop on Stress Controlled Catalysis via Engineered Nanostructures Brown University, Providence, RI 02912, June 1 – 2, 2015

#### Agenda

##### June 1, 2015

- 7:30 – 8:15 am      Arrival and Breakfast
- 8:15 – 8:30 am      Overview of the ARO MURI Program on “Stress Controlled Catalysis via Engineered Structures.”  
Pradeep R. Guduru, Brown University

##### Session I: Experimental Investigations of Stress Effects in Catalysis

Session Chair: William Curtin, École polytechnique fédérale de Lausanne

- 8:30 – 9:05 am      *On the Coupling between Chemistry and Mechanics at Surfaces*  
Jörg Weissmüller, University of Hamburg
- 9:05 – 9:40 am      *Surface and Growth Stress at Solid Electrodes*  
Gery Stafford, National Institute of Standards and Technology
- 9:40 – 10:15 am      *The influence of stress on electrochemical reactions on metal surfaces*  
Pradeep R. Guduru, Brown University
- 10:15 – 10:35 am      Break (Poster session)
- 10:35 – 11:10 am      *Elastic Strain Effects on Electrochemical Catalysis of Pd-based Metallic Glass Films*  
Sharvan Kumar, Brown University
- 11:10 – 11:45 am      *Nanostructured catalysts and interfaces for electrochemical energy storage and conversion*  
Peter Strasser, University of Berlin
- 11:45 – 12:20 pm      *Insight into catalyst and battery degradation from operando X-ray scattering*  
Mike Toney, Stanford Synchrotron Radiation Lightsource
- 12:20 – 1:30 pm      Lunch (Poster session)

##### Session II: Theoretical and Computational Investigations of Stress Effects in Catalysis

Session Chair: Andrew Peterson, Brown University

- 1:30 – 2:05 pm      Theory of stress effects on catalysis at close-packed surfaces  
Andrew Peterson, Brown University

2:05 – 2:40 pm	Theory of stress effects on catalysis at surface steps William Curtin, École polytechnique fédérale de Lausanne
2:40 – 3:15 pm	Multiscale Quantum Mechanics/Molecular Mechanics Method Gang Lu, California State University Northridge
3:15 – 3:35 pm	Break (Poster session)
3:35 – 4:10 pm	Oxygen reduction reaction at the atomic scale Jan Rossmeisl, Technical University of Denmark
4:10 – 4:45 pm	Modeling Material-Environment Chemistry and the Effects of stress, electrochemistry and the nanoscale Christopher Taylor, Det Norske Veritas
4:45 – 5:20 pm	Ab-initio and multiscale studies of catalytic processes on metal and semiconductor surfaces Eftimos Kaxiras, Harvard University

## **June 2, 2015**

7:30 – 8:30am	Breakfast
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### **Session III: Stress effects in catalysis – Applications**

Session Chair: Pradeep R. Guduru, Brown University

8:30 – 9:05 am	Catalysis on core/shell nanoparticles: fabrication and optimization Shouheng Sun, Brown University
9:05 – 9:40 am	Maximizing the Mass Activity of Pt-Based Catalysts toward Oxygen Reduction Younan Xia, Georgia Tech
9:40 – 10:15 am	Strained platinum and its alloy nanoparticle electrocatalysts Hong Yang, University of Illinois, Urbana Champaign
10:15 – 10:30am	Break
10:30 – 11:05 am	Theory and design of core/shell nanoparticles Gang Lu, California State University Northridge
11:05 – 11:30 am	Design tool for rapid core/shell nanoparticle optimization William Curtin, École polytechnique fédérale de Lausanne
11:30 – 12:15 pm	Discussion
12:15pm – 1:30pm	Lunch

## Appendix B: List of Participants

First Name	Last Name	University/Organization	First Name	Last Name	University/Organization
Ismael	Abu-Baker	Brown University	Tayhas	Palmore	Brown University
Steven	Ahn	Brown University	Andrew	Peterson	Brown University
Jaydeep	Bardhan	Northeastern University	Dawanne	Poree	U.S. Army Research Office
Eric	Chason	Brown University	Shaghayegh	Rezazadeh	Brown University
Zhengzheng	Chen	California State University Northridge	Christoph	Rose-Petruck	Brown University, Chemistry
Bill	Curtin	Brown University/EPFL	Jan	Rossmesl	University of Copenhagen
Nicholas	Dee	Massachusetts Institute of Technology	Francisco	Schunk	Brown University
Ronald	Dunn	Brown University	Bo	Shen	Brown University
Daniel	Erdosy	Brown University	Odysseas	Skartsis	Brown University
David	Fenning	University of California -San Diego	Gery	Stafford	NIST
Michael	Francis	Brown University/EPFL	Dave	Stepp	U.S. Army Research Office
Forrest	Gittleson	Yale University	Peter	Strasser	Technical University of Berlin
Franklin	Goldsmith	Brown University	Shouheng	Sun	Brown University
Pradeep	Guduru	Brown University	Hyokyung	Sung	Brown University
Qing	Hu	Massachusetts Institute of Technology	Zheng Jie	Tan	Massachusetts Institute of Technology
Daniel	Janda	Brown University	Andre D.	Taylor	Yale University
Rongzhong	Jiang	U.S. Army Research Laboratory	Christopher	Taylor	Det Norske Veritas
Naba	Karan	Brown University	Michael	Toney	Stanford Synchrotron Radiation Lightsource
Efthimios	Kaxiras	Harvard University	Li-Qiong	Wang	Brown University
Alireza	Khorshidi	Brown University	Jörg	Weissmuller	TUHH
Seokki	Kim	Brown University	Liheng	Wu	Brown University
Taehee	Kim	Brown University	Younan	Xia	Georgia Institute of Technology
Sharvan	Kumar	Brown University	Zheng	Xia	Brown University
Qing	Li	Brown University	Kai	Yan	Brown University
Jinyang	Li	Yale University	Yiyi	Yang	Brown University
Junrui	Li	Brown University	Hong	Yang	University of Illinois at Urbana-Champaign
Gang	Lu	California State University Northridge	Insun	Yoon	Brown University
Pinkesh	Malhotra	Brown University	Xu	Zhang	California State University Northridge
Robert	Mantz	U.S. Army Research Office	Yin-Jia	Zhang	Brown University
Adriana	Mendoza-Garcia	Brown University			