Final Report for Period: 02/2009 - 07/2009
Submitted on: 07/30/2009
Principal Investigator: Grover, Martha A.
Award ID: 0348397
Organization: GA Tech Res Corp - GIT
Submitted By:
Grover, Martha - Principal Investigator
Title:
CAREER: A Systems Approach to Materials Processing

Project Participants

Senior Personnel

Name: Grover, Martha
Worked for more than 160 Hours: Yes
Contribution to Project:
Project director.

Post-doc

Graduate Student

Name: Xiong, Rentian
Worked for more than 160 Hours: Yes
Contribution to Project:
Rentian Xiong was financially supported by this grant. He has worked on the design and construction of the chemical vapor deposition reactor, and has integrated the sensors and actuators into a single LabView program. He implemented moving horizon estimation on the experimental system, using an optical reflection sensor. He graduated with his Ph.D. in the spring of 2008, and is now working as a research engineer with Dow Solar.

Name: Wissmann, Paul
Worked for more than 160 Hours: Yes
Contribution to Project:
Paul Wissmann's stipend and tuition were supported in part by this grant and in part by Georgia Tech through Prof. Grover's startup support. Paul worked on the design and construction of the chemical vapor deposition reactor. Paul's research topic was the application of experimental design and the development of minimal process models from data. He designed optimal process recipes, and then implemented them in the experimental system. Paul Wissmann graduated with his Ph.D. in the fall of 2008.

Undergraduate Student

Name: Shah, Sunny
Worked for more than 160 Hours: Yes
Contribution to Project:
This student worked during the spring of 2008. He supported the experimental design work of graduate student Paul Wissmann. He was supported by an REU supplement.

Name: Jamrog, Derrick
Worked for more than 160 Hours: Yes
Contribution to Project:
Derrick worked with Paul Wissmann during 2007, and helped to implement the process recipes design by Paul on the experimental system. This work was supported by an REU supplement.

Name: Dromgoole, Ahmad
Worked for more than 160 Hours: Yes
Contribution to Project:

Ahmad worked with our project during the first year of the project. He developed an optical model of our thin films, and helped to support the initial construction of the chemical vapor deposition system. He was supported by an REU supplement.

Name: Kiyono, Mayumi

Worked for more than 160 Hours: Yes

Contribution to Project:

Mayumi Kiyono worked on an independent research project. She used an alternate deposition process (sputtering of aluminum), and investigated the effect of process recipe on the resulting surface properties and the film thickness. She was supported by an REU supplement.

Name: Jolissaint, Matt

Worked for more than 160 Hours: No

Contribution to Project:

Matt Jolissaint worked with Rentian Xiong during the summer of 2007. He helped to implement the moving horizon estimation on the experimental system.

Technician, Programmer

Other Participant

Research Experience for Undergraduates

Organizational Partners

University of Tokyo

Rentian Xiong spent three months at the University of Tokyo with Professor Yamaguchi and Professor Noda in the Chemical System Technology Department. This exchange was supported by a supplement from the NSF IREE program.

Other Collaborators or Contacts

With the IREE supplement grant the authors collaborated with the research group headed by Professor Yukio Yamaguchi at the University of Tokyo, Japan from December 13, 2006 to March 7, 2007. The project is titled 'Investigation of catalyst by a combinatorial method and synthesis of single-walled carbon nanotube.' A combinatorial method was used to obtain different nominal thickness of Cobalt/Aluminum binary catalyst on one substrate and to study their effect on carbon nanotube (CNT) growth. An in-situ video camera was used to monitor CNT growth. It was observed that CNT growth rate on Co/Al binary catalyst is much faster than on Co catalyst alone. The growth rate is also dependent on the location on the Co/Al catalyst library. Various amount of Al was sputtered to study the effect of Al. When twice of regular amount of Al was sputtered, a novel columnar structure of CNT was obtained. This is the first time to observe such phenomena of self-organization of CNT. Further increase of Al thickness, however, did not yield any CNT. This project provided additional context and examples for the systematic approach to material processing proposed in the original project. The design of the CVD system in the host group also helped to solve a nonuniformity problem in the authors' own CVD system. Through this project new material characterization techniques such as Raman spectroscopy and TEM were also learned. In addition to research activities, the travel to Japan also increased Mr. Xiong's familiarity with Japanese language, culture, and technological trends and business practice in nanotechnology. He attended a Japanese Language class for Foreign Students and Researchers offered by the University of Tokyo, visited several museums in Tokyo, and visited other ancient and modern cities such as Kamakura, Yokohama and Nikko. Mr. Xiong also attended Nano Tech 2007 International Nanotechnology Exhibition & Conference.

Activities and Findings

Research and Education Activities:

Two graduate students have been supported by this grant. The research of Rentian Xiong focused on in-situ sensing and estimation using reflectometry. He graduated with his PhD in May 2008. The research of Paul Wissmann is on microstructure design via experimental design. He graduated with his PhD in December 2008. Our chemical vapor deposition testbed supported the methodology development in both

projects. Characterization of our films is done via atomic force microscopy, ellipsometry, and scanning probe microscopy. During the grant five undergraduate student also participated in and supported the experimental work of Paul Wissmann. Over the summer of 2008, we also had a local high school teacher working with the project, adapting our data for the statistics and data analysis part of her high school math class.

Findings:

In the area of in-situ sensing, we applied moving horizon estimation to our reflectometry data and shown that MHE provides robust estimation of multiple surface and film properties, such as thickness, refractive index, and surface roughness. Compared to the current state of the art in sensor interpretation, which is based on least squares fitting, MHE can provide significantly improved estimation, due to its uncertainty quantification and its ability to adapt. MHE is more robust to unmodeled effects such as roughness, compared to other alternatives in practice, but when roughness is directly modeled and estimated, the film properties from MHE can be even more accurate. These results are documented in three journal publications.

In the area of experimental design, we proposed a new methodology for experimental design, when process design is the ultimate objective of the experiments. This approach combines features of the factorial design of experiments with model-based experimental design methods, which currently have different objective functions. In our new methodology, both empirical and mechanistic models are used simultaneously in the experimental design. Based on the current data available, the probability of each model is computed, so that the predictions of the more probable models are weighted more highly in the experimental design. This new methodology was recently published. The experimental implementation is now complete as well and a publication is under review. We believe that this design approach, using experimental design, will be critical for systematic design of a range wide of nanomaterials.

Training and Development:

Two PhD students have been educated under this grant. They have learned systems engineering, and also have learned about vacuum deposition systems and materials characterization. This is a unique combination and both have recently earned their PhD degrees and have begun positions in the U.S. in related fields. Rentian Xiong joined Dow to work on in-situ sensing and control in photovoltaics. Paul Wissmann joined Owens-Corning in their advanced process control division.

Five undergraduate students have also worked with us on the project. Their focus has been on the experimental work, and they have also learned a number of skills in deposition systems and characterization, as well as problem solving and trouble-shooting. A local high school math teacher also worked with our group during the summer of 2008, supported by an RET supplement. She worked with the experimental design project on data analysis. In addition to her training in our lab, she can bring this research experience back to share with her students. This outreach is described in the following section of the report.

Outreach Activities:

Professor Grover has been working with Atlanta area high school math classes to develop curriculum modules based on chemical engineering research. Over the past few years, the curriculum development has been focused on microelectronics processing, as well as how computers work. Most recently Professor Grover worked with the Georgia Tech K-12 outreach office (CEISMC) to identify a high school teacher to work with her at Georgia Tech during the summer of 2008. Mrs. Jayosree Mukherjee worked in the group for 7 weeks. Her project was to develop course materials for her math classes to reinforce concepts in data analysis and statistics, based on our experimental design project. Georgia has recently changed its performance standards for high school math, and now includes data analysis and statistics in each year of high school math. Unfortunately, many teachers do not have experience in this area, and a single text book that covers all topics has yet to be identified. Professor Grover is now working with CEISMC to adapt Mrs. Mukherjee's course materials into the TeachEngineering format, so that they can be widely disseminated across Georgia schools.

Journal Publications

Rentian Xiong, Paul J. Wissmann, Martha A. Gallivan, "An extended Kalman filter for in situ sensing of yttria-stabilized zirconia in chemical vapor deposition", Computers & Chemical Engineering, p. 1657, vol. 30, (2006). Published,

Rentian Xiong and Martha A. Grover, "A modified moving horizon estimator for in situ sensing of a chemical vapor deposition process", IEEE Transactions on Control Systems Technology, p. , vol. , (2009). Accepted,

Rentian Xiong and Martha A. Grover, "In situ estimation of thin film growth rate, complex refractive index, and roughness during chemical vapor deposition using a modified moving horizon estimator", Journal of Applied Physics, p. 124901, vol. 103, (2008). Published,

P. J. Wissmann and M. A Grover, "A new approach to batch process optimization using experimental design", AIChE Journal, p. 341, vol. 55, (2009). Published,

P. J. Wissmann and M. A. Grover, "Optimization of a chemical vapor deposition process using sequential experimental design", Industrial and Engineering Chemistry Research, p. , vol. , (2009). Submitted,

Books or Other One-time Publications

R. Xiong and M. A. Gallivan, "Moving horizon estimation for in situ monitoring of chemical vapor deposition process", (2007). Conference Proceedings, Published
Collection: Proceedings of the American Control Conference
Bibliography: New York, page 3648-3653

R. Xiong and M. Grover Gallivan, "Real-time monitoring of thin film microstructure in chemical vapor deposition using a modified moving horizon estimation", (2008). Conference Proceedings, Published
Collection: Proceedings of the International Federation on Automatic Control
Bibliography: Seoul, South Korea

Web/Internet Site

URL(s):

http://www.chbe.gatech.edu/grover

Description:

The website for my research group features this project, including individual pages for each student with research summaries. Conference papers are also posted on this website.

Other Specific Products

Contributions

Contributions within Discipline:

In process systems engineering, the development of our new approach to experimental design is a key contribution. In the design of complex systems, whether in materials, nanotechnology, or synthetic biology, one must proceed with design, having only partial models, and this methodology is developed for this situation.

Contributions to Other Disciplines:

The application of moving horizon estimation in in-situ sensing can have wide application beyond reflectometry. The interpretation of in-situ sensors based on optical, electron, or x-ray beams, is a key challenge, and limits the application of feedback control in surface processing.

Contributions to Human Resource Development:

The two Ph.D. students working on this project graduated during the past year. They have both accepted positions working in U.S. technology industries. Additionally, approximately 5 undergraduate students and one high school teacher have worked on this project over its duration.

Contributions to Resources for Research and Education:

The chemical vapor deposition testbed has provided invaluable data to this project. The sensors and data acquisition system are a unique feature.

Contributions Beyond Science and Engineering:

While there has been no direct impact of this project on public welfare, this investigator believes that the rational design of nanomaterials using models can have broad impact in the long term. Additionally, the training of students in technology, and especially in process systems engineering, is critical for future U.S. competitiveness.

Conference Proceedings

Categories for which nothing is reported:

Any Product
Any Conference