

Nanoprobe Laboratory for Bio- & Nanotechnology and Biomimetics (NLBB) W390 Scott Laboratory, 201 W. 19th Avenue

The Ohio State University, Columbus, Ohio 43210-1142 USA

Advancing the State-of-the-Art in Nanotribology, Nanomechanics, Nanomaterials Characterization, and Bioinspired Nanostructured Surfaces

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Director's Report

by: Professor Bharat Bhushan Ohio Eminent Scholar and The Howard D. Winbigler Professor

Goals and Scope



NLBB's main goals are to carry out fundamental studies in the interdisciplinary areas of bioand nanotribology, bio- and nanomechanics and bio- and nanomaterials characterization and applications to bio- and

nanotechnology. The scope of the research includes the development of theoretical models, development of sophisticated instrumentation and techniques, fabrication of nanostructures, and the acquisition of research data for fundamental understanding. Measurement of mechanical and electrical properties of nanostructures is carried out. Self-assembled monolayers and perfluoropolyether lubricant and ionic liquid films are developed for micro- or nanostructures. Biomolecular films are studied for biomedical applications. In particular, bioadhesion, friction and wear issues are addressed. Another main goal is to develop bio-inspired nanostructured There is а large activity surfaces. on superhydrophobic and self-cleaning surfaces with low drag (Lotus effect) and interfaces with smart adhesion (Gecko feet). There are also research activities in nanoprobe based data recording technology and beauty care products (hair, hair conditioner, and skin cream). Instrumentation used in the lab includes atomic force microscopy /scanning tunneling microscopy (AFM/STM), microtriboapparatus, nanoindenter. and industrial simulators.

The research activity is truly interdisciplinary, embodying materials science, physics, physical chemistry, biomedical engineering, biotribology, lubricant rheology, mechanics, applied mathematics, bio/nanomaterials characterization, biomimetics, and applications to bio/nanotechnology.

Facilities and Equipment

The Nanoprobe Laboratory is housed in Scott Laboratory. The laboratory occupies more than 4000 square feet of space. Office space includes offices for the two faculty members, an assistant, six visiting scientists/industrial fellows, and 16 graduate students. Part of the laboratory is environmentally controlled and contamination controlled to a specification of Class 100. The air tables are used to provide vibration isolation.

The laboratory is equipped with many pieces of sophisticated research equipment including a scanning tunneling microscope, five atomic force microscopes, noncontact optical profiler, stylus profiler, nanoindenter, ultra-high vacuum tribotest apparatus, microtriboapparatus, and scanning ellipsometer, and contact angle measurement The other pieces of equipment and apparatus. facilities include vapor and liquid coating-deposition facilities. pin-on-disk continuous sliding test apparatus, reciprocating sliding test apparatus, optical microscope. microhardness tester. metallurgical sample preparation and polishing facility. particle counter. microbalance. and environmental chambers.

Central facilities for micro/nanofabrication of structures are used for bio/nanotechnology and biomimetics research. An easy access exists to various physical and chemical analysis facilities such as scanning electron microscope, scanning transmission electron microscope, EDS, scanning Auger spectrometry, X-ray photon spectroscopy, time-of-flight SIMS, DTA, DSC, TMA, TGA balances, porosimeter, NMR, FTIR, mass spectrometry, laser spectroscopy, and computer controlled mechanical test equipment.

The department has extensive computer facilities. In addition, the University maintains the Ohio Supercomputer Center. All of the campus computers are connected electronically.

Technical Report

2013 has been a busy and productive year for the Nanoprobe Laboratory for Bio- & Nanotechnology and Biomimetics.

Sponsors

Industrial Procter & Gamble Sonv

Government Sponsors

National Science Foundation Department of Energy ACS Petroleum Research Fund

Instrumentation Support

Hysitron Bruker/Tribology & Mech. Testing

Following is a partial list of ongoing projects at NLBB:

Nanotribology and Nanomechanics

- Nanotribology and Nanomanipulation of Nanoobjects in Liquid Environments Using AFM
- Nanotribology of Polymers

Bio- & Nanotechnology

- Imaging of Nanobubbles and Measurement of Surface Charge and Slip on Hydrophobic Surfaces
- Electrowetting and Tunable
 Superhydrophobicity
- Bioadhesion of Various Proteins on Block Copolymer Films for Bone Regeneration Scaffold Applications (with Prof. S. Schricker, College of Dentistry)

Biomimetics and Green Tribology

- Lotus-inspired Durable and Transparent Superhydrophobic and Superoleophobic Surfaces for Anti-Smudge, Self-Cleaning, and Antifouling
- Shark-skin, Rice Leaf and Butterfly Wings Inspired Structured Surfaces for Low Drag and Antifouling
- Fluid Flow Modeling of Biomimetic Structures
- Other Projects: Fish and Nacre

Beauty Care Products

 Adhesion, Friction, and Wear of Skin and Skin Cream Using AFM and Development of Synthetic Skin

Batteries Aging

 Multi-scale Characterization of Aging Phenomena and In-situ Electrochemistry for Li-Ion Batteries.

NLBB Publications

Technical Papers (from 2013)

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- 2. Sun, J., Bhushan, B., and Tong, J., "Structural Coloration in Nature," (Invited), *RSC Advances* **3**, 14862-14899 (2013).
- 3. Bixler, G.D. and Bhushan, B., "Fluid Drag Reduction with Shark-skin Riblet Inspired Microstructured Surfaces," (Invited), Advanced Functional Materials) 23, 4507-4528 (2013); Highlighted in Materials Views.
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- 15. Pan, Y., Bhushan, B., and Maali, A., "Slip Length Measurement of Confined Air Flow on Three Smooth Surfaces," *Langmuir* **29**, 4298-4302 (2013).
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- 3. Bhushan, B., "Tractor/Trailer Safety Device," U.S. Patent No. 4,269, 426, May 6, 1981.
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- 5. Bhushan, B., Coe, J. V., and Gupta, B. K., "Method for Coating Fullerene Materials for Tribology," U.S. Patent No. 5,558,903, September 24, 1996.
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- 12. Bhushan, B. and Hahm, C. D., "Pin and Cup Devices for Measuring Film Thickness," U.S. Patent No. 6, 724, 199 B1, April 20, 2004.
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- 16. Bhushan, B., Jung, Y. C., and Nosonovsky, M., "Hierarchical Structures for Superhydrophobic Surfaces and Methods of Making," U. S. Patent No. 8,137,751 B2, March 20, 2012.
- 17. Bhushan, B., Jung, Y. C., and Nosonovsky, M., "Hierarchical Structures for Superhydrophobic Surfaces and Self-Cleaning and Methods of Making," U. S. Patent Pending.
- 18. Ebert, D.R. and Bhushan, B., "Methods of Fabricating Superhydrophobic, Optically Transparent Surfaces," U.S. Patent Pending.
- 19. Bixler, G. D. and Bhushan, B., "Fluid Conveying Apparatus with Low Drag, Anti-fouling Flow Surface and Methods of Making Same," U.S. Patent Pending.
- 20. **Published:** Bhushan, B. and Nosonovsky, M., "Hydrophobic Surface with Geometric Roughness Pattern," U.S. Patent Application Publication No. US 2006/0078724 A1, April 13, 2006.

Editor of Book Series

- 1. Avoris, P., Bhushan, B., Bimberg, D., von Klitzing, K., Sasaki, H., and Wiesendanger, R., NanoScience and Technology Series, Springer, Heidelberg, Germany, 2004-.
- 2. Bhushan, B., Mechanics and Materials Science Series, CRC Press Inc., Boca Raton, Florida, 1993-2002.
- 3. Bhushan, B., Advances in Information Storage Systems Series, ASME Press, New York, 1991-1993; World Scientific, Singapore, 1994-1999.

Editor-In-Chief of a Journal

 Editor-in-Chief - Microsystem Technologies (with B. Michel), Springer Berlin-Heidelberg, Vol. 8, 2002-; formerly, Journal of Information Storage and Processing Systems, Birkhauser Boston (A Division of Springer), Vol. 1-3, 1999-2001.

Editorial Advisory Board

- 1. Honorary Member, The Research Board of Advisors, *The American Biographical Institute*, Raleigh, North Carolina (1996-).
- 2. Journal of Friction and Wear, The National Academy of Sciences, Belarus (1998-).
- 3. International Journal of Surface Science and Engineering, Inderscience Publishers (2006-).
- 4. International Journal of Materials and Structural Integrity, Inderscience Publishers (2006-).
- 5. International Journal of Nanosystems, Inderscience Publishers (2006-).
- 6. Advances in Tribology, Hindawi Publishing Corporation (2007-).
- 7. Research Letters in Nanotechnology, Hindawi Publishing Corporation (2008-).
- 8. Journal of Nanotechnology, Hindawi Publishing Corporation (2008-).
- 9. The Open Mechanical Engineering Journal, Bentham Science Publishers (2007-).
- 10. The Open Ceramic Science Journal, Bentham Science Publishers (2008-).
- 11. The Open Polymer Science Journal, Bentham Science Publishers (2008-).
- 12. The Open Surface Science Journal, Bentham Science Publishers (2008-).
- 13. The Open Condensed Matter Physics Journal, Bentham Science Publishers (2008-).
- 14. The Open Colloid Science Journal, Bentham Science Publishers (2008-).
- 15. The Journal of Biotechnology & Biomaterials–Open Access, OMICS Publishing Group (2010-).
- 16. The Journal of Petroleum & Environmental Biotechnology–Open Access, OMICS Publishing Group (2010-).
- 17. Journal of Nanomedicine and Biotherapeutic Delivery-Open Access, OMICS Publishing Group (2010-).
- 18. The Journal of Biotechnology Applications, Bioinfo Publications, Mumbai, India (2010-).
- 19. Tribology in Industry, Serbian Tribology Society (2011-).
- 20. Open Journal of Organic Polymer Materials, Scientific Research Publishing, USA (2011-).
- 21. The Open Nanoscience Journal, Bentham Science Publishers (2011-).

22. Lubricants, MDPI (2011-).

23. Surface Innovations, Ice Publishing (2012-).

Annual Research Meeting

The next Annual Research Meeting will be held soon.

Short Courses Taught

- 1. "Tribology Graduate Course (Guest Lectures)," Mechanical Engineering Department, King Fahd University of Petroleum & Minerals, Dec. 2013.
- 2. "Principles and Applications of Tribology (Accelerated 4 days (16 hours) course)," School of Mechatronics Engineering, Harbin Institute of Technology, Harbin, China, Aug. 2014.

Students Updates

We presently have five postdoctoral fellows/visiting senior scientists, and five Ph.D. graduate students. The following students graduated in 2013-14:

<u>Ph.D.</u>

- 1. Gregory D. Bixler, "Bioinspired Surface for Low Drag, Self-Cleaning, and Antifouling: Shark Skin, Butterfly and Rice Leaf Effects," Ph.D. Thesis, Dept. of Mech. and Aerospace Eng., Dec. 2013.
- Yunlu Pan, "Study of the Relation Between Electric Field and the Drag of Liquid Flow on a Solid Surface in Micro/Nanoscale," Ph.D. Thesis, Dept. of Mechatronics, Harbin Institute of Technology, China (co-advised with Prof. X. Zhao), June, 2014. Abstract available at www.mecheng.osu.edu/nlbb/content/theses.

<u>M.S.</u>

1. Sanjay Ramdon, "Nanoscale Characterization of Aged Li-ion Battery Cathodes," M.S. Thesis, Dept. of Mech. and Aero. Eng., Aug. 2013.

Student Ph.D. Theses Abstracts

Full theses can be found on the web at http://www.mecheng.osu.edu/nlbb

Gregory Bixler, "Bioinspired Surface for Low Drag, Self-Cleaning, and Antifouling: Shark Skin, Butterfly and Rice Leaf Effects."

Researchers are continually inspired by living nature to solve complex challenges through the field of biomimetics. Nature thrives on effective designs while optimizing the use of precious resources, which is something that inspires engineers worldwide. Gaining a deeper understanding of nature can lead to bioinspired products that save time, money, and lives. Examples include "low drag" boat hulls inspired by shark skin as well as "self-cleaning" windows and "antifouling" medical devices inspired by the superhydrophobic and low adhesion lotus leaf. Common engineering challenges solved in nature but hindering many industries are fluid drag reduction and antifouling. Nature holds clues to these challenges, including the unique surface characteristics of rice leaves and butterfly wings that combine the shark skin (anisotropic flow leading to low drag) and lotus leaf (superhydrophobic and self-cleaning) effects, producing the so-called rice and butterfly wing effect.

In this thesis, first presented is a chapter on biofouling and inorganic-fouling which is generally undesirable for many medical, marine, and industrial applications. A survey of promising flora and fauna are studied in order to discover new antifouling methods that could be mimicked for engineering applications. New antifouling methods will presumably incorporate a combination of physical and chemical controls. Mechanisms and experimental results are presented focusing on my new drag reducing shark skin inspired surfaces. This includes my new laser etched and microtextured film samples for closed channel drag using water, oil, and air as well as in wind tunnel. Finally mechanisms and experimental results are presented focusing on my newly discovered rice and butterfly wing effect surfaces. I collected and present morphology, drag, antifouling, contact angle, and contact angle hysteresis results to understand the role of sample geometrical dimensions, wettability, viscosity, and velocity. Hierarchical liquid repellent coatings combining nano-and micro-sized features and particles are utilized to recreate or combine various effects. I fabricated such samples with photolithography, soft lithography, hot embossing, and coating techniques. Discussion is provided along with new conceptual models describing the role of surface structures related to low drag and antifouling properties. Modeling provides design guidance when developing novel low drag and antifouling surfaces for medical, marine, and industrial applications.

Student M.S. Theses Abstracts

Sanjay Ramdon, Nanoscale Characterization of Aged Li-Ion Battery Cathodes

Lithium-ion (Li-ion) batteries have become very prominent as a form of energy storage for numerous applications due to its high energy and power densities. They are used for numerous portable devices and more recent electric vehicles (EVs). It is important to increase the cycle life of Li-ion batteries in order for them to be more viable for the automotive industry. With use, these batteries undergo an aging process which reduces the battery storage capacity and increases internal resistance. To reduce the aging process it is essential to first understand the degradation mechanisms on the electrodes of the battery.

A multi-scaled approach has been previously applied to the study of the degradation of the LiFePO4 cathodes. It has been shown that nanoparticles in cathodes coarsen as a result of aging. Coarsening of nanoparticles has been shown to lead to an increase in surface resistance and decrease in surface conductivity, which is responsible for reduced lithium retaining capacity. It is therefore important to study the cause of these aging mechanisms in order to increase the life of the battery. An in depth study of cathode on the nanometer scale is necessary using atomic force microscope (AFM) related techniques. In this work, both ex-situ and in-situ studies were conducted to understand the aging phenomenon in LiFePO4 battery cathodes. High resolution AFM imaging and current measurements were conducted to study the difference of the unaged cathode from the aged. This was done to quantify the coarsening process. Particle agglomeration was observed in the aged cathode, which is believed to reduce surface conductivity.

Nanomechanical characterization and mechanical integrity studies were then conducted on unaged and aged cathodes using AFM equipped with nanoindentor. This was done to determine the effect of increased internal stress within the cathode created during aging on the nanomechanical and mechanical integrity properties. Properties of hardness, elastic modulus, creep, nanowear, nanoscratch and nanofriction were examined and significant differences were observed between unaged and aged cathodes. The aged cathodes showed higher hardness, creep depth and critical load in scratch and lower wear depth and coefficient of friction.

In order to further understand aging mechanisms, real time examination of battery cathodes were done during operation of the cell using in-situ techniques. AFM in-situ techniques allow direct observation of cathodes during cycling of the cell on the nanometer scale. Morphology data showed increase in particle size from FePO4 phase to LiFePO4 phase during discharge of the cell due to lithiation. In-situ AFM electrochemical characterization has been shown to be a useful technique to study Li-ion battery aging mechanisms.

These studies further the understanding of the degradation mechanisms of Li-ion battery cathodes. The in situ electrochemistry approach advanced in this work is believed to be useful for fundamental understanding of aging mechanisms.

Technical Seminars

We invite industrial members to visit the NLBB and present seminars. If you would like to give a seminar, please contact Professor B. Bhushan.

Honors and Awards (B. Bhushan)

- 2014 STLE International Award for Outstanding Contribution to Tribology and/or its Related Sciences; It is Society's highest honor for lifetime achievement and bestows lifetime honorary membership.
- Sept. 2013-
Aug. 2014ASME/AAAS Science & Technology Policy Fellow, House Committee on Science, Space, and
Technology; detailed to House Committee on Energy and Commerce and House Committee on
Oversight and Government Reform, United States Congress, Washington, D.C.
- Nov. 12- 14 How Butterfly Wings Can Inspire New High-Tech Surfaces, NSF Science Now (11/16/12); Covered by 2014 NSF Discoveries News, Columbus Dispatch (12/2/12); sciencenewsline.com (11/7/12); gizman.com (11/8/12); qmed.com (11/12); eetimes.com (11/8/12); redorbit.com (11/8/12); scilifestyle.com (11/12/12); microscopy-analysis.com (11/12/12); eetasia.com (11/12/12); sciencebusiness.technewslit.com (11/12/12); silobreaker.com (12/2/12); MRS Materials 360 online (12/18/12); NSF Livescience and Discovery Page (1/23/13); ISTV & American Institute of Physics (11/13); Awake (4/14), www.jw.org; The journal with the second highest worldwide monthly circulation (45 million in 99 languages).etc.
- Oct. 19, 2014 "Biomimetics: Bioinspired Superomniphobic, Self-cleaning/Antifouling and Low Drag Surfaces," Lunch with a Scientist, ScienceWriters 2014, The Annual Joint Meeting of the National Association of Science Writers (NASW) and the Council for the Advancement of Science Writing (CASW), Columbus, Ohio, 10/17-21, 2014.



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