NASA/CP—2019-219576

NASA Workshop on Low Ice Adhesion Materials

Richard E. Kreeger, Compiler
Glenn Research Center, Cleveland, Ohio

August 2019

NASA STI Program . . . in Profile

Since its founding, NASA has been dedicated to the advancement of aeronautics and space science. The NASA Scientific and Technical Information (STI) Program plays a key part in helping NASA maintain this important role.

The NASA STI Program operates under the auspices of the Agency Chief Information Officer. It collects, organizes, provides for archiving, and disseminates NASA's STI. The NASA STI Program provides access to the NASA Technical Report Server—Registered (NTRS Reg) and NASA Technical Report Server—Public (NTRS) thus providing one of the largest collections of aeronautical and space science STI in the world. Results are published in both non-NASA channels and by NASA in the NASA STI Report Series, which includes the following report types:

- TECHNICAL PUBLICATION. Reports of completed research or a major significant phase of research that present the results of NASA programs and include extensive data or theoretical analysis. Includes compilations of significant scientific and technical data and information deemed to be of continuing reference value. NASA counter-part of peer-reviewed formal professional papers, but has less stringent limitations on manuscript length and extent of graphic presentations.

- TECHNICAL MEMORANDUM. Scientific and technical findings that are preliminary or of specialized interest, e.g., "quick-release" reports, working papers, and bibliographies that contain minimal annotation. Does not contain extensive analysis.

- CONTRACTOR REPORT. Scientific and technical findings by NASA-sponsored contractors and grantees.

- CONFERENCE PUBLICATION. Collected papers from scientific and technical conferences, symposia, seminars, or other meetings sponsored or co-sponsored by NASA.

- SPECIAL PUBLICATION. Scientific, technical, or historical information from NASA programs, projects, and missions, often concerned with subjects having substantial public interest.

- TECHNICAL TRANSLATION. English-language translations of foreign scientific and technical material pertinent to NASA's mission.

For more information about the NASA STI program, see the following:

- Access the NASA STI program home page at http://www.sti.nasa.gov

- E-mail your question to help@sti.nasa.gov

- Fax your question to the NASA STI Information Desk at 757-864-6500

- Telephone the NASA STI Information Desk at 757-864-9658

- Write to:
  NASA STI Program
  Mail Stop 148
  NASA Langley Research Center
  Hampton, VA 23681-2199

NASA/CP—2019-219576

NASA Workshop on Low Ice Adhesion Materials

Richard E. Kreeger, Compiler
Glenn Research Center, Cleveland, Ohio

Proceedings of a conference held at and sponsored by
Ohio Aerospace Institute
Brook Park, Ohio
August 10, 2017

National Aeronautics and
Space Administration

Glenn Research Center
Cleveland, Ohio 44135

August 2019

Acknowledgments

The authors wish to thank Ashraf Bastawros, Laura Becker, Galdemir Botura, Michelle Berg, Paula Dempsey, Ali Dhinojwala, Hui Hu, Susan Johnson, Ki-Han Kim, Jiong Liu, Tony Nerone, Jose Palacios, Ganesh Skandan, Joe Smith, David Thompson, Jen-ching Tsao, Anesh Tuteja, Mario Vargas, Yan Wang, Chris Wohl, Mary Wadel, Andrew Work, and Paulette Ziegfeld.

This report contains preliminary findings, subject to revision as analysis proceeds.

Contents were reproduced from author-provided presentation materials.

This work was sponsored by the Advanced Air Vehicle Program at the NASA Glenn Research Center.

Level of Review: This material has been technically reviewed by technical management.

Available from

NASA STI Program
Mail Stop 148
NASA Langley Research Center
Hampton, VA 23681-2199

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
703-605-6000

Contents

Introduction

The first NASA Workshop on Low Ice Adhesion Materials was held on Thursday, August 10, 2017, at the Ohio Aerospace Institute in Cleveland, Ohio. This meeting allowed government, industry, and academia to meet in a collaborative environment to discuss the future of "icephobics" research for inflight icing. NASA presented its ongoing research, and organizations that currently have partnerships with NASA presented their recent findings. Presenters from academia included Iowa State University, Mississippi State University, Pennsylvania State University, and the University of Michigan. Presenters from industry included United Technologies Aerospace Systems; Nanosonic, Inc.; and NEI Corporation. Researchers from NASA Glenn Research Center, NASA Langley Research Center, and the Office of Naval Research also shared their current research. There were over 60 participants who attended the conference plus more than 10 who participated remotely. The meeting was highly successful, and although a second such conference was proposed, it is anticipated that future technical presentations on the subject(s) will take place in other venues. Presentations from this workshop that are suitable for public release are included in this document. This effort supports both the Advanced Air Transport Technology (AATT) Project and the Revolutionary Vertical Lift Technology (RVLT) Project.

Ice Adhesion Research at NASA GRC

Andrew Work & Eric Kreeger

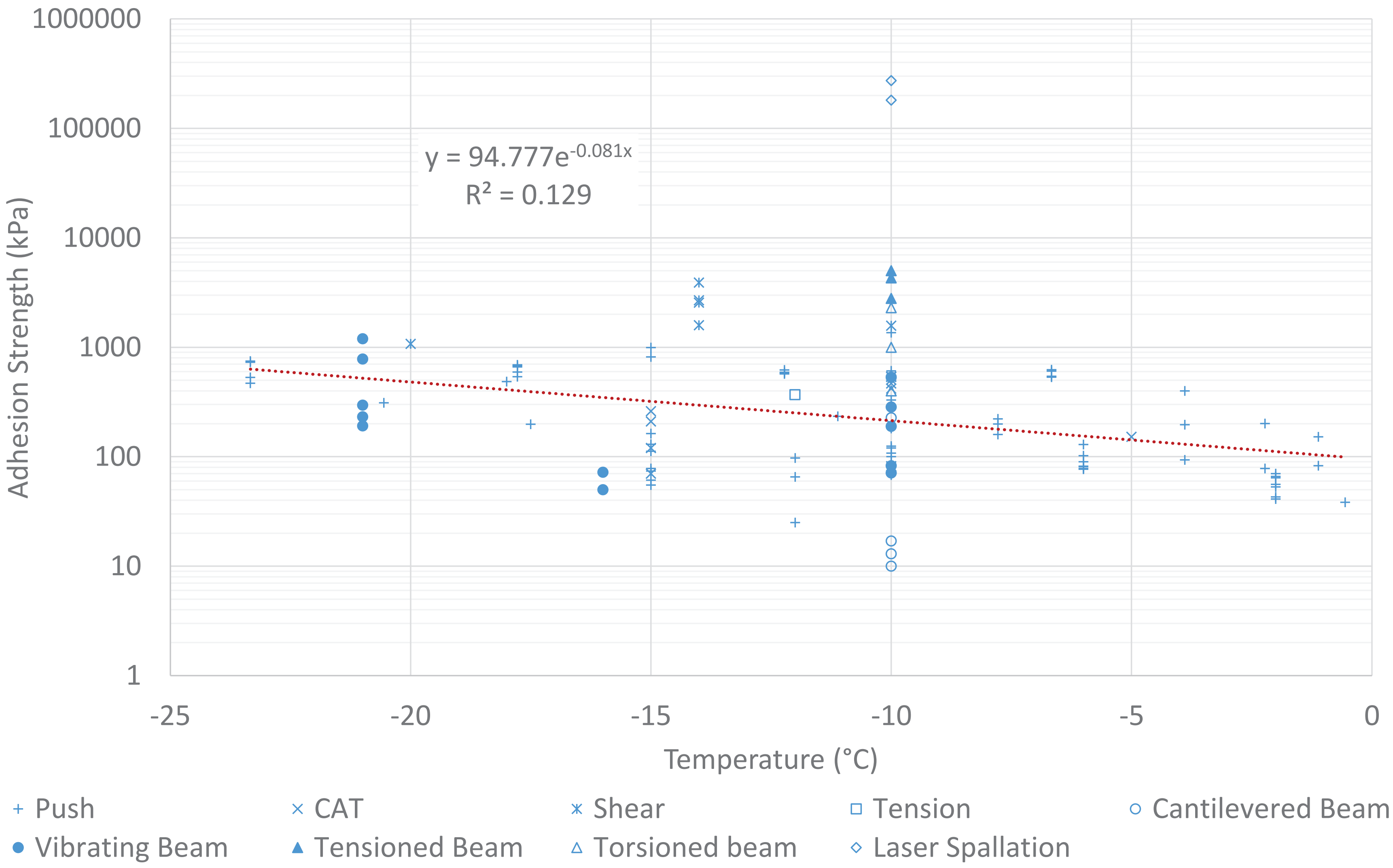
GRC Goals

- Measure the adhesion of ice to aircraft materials
- Measure tensile properties of ice for modeling
- Develop a shedding model for use in LEWICE
  - Rotorcraft, deicing equipment, engine icing
- Develop a quantitative method for testing the adhesion of ice to low-ice-adhesion surfaces (Icephobics)

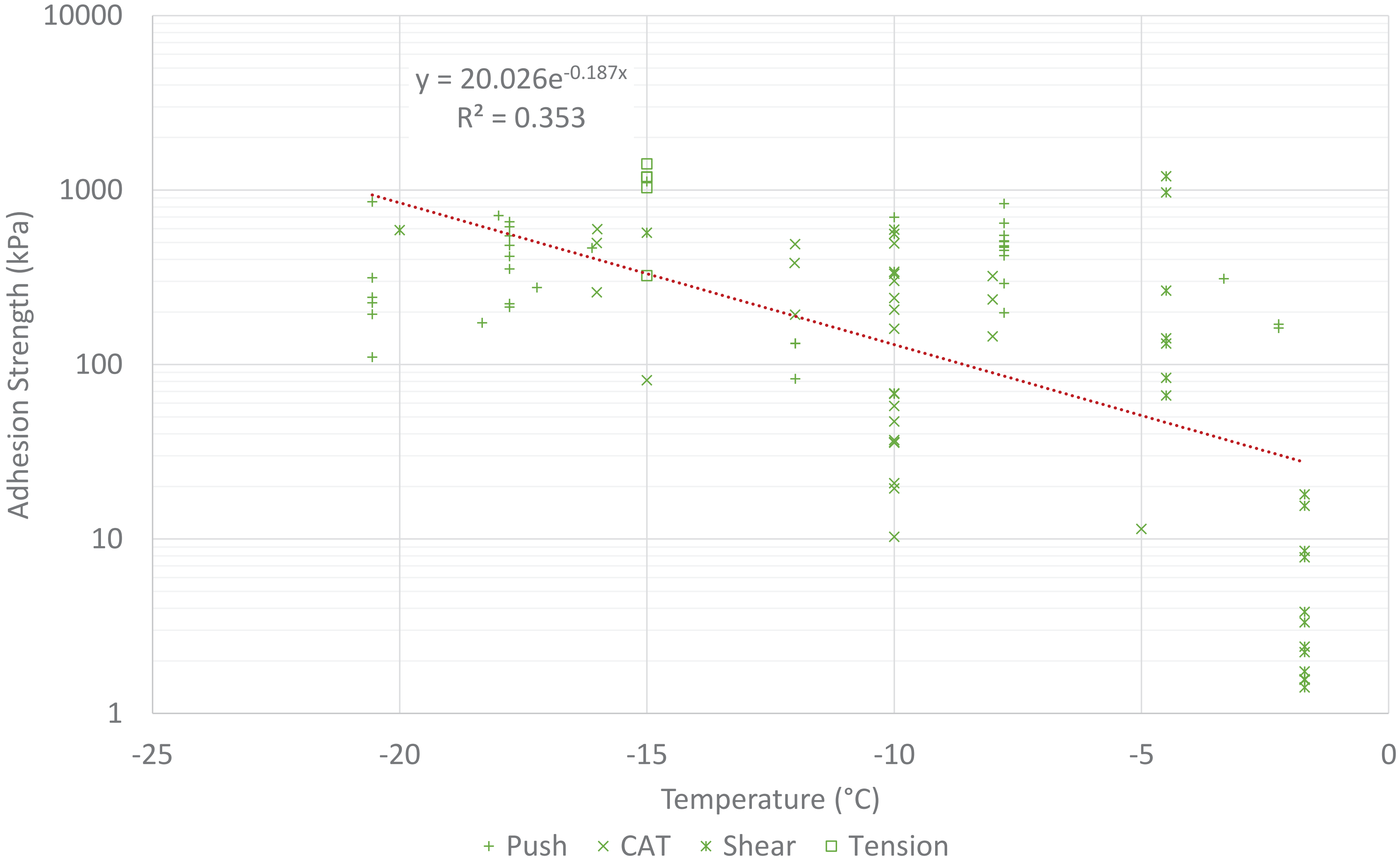
A Critical Review of the Measurement of the Adhesion of Ice to Solid Substrates

- Review of the literature on the measurement of the adhesion of ice
  - Publication pending
  - 110+ articles presenting adhesion testing on ice included
- Has references for data on the next two slides
- Each data point averaged set of >= 2 data points from literature

Data in the Literature - Aluminum



Data in the Literature - Steel

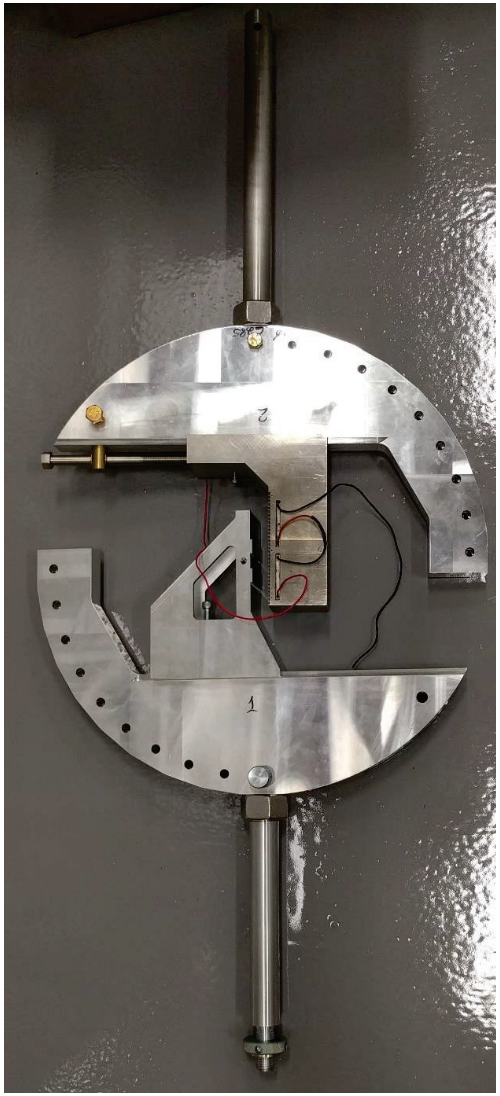


Planned Methodology

- New IRT model (XT Model) to collect samples of ice
  - 48 per run
  - Wrapped in airtight bags, carried to FASTLab
- Walk-in freezer
  - Obtain 3D scans of ice
  - Perform microscopy
  - Microtome to cut ice
  - Store ice long term to test for time-dependent effects
- Test in temperature/humidity controlled chamber
  - Window & glove ports for strain imaging

Our Lap Test

- Samples mounted on dovetail rails
- Potential problems:
  - Melting and refreezing ice away from interface
  - Handling could damage samples
  - Temperature change in IRT test section and transit could damage samples
- Potential Advantages:
  - Allows 2D/3D strain measurement
  - Stress state at interface can be modified by rotating rig
  - Testing under compression possible
  - Preserves sample of ice for further measurements
  - Could potentially pre-crack interface
  - Flexible sample geometry



FAST Lab

- Plan to develop ability to determine other material properties
- Compare to other test methods
  - Centrifuge test on order
- Plan to develop in-situ methods
- First IRT test October 2nd/3rd

Questions?

Effect of Hydrogen-Bonding Surfaces upon Ice Adhesion Shear Strength

Joseph G. Smith Jr. and Christopher J. Wohl

NASA Langley Research Center, Hampton, VA 23681, United States of America

NASA Workshop on Low Ice Adhesion Materials

Ohio Aerospace Institute, Cleveland, Ohio

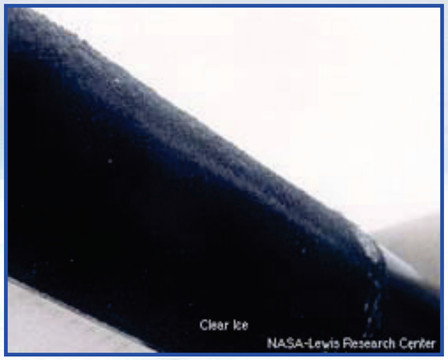
Background

- Icing
  - Ground problem during cold months
    - Freezing drizzle/rain
  - In-flight problem year round
    - Results from supercooled water droplets impacting the aircraft surface while flying through a cloud
    - Most occurrences are between 0 and -20°C

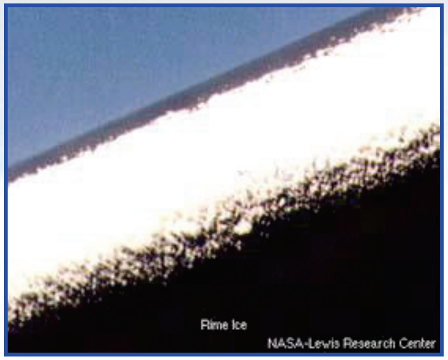
- Icing types encountered in-flight
  - Glaze/Clear, Rime, Mixed
  - Dependent upon
    - Air temperature (-5 to -20°C)
    - Liquid water content (0.3-0.6 g/m^3)
    - Droplet size (median volumetric diameter of 15-40 µm)

M.K. Politovich, “Aircraft Icing” in Encyclopedia of Atmospheric Sciences, Academic Press, Oxford, 2003, 68-75.
H.E Addy Jr., M.G. Potapczuk, and D.W. Sheldon, “Modern Airfoil Ice Accretions,” NASA TM 107423, 1997.

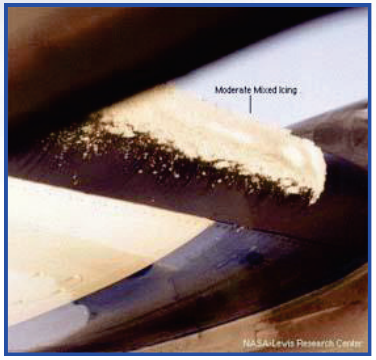
Accreted Ice Types



Glaze/Clear



Rime



Mixed

- Large droplets
- Clear, nearly transparent, smooth, waxy thus hard to see
- Gradual freezing after droplet impact can result in runback along surface generating raised edges (i.e. horns)
- Difficult to remove

- Small droplets
- Brittle and opaque, milky appearance
- Rapid freezing after droplet impact with growth into the airstream
- Easier to remove than glaze

- Variable droplet size
- Combination of glaze and rime ice

M.K. Politovich, “Aircraft Icing” in Encyclopedia of Atmospheric Sciences, Academic Press, Oxford, 2003, 68-75.
H.E Addy Jr., M.G. Potapczuk, and D.W. Sheldon, “Modern Airfoil Ice Accretions,” NASA TM 107423, 1997.

Objective

To assess the effect of surface chemical functionalities upon ice adhesion shear strength (IASS)

Approach

Investigate IASS of coated surfaces having controlled chemical functionality and carbon chain length between the substrate surface and the chemical functionality

- Prepare and characterize substituted n-alkyldimethylalkoxysilanes containing hydrogen bonding and non-hydrogen bonding groups
- Prepare and characterize aluminum (Al) substrates coated with substituted n-alkyldimethylalkoxysilanes
  - Receding water contact angle - First Ten Angstroms FTA 1000B goniometer
  - Surface roughness - Bruker Dektak XT Stylus Profilometer
- Determine IASS of coated Al substrates in a simulated environment with comparison to uncoated Al as the control
  - Adverse Environment Rotor Test Stand (AERTS)

Adverse Environment Rotor Test Stand

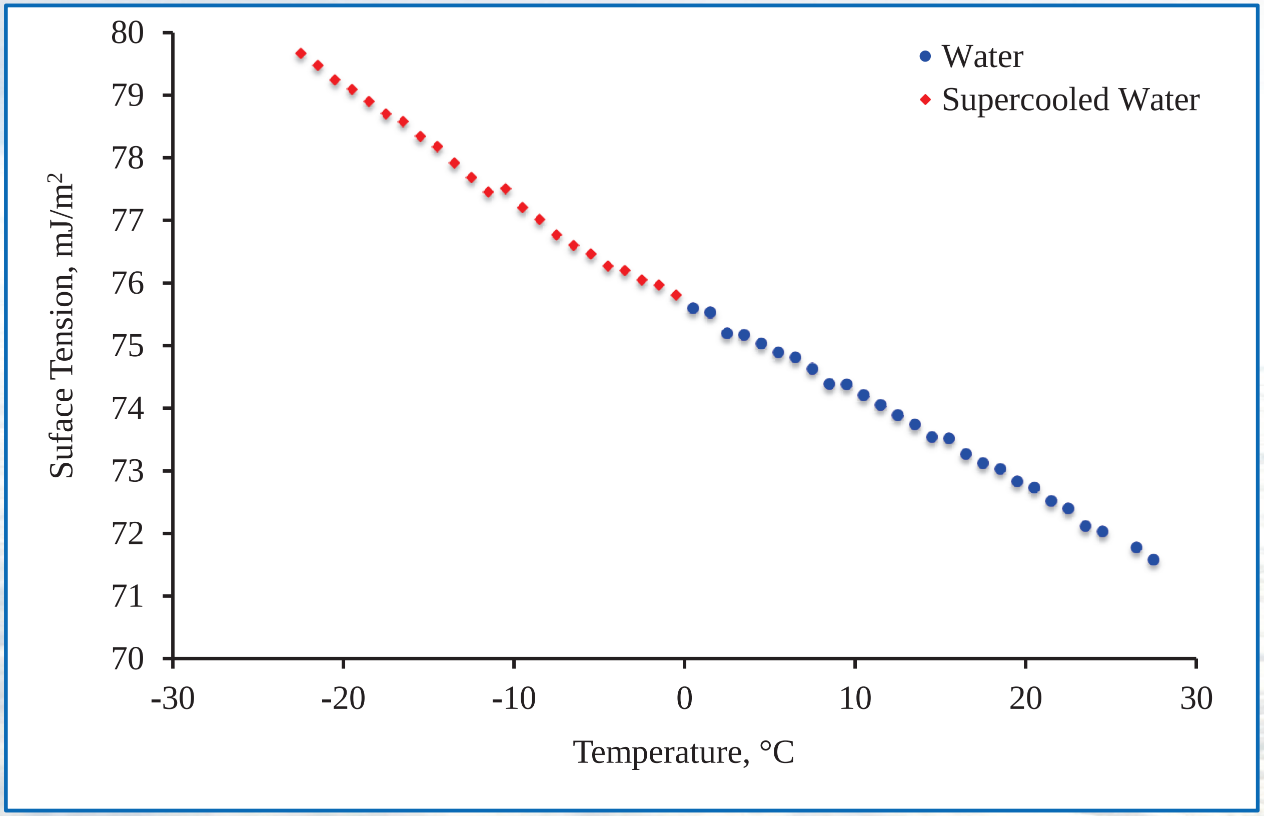
- ❄ Pennsylvania State University
- ❄ Testing performed under simulated icing conditions within the FAR Part 25/29 Appendix C icing envelope
    - Supercooled water injected into test chamber
    - Tests conducted at -8, -12, and -16°C
    - Icing cloud density (i.e. liquid water content) of 1.9 g/m^3
    - Water droplet mean volumetric diameter of 20 μm

Credit: J. Palacios

- ❄ Ice accumulation and subsequent shedding enabled determination of IASS after data analysis and visual assessment

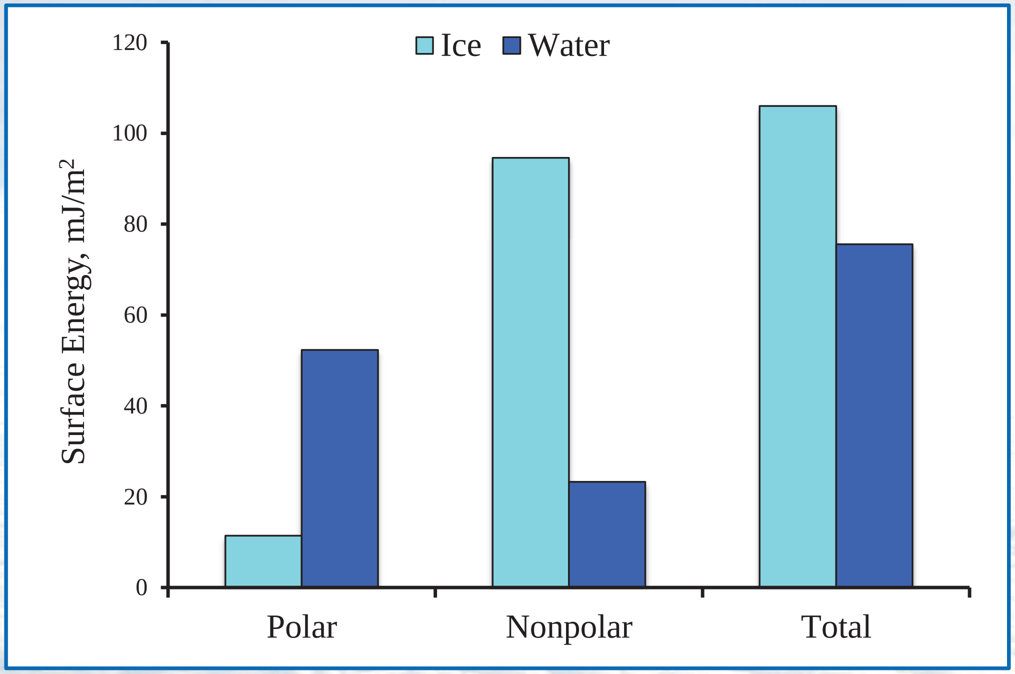
J. Soltis, J. Palacios, T. Eden, and D. Wolfe, "Evaluation of Ice Adhesion Strength on Erosion Resistant Materials," 54th AIAA/ASME/ASCE/AHS/ASC Structures, Structural Dynamics, and Materials Conference, Apr 8-11, 2013, Boston, MA, AIAA 2013-1509.

Surface Tension of Supercooled Water



Graph created from data in P. T. Hacker, “Experimental values of the surface tension of supercooled water,” Technical Note 2510, National Advisory Committee for Aeronautics, 1951.

Surface Energy at 0°C

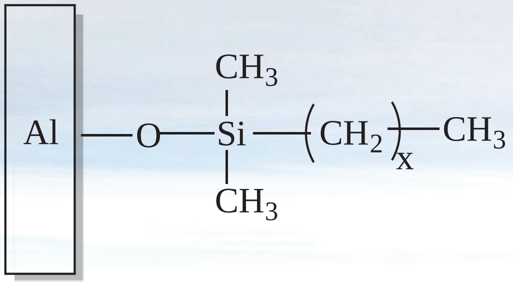


Upon phase change from water to ice, ice exhibits a high nonpolar characteristic even though it has a high total surface energy like water

Graph created from data in J. Kloubek, "Calculation of Surface Free Energy Components of Ice According to Its Wettability by Water, Chlorobenzene, and Carbon Disulfide," *J. Colloid Interf. Sci.*, Vol. 46, 1974, pp. 185-190.

NASA Workshop on Low Ice Adhesion Materials, 10 August 2017

Substituted n-Alkyldimethylsilyl Coatings

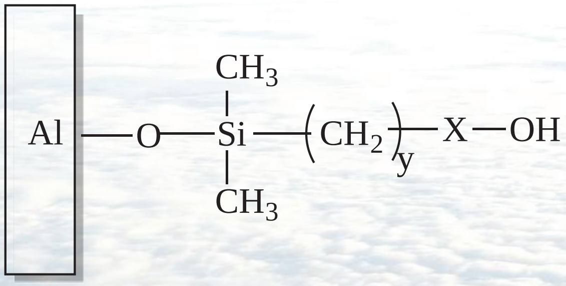


$Al-O-Si(CH_3)_2-(CH_2)_x-CH_3$

non-Hydrogen Bonding

Aliphatic

x = 2 (C3A), 6 (C7A), 10 (C11A)

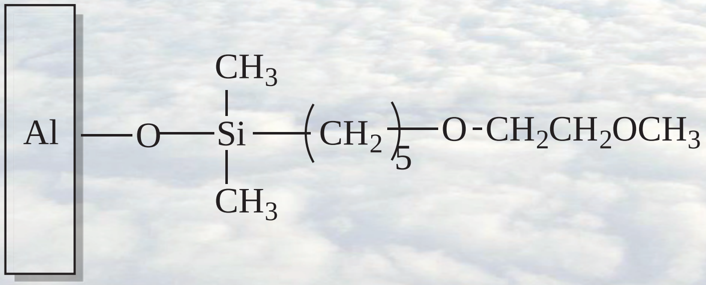


$Al-O-Si(CH_3)_2-(CH_2)_y-X-OH$

Hydrogen Bonding (Donor/Acceptor)

Hydroxyl

X = bond, y = 7 (C7H), 10 (C10H), 11 (C11H)

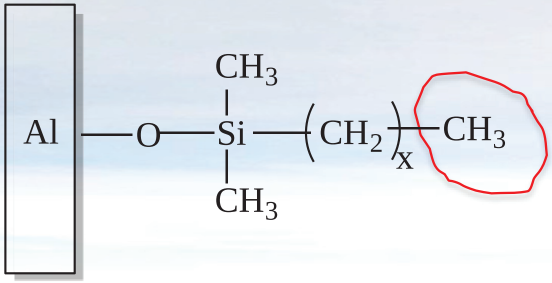


$Al-O-Si(CH_3)_2-(CH_2)_5-O-CH_2CH_2OCH_3$

Hydrogen Bonding (Acceptor)

C5MEG

Substituted n-Alkyldimethylsilyl Coatings

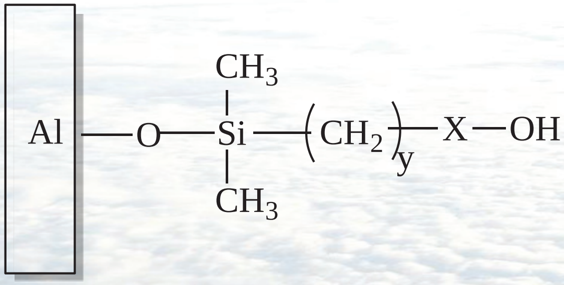


$$\mathrm{Al-O-\overset{CH_3}{\underset{CH_3}{Si}}-(CH_2)_x-CH_3}$$

non-Hydrogen Bonding

Aliphatic

x = 2 (C3A), 6 (C7A), 10 (C11A)

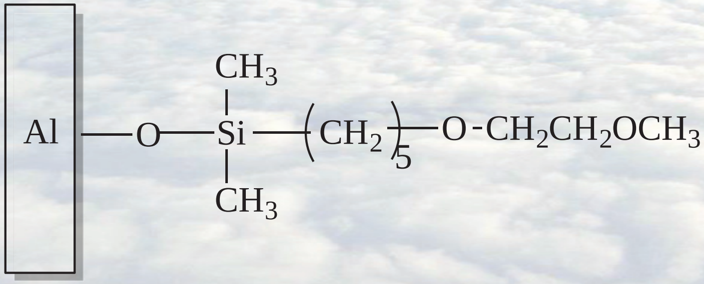


$$\mathrm{Al-O-\overset{CH_3}{\underset{CH_3}{Si}}-(CH_2)_y-X-OH}$$

Hydrogen Bonding (Donor/Acceptor)

Hydroxyl

X = bond, y = 7 (C7H), 10 (C10H), 11 (C11H)

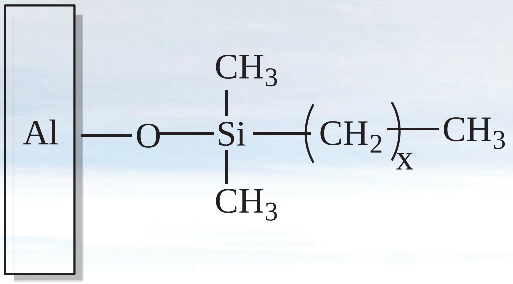


$$\mathrm{Al-O-\overset{CH_3}{\underset{CH_3}{Si}}-(CH_2)_5-O-CH_2CH_2OCH_3}$$

Hydrogen Bonding (Acceptor)

C5MEG

Substituted n-Alkyldimethylsilyl Coatings

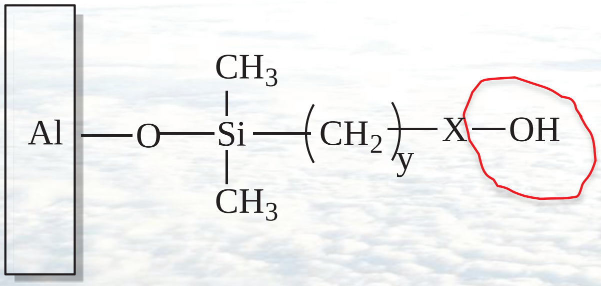


$$\mathrm{Al{-}O{-}Si(CH_3)_2{-}(CH_2)_x{-}CH_3}$$

non-Hydrogen Bonding

Aliphatic

x = 2 (C3A), 6 (C7A), 10 (C11A)

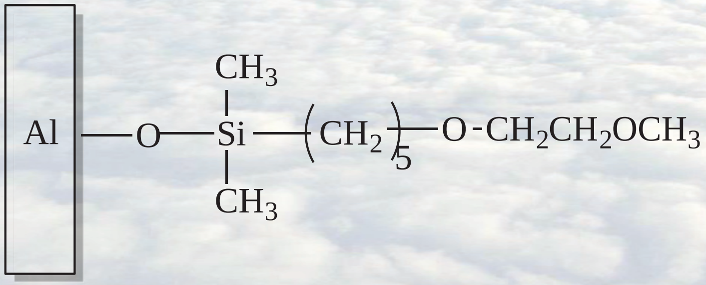


$$\mathrm{Al{-}O{-}Si(CH_3)_2{-}(CH_2)_y{-}X{-}OH}$$

Hydrogen Bonding (Donor/Acceptor)

Hydroxyl

X = bond, y = 7 (C7H), 10 (C10H), 11 (C11H)

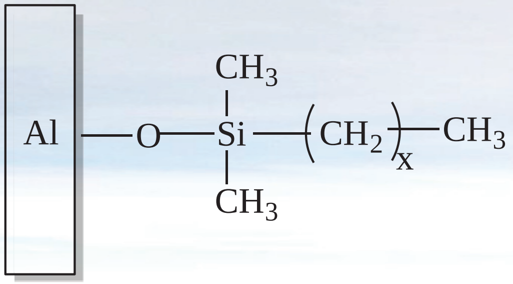


$$\mathrm{Al{-}O{-}Si(CH_3)_2{-}(CH_2)_5{-}O{-}CH_2CH_2OCH_3}$$

Hydrogen Bonding (Acceptor)

C5MEG

Substituted n-Alkyldimethylsilyl Coatings

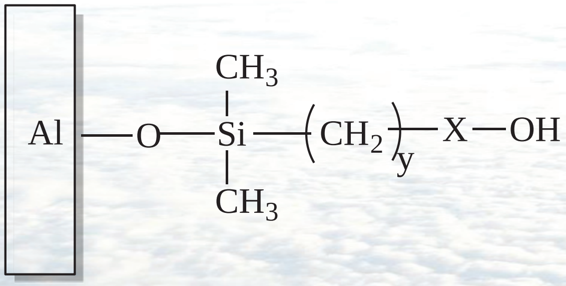


$\mathrm{Al{-}O{-}Si(CH_3)_2{-}(CH_2)_x{-}CH_3}$

non-Hydrogen Bonding

Aliphatic

x = 2 (C3A), 6 (C7A), 10 (C11A)

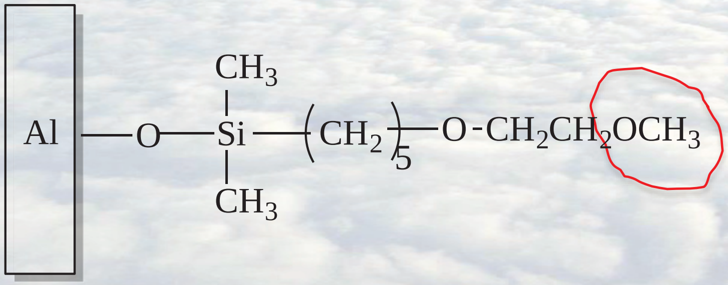


$\mathrm{Al{-}O{-}Si(CH_3)_2{-}(CH_2)_y{-}X{-}OH}$

Hydrogen Bonding (Donor/Acceptor)

Hydroxyl

X = bond, y = 7 (C7H), 10 (C10H), 11 (C11H)



$\mathrm{Al{-}O{-}Si(CH_3)_2{-}(CH_2)_5{-}O{-}CH_2CH_2OCH_3}$

Hydrogen Bonding (Acceptor)

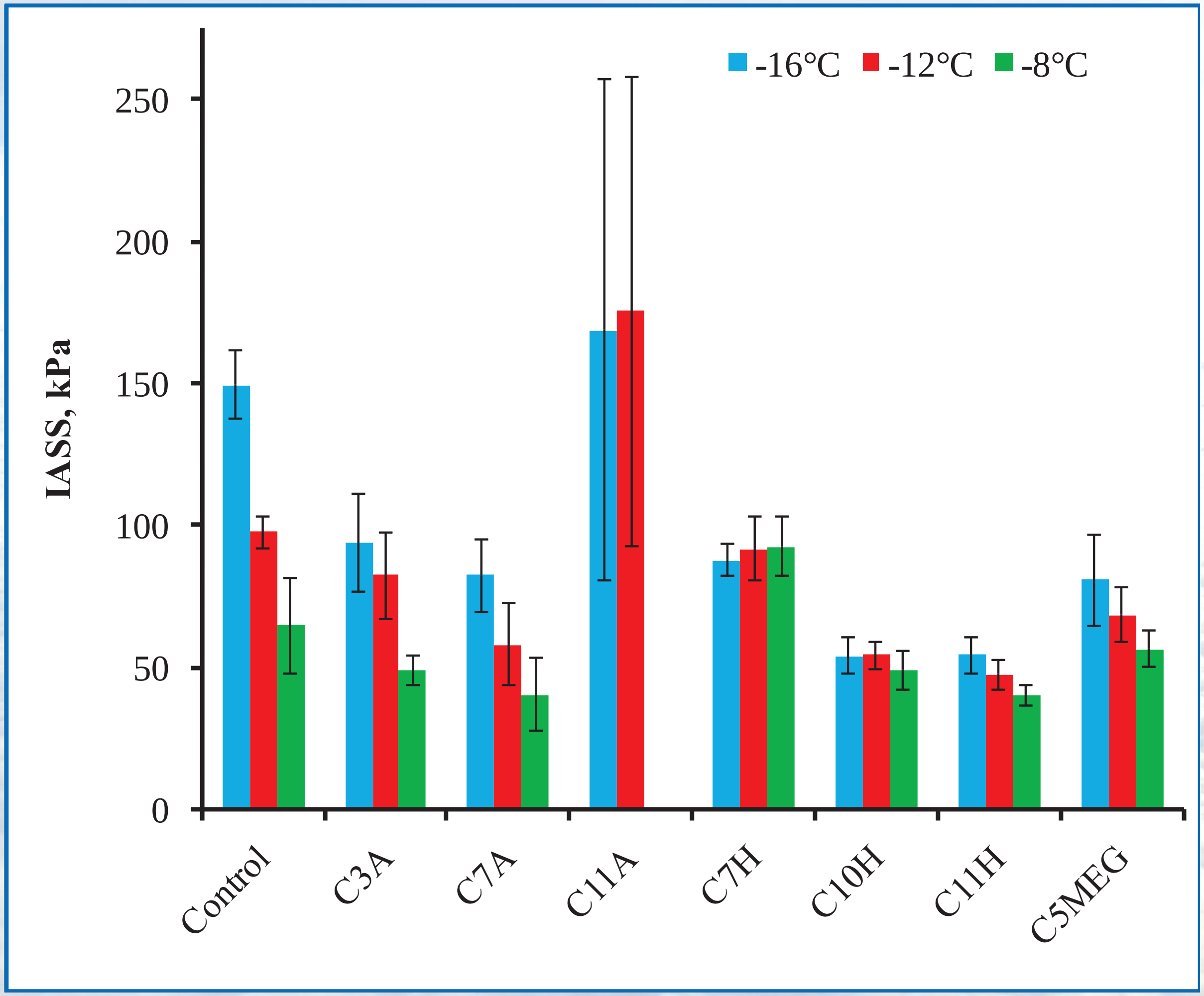
C5MEG

Surface Properties of Neat Substituted n-Alkyldimethylsilyl Coatings

Surface	Mean Roughness (Ra), μm		Receding Water Contact Angle, °	
	Avg	Stnd Dev	Avg	Stnd Dev
Control	0.326	0.048	58	14
non-Hydrogen-Bonding				
C3A	0.324	0.078	87	2
C7A	0.282	0.105	88	2
C11A	0.702	0.298	78	5
Hydrogen Bonding (Donor/Acceptor)				
C7H	0.512	0.013	73	3
C10H	0.708	0.100	24	2
C11H	0.320	0.040	31	2
Hydrogen Bonding (Acceptor)				
C5MEG	0.390	0.199	79	1

ASTM A480: Finish #7

Ice Adhesion Shear Strength of Substituted n-Alkyldimethylsilyl Coatings

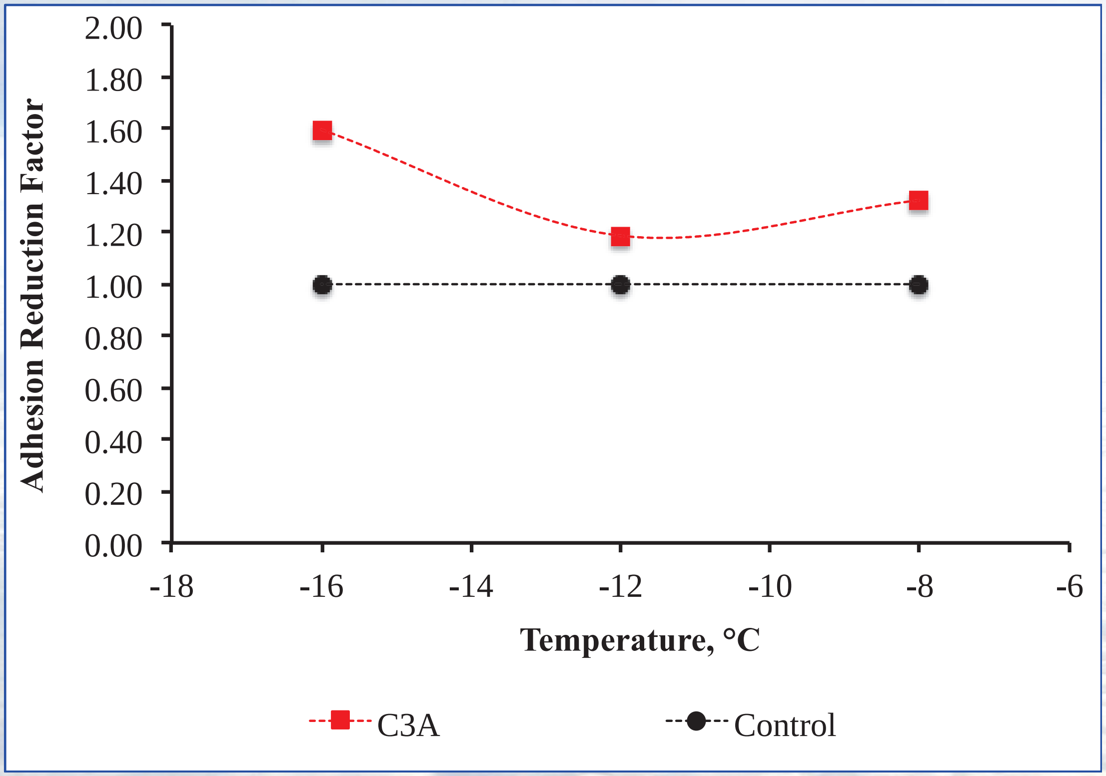


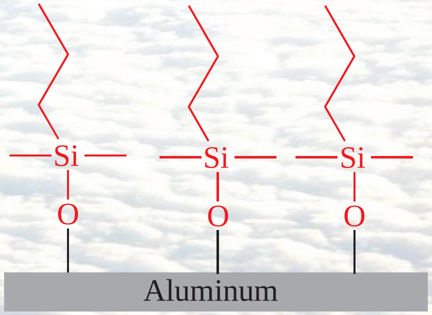
Adhesion Reduction Factor

$$\text{ARF} = \frac{\text{IASS of uncoated Al surface}}{\text{IASS of coated Al surface}}$$

An Adhesion Reduction Factor (ARF) > 1 implies ice did not adhere as well to the coating relative to the uncoated Al surface, whereas values < 1 indicate greater adhesion.

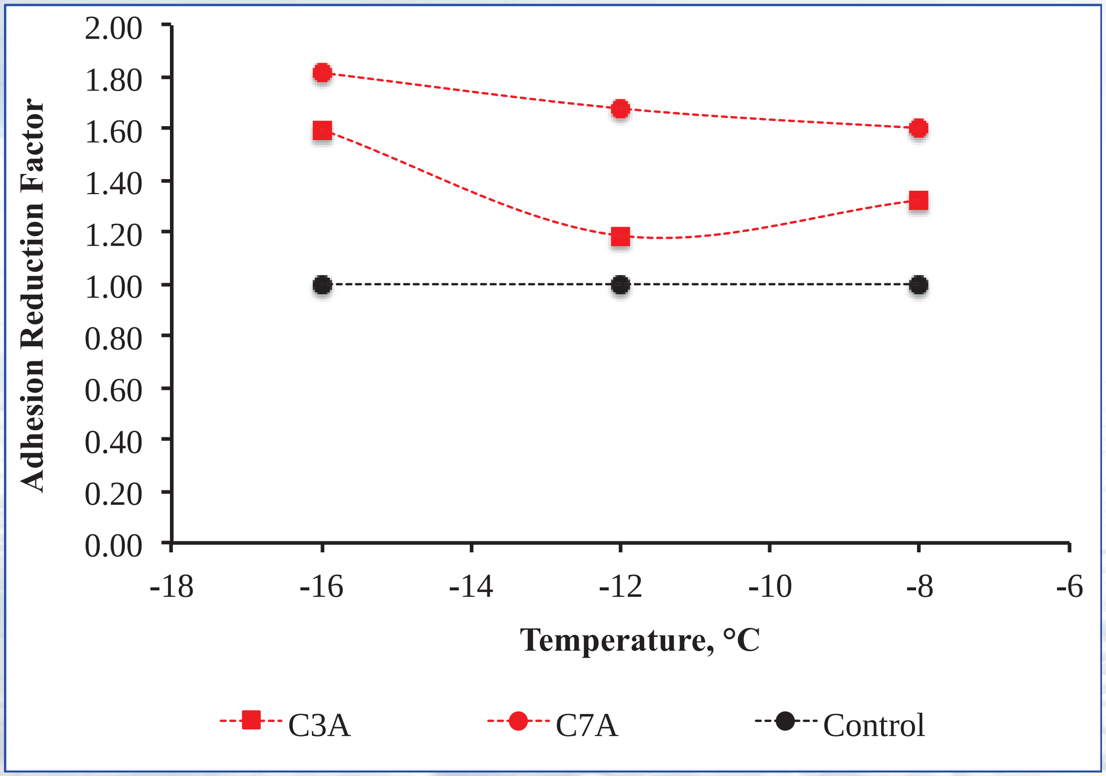
Substituted n-Alkyldimethylsilyl Coatings: non-Hydrogen Bonding

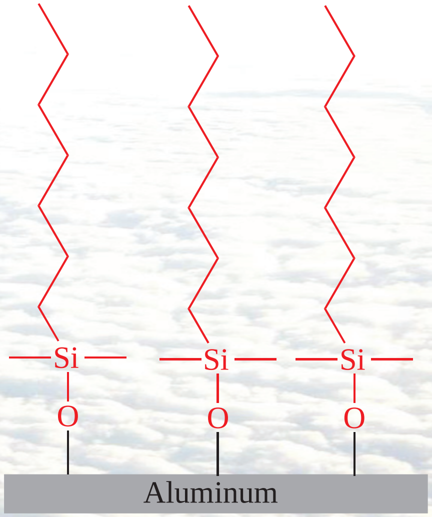




C3A

Substituted n-Alkyldimethylsilyl Coatings: non-Hydrogen Bonding

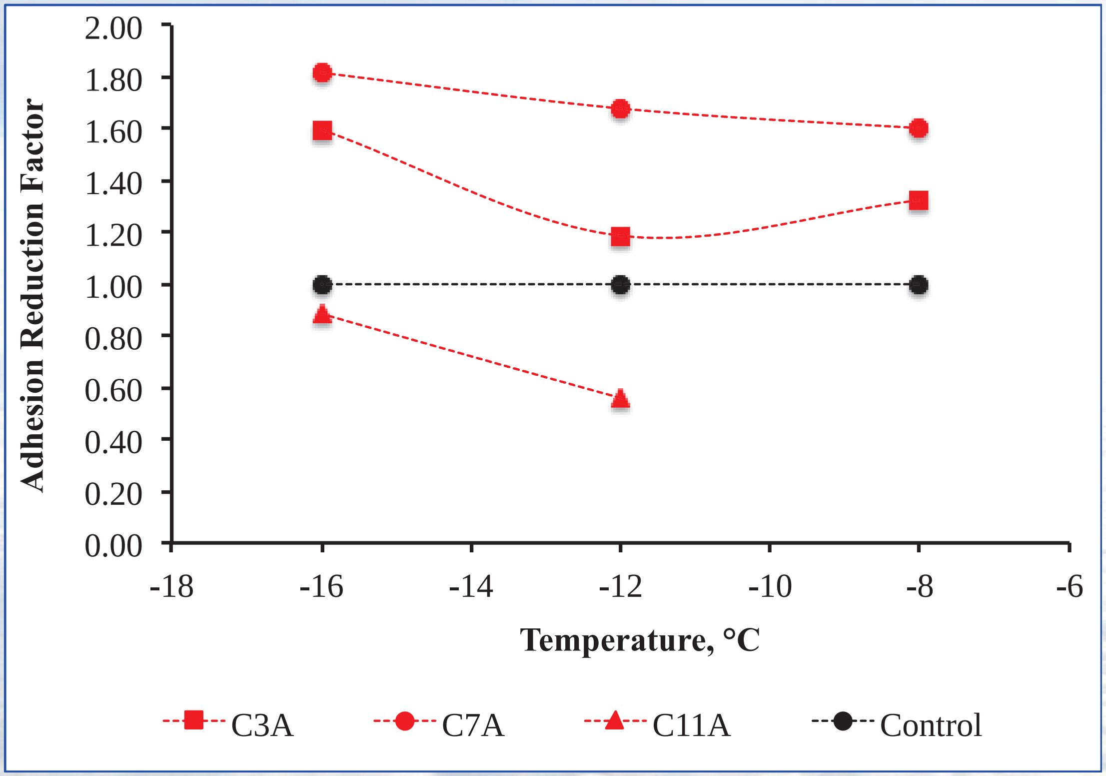


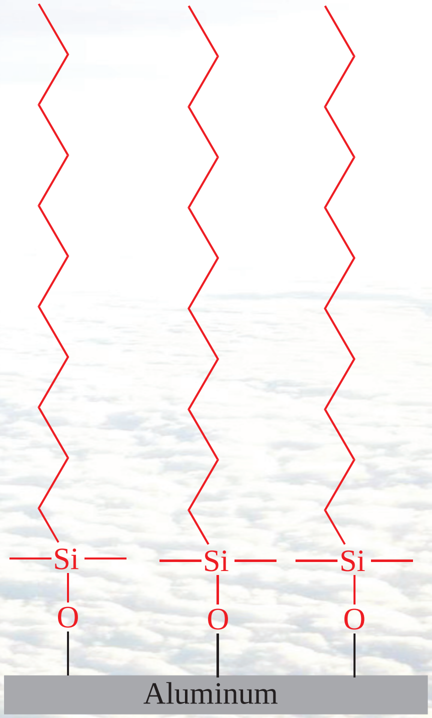


C7A

C3A + 4 (–CH_2-) groups = C7A

Substituted n-Alkyldimethylsilyl Coatings: non-Hydrogen Bonding

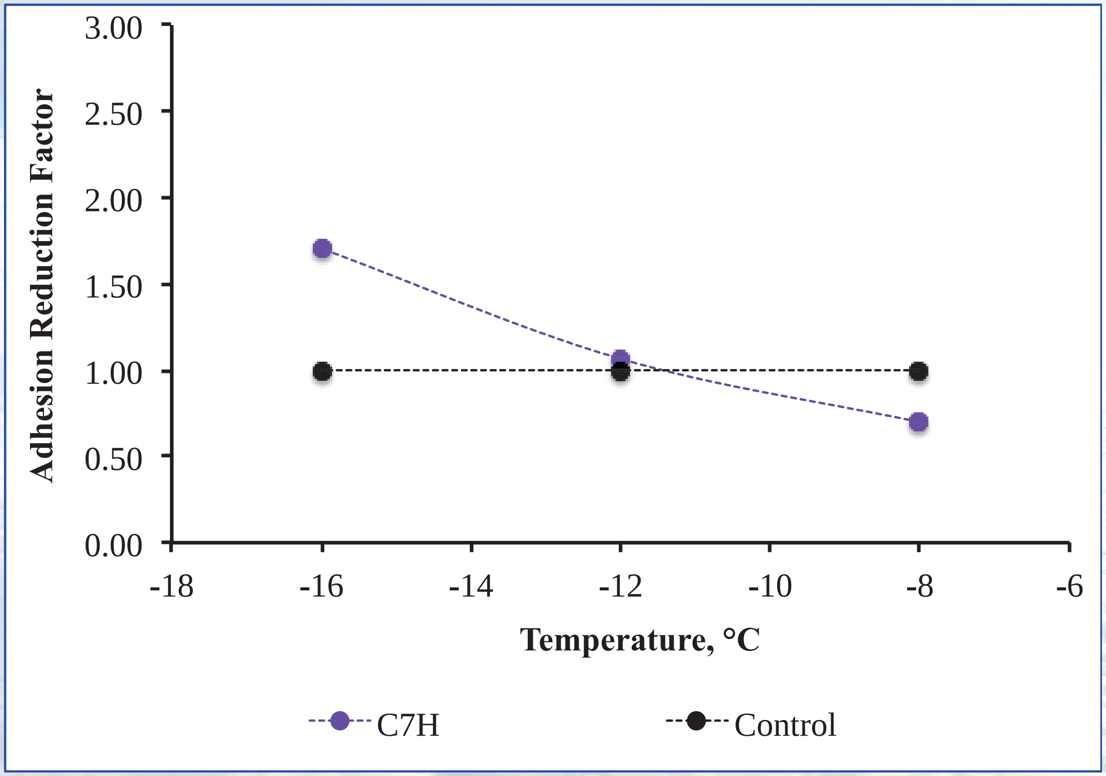


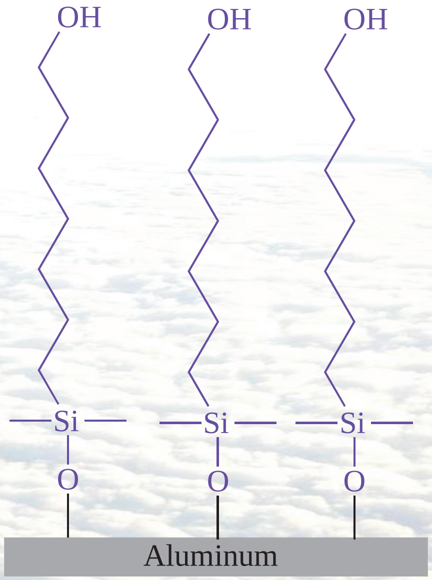


C11A

C7A + 4 (–CH_2-) groups = C11A

Substituted n-Alkyldimethylsilyl Coatings: Hydrogen Bonding (Donor/Acceptor)

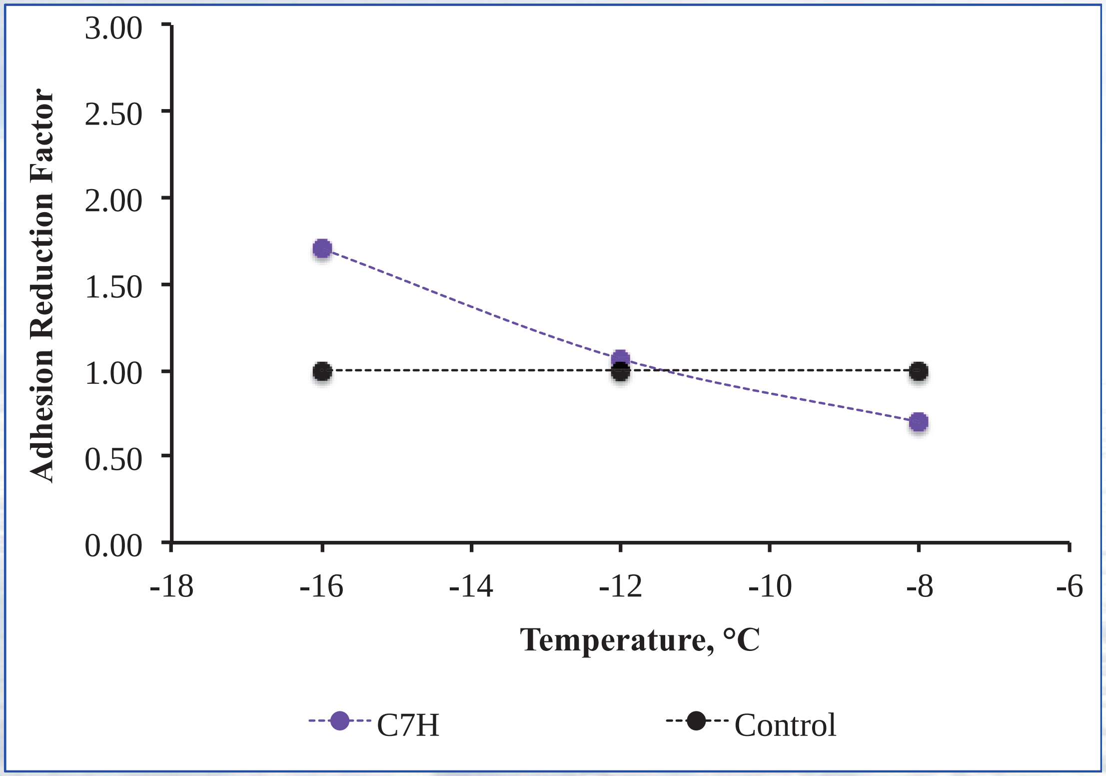


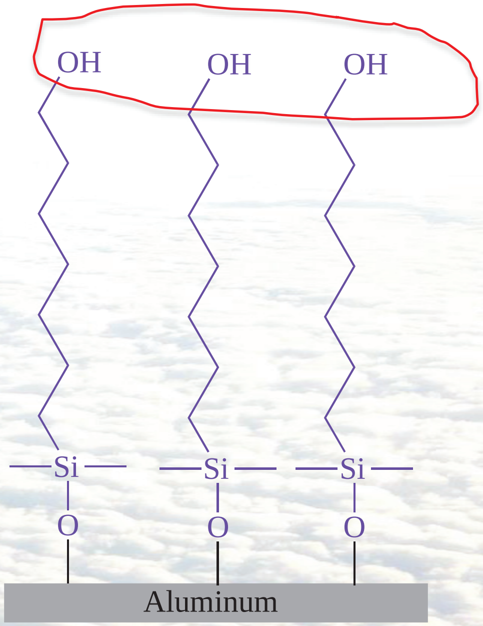


C7H

Change hydrogen atom on terminal methyl group (i.e., non-hydrogen bonding) to a hydroxyl group [i.e., hydrogen bonding (donor/acceptor)]

Substituted n-Alkyldimethylsilyl Coatings: Hydrogen Bonding (Donor/Acceptor)



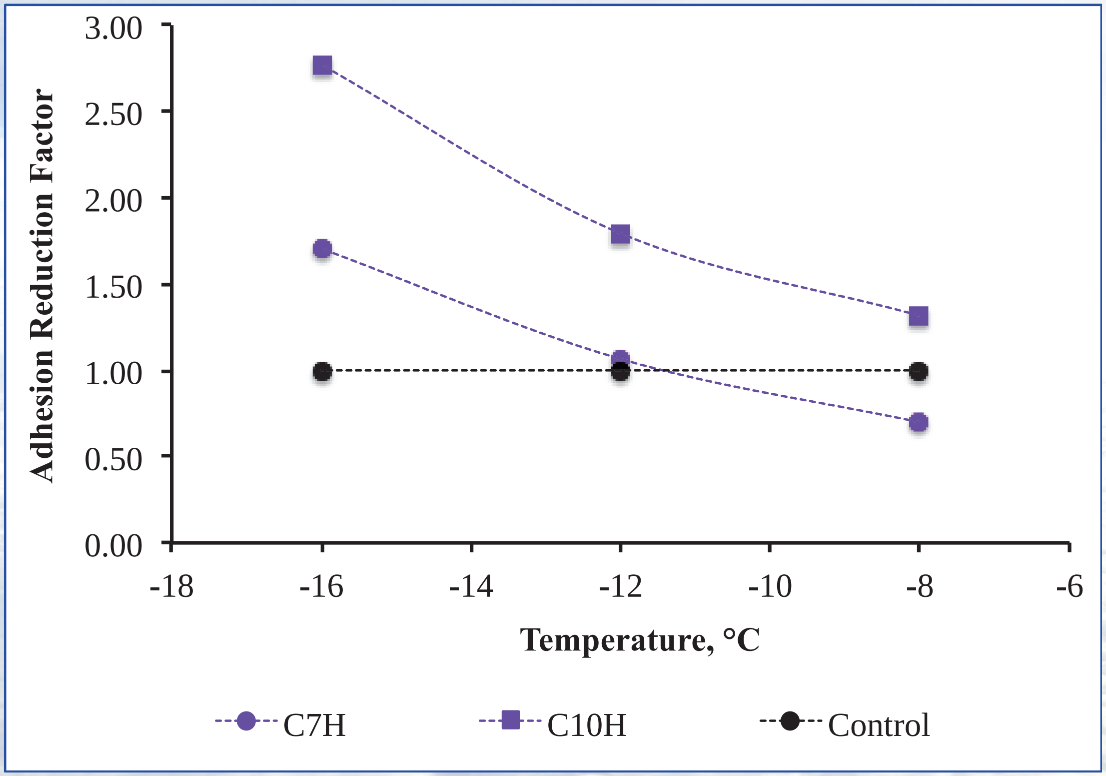


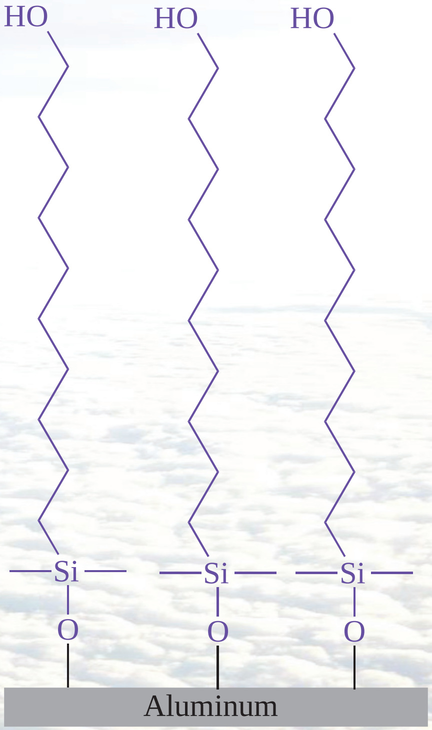
C7H

Change hydrogen atom on terminal methyl group (i.e., non-hydrogen bonding) to a hydroxyl group [i.e., hydrogen bonding (donor/acceptor)]

NASA Workshop on Low Ice Adhesion Materials, 10 August 2017

Substituted n-Alkyldimethylsilyl Coatings: Hydrogen Bonding (Donor/Acceptor)

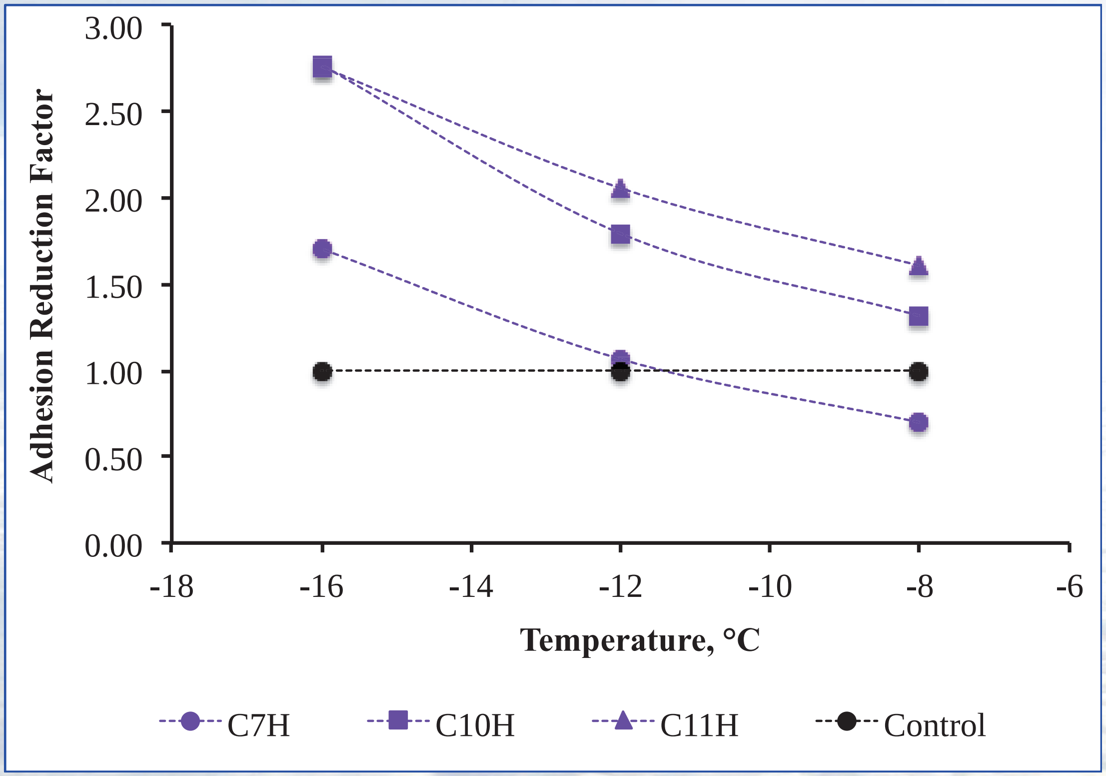




C10H

C7H + 3 ($–CH_2$-) groups = C10H

Substituted n-Alkyldimethylsilyl Coatings: Hydrogen Bonding (Donor/Acceptor)

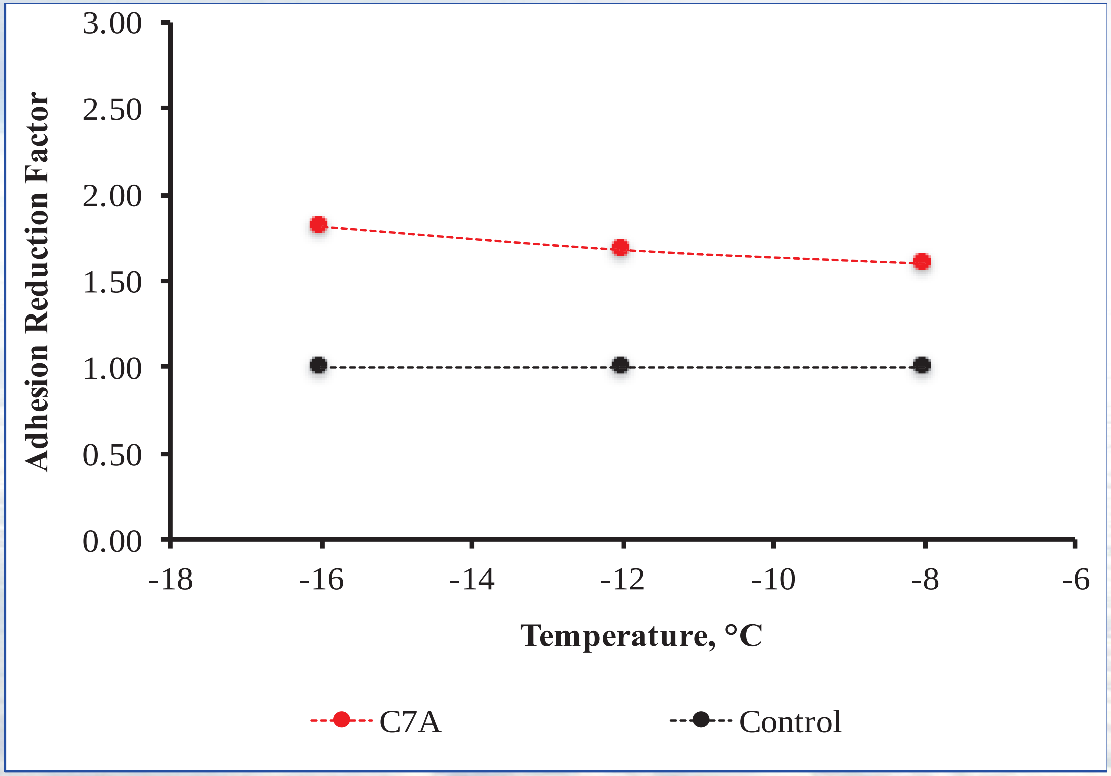


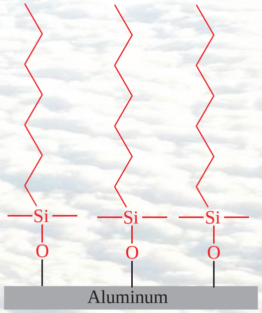
C11H

C10H + 1 ($-CH_2-$) group = C11H

NASA Workshop on Low Ice Adhesion Materials, 10 August 2017

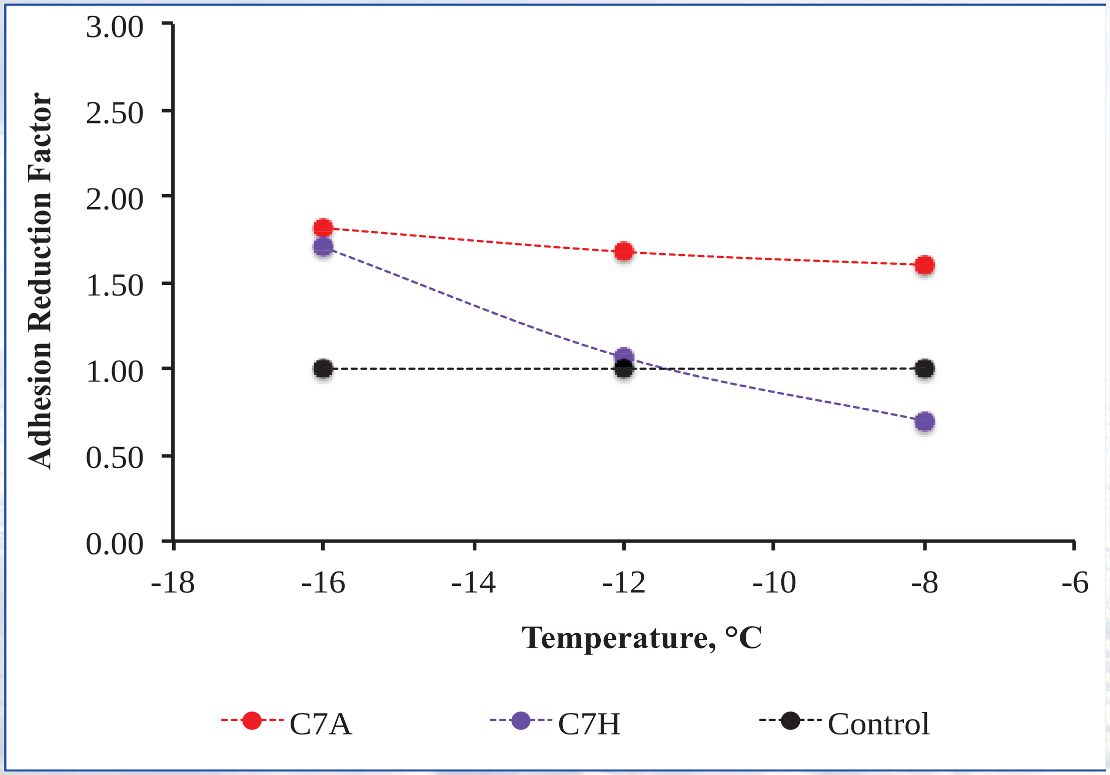
Comparison of non-Hydrogen Bonding & Hydrogen Bonding (Donor/Acceptor) Coatings

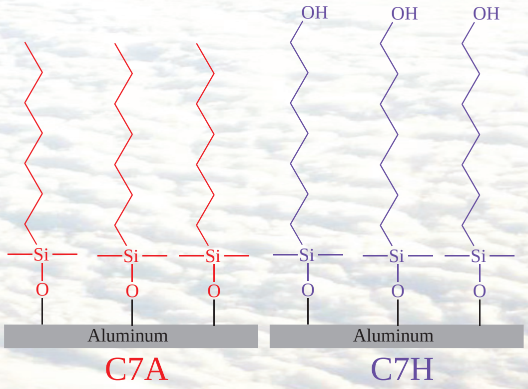




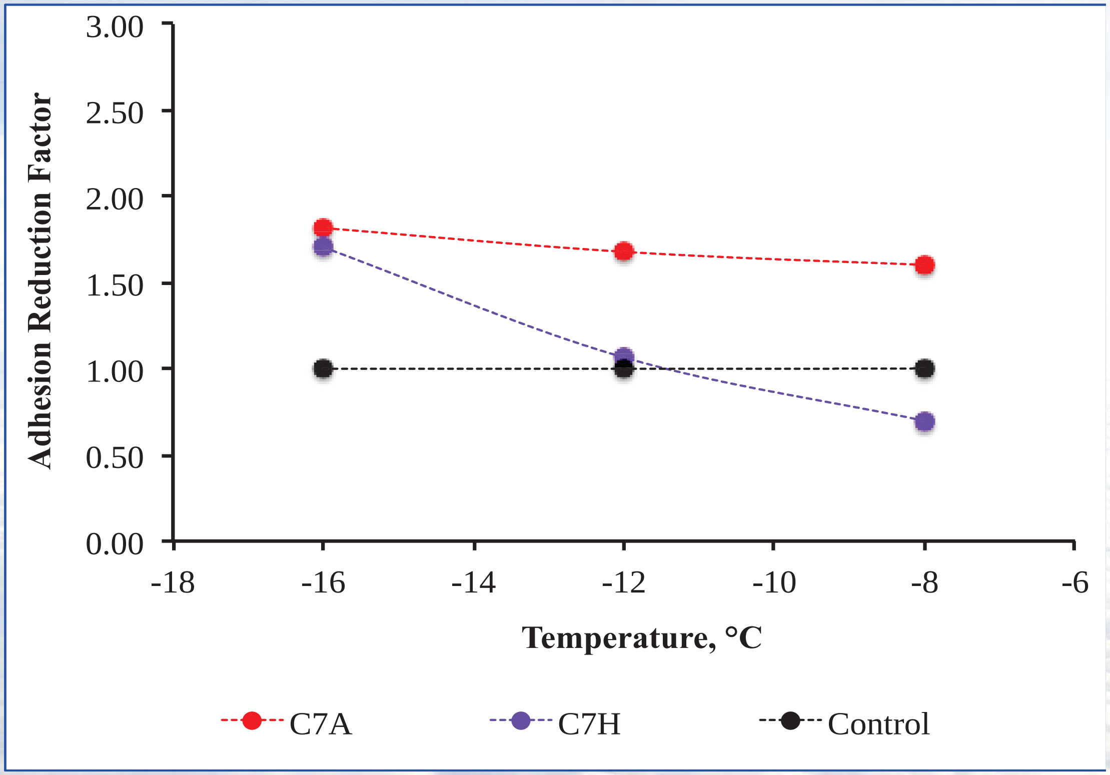
C7A

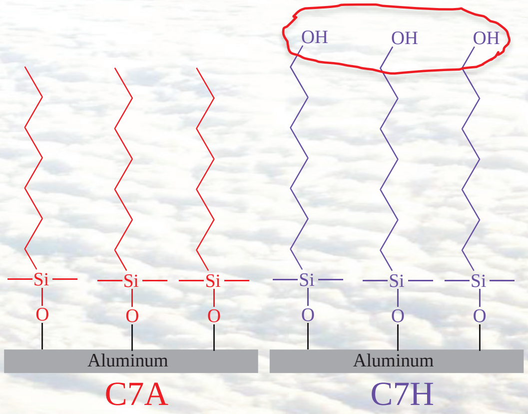
Comparison of non-Hydrogen Bonding & Hydrogen Bonding (Donor/Acceptor) Coatings



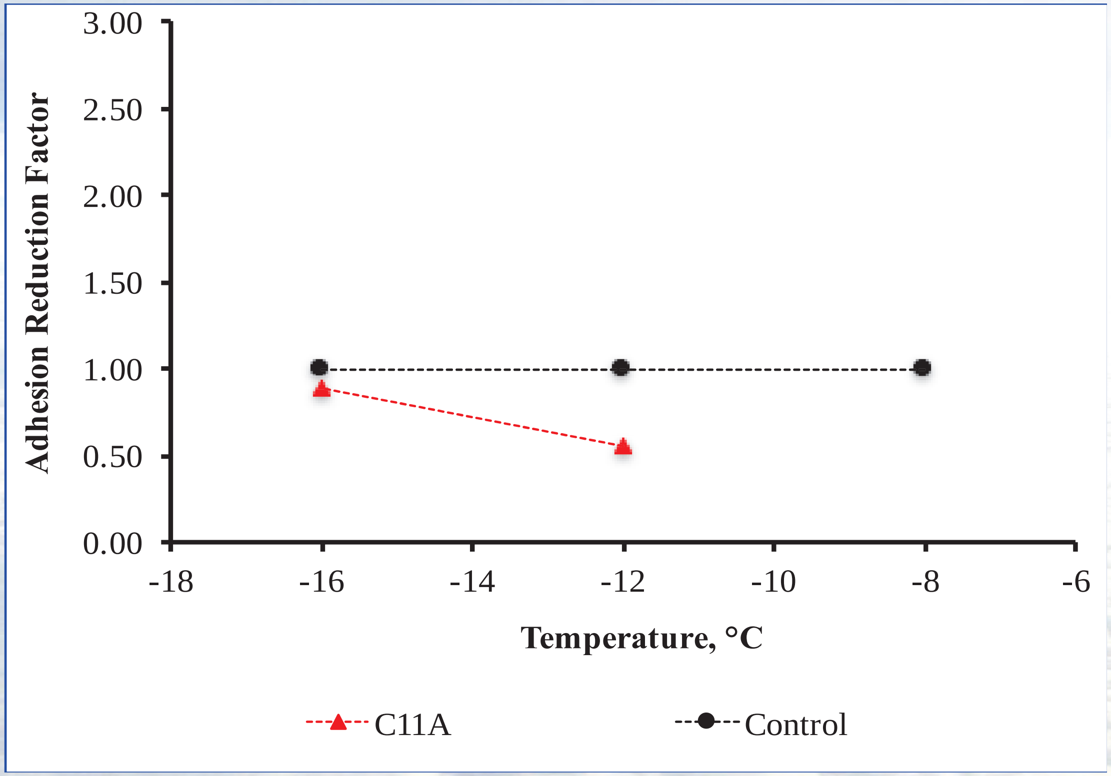


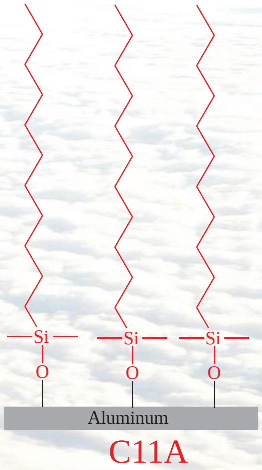
Comparison of non-Hydrogen Bonding & Hydrogen Bonding (Donor/Acceptor) Coatings





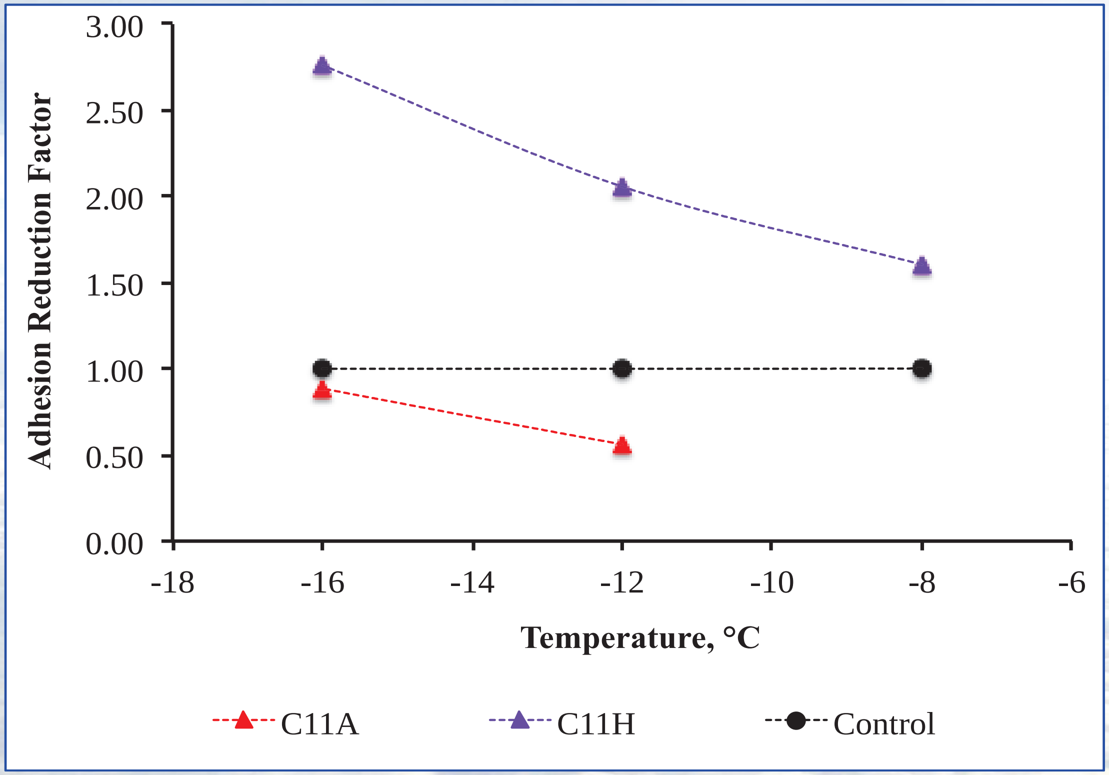
Comparison of non-Hydrogen Bonding & Hydrogen Bonding (Donor/Acceptor) Coatings

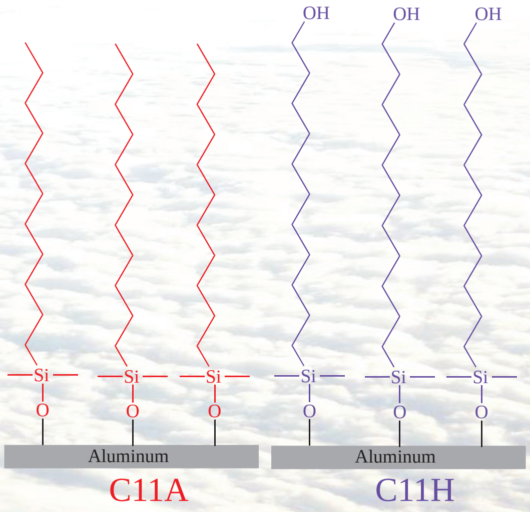




C7 + 4 (–CH_2-) groups = C11

Comparison of non-Hydrogen Bonding & Hydrogen Bonding (Donor/Acceptor) Coatings

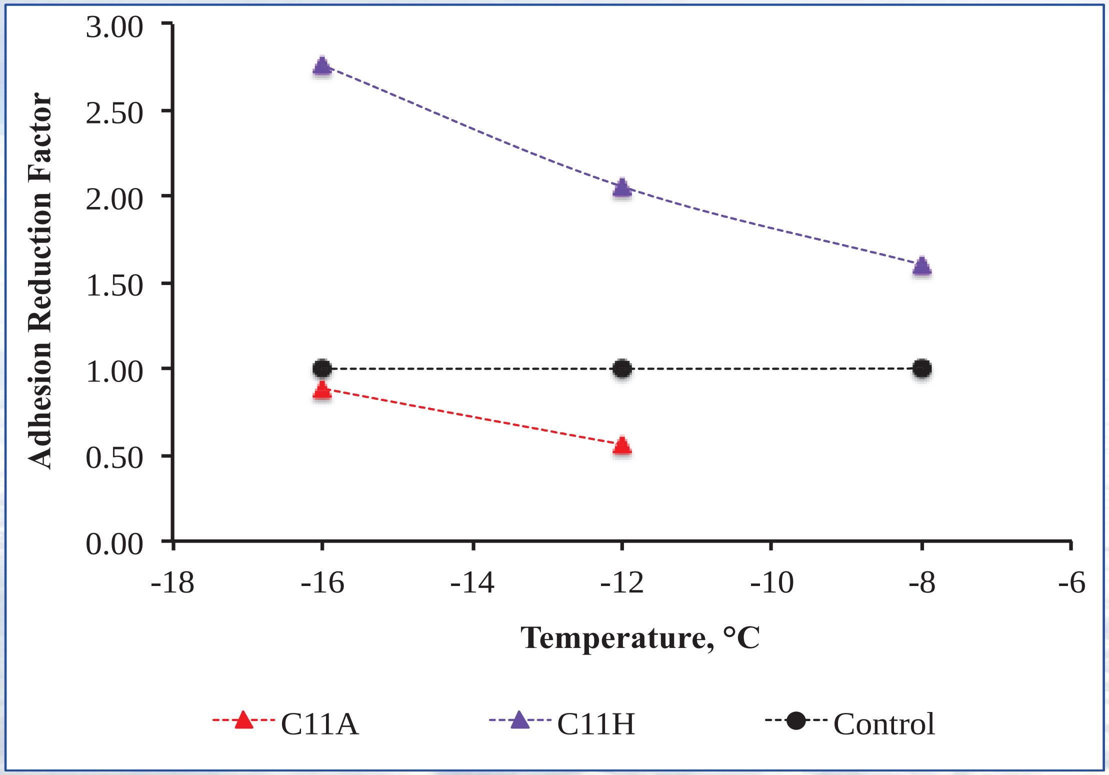


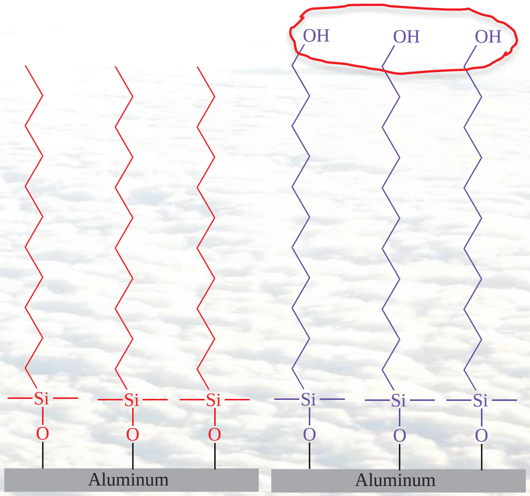


C7 + 4 (–CH_2-) groups = C11

NASA Workshop on Low Ice Adhesion Materials, 10 August 2017

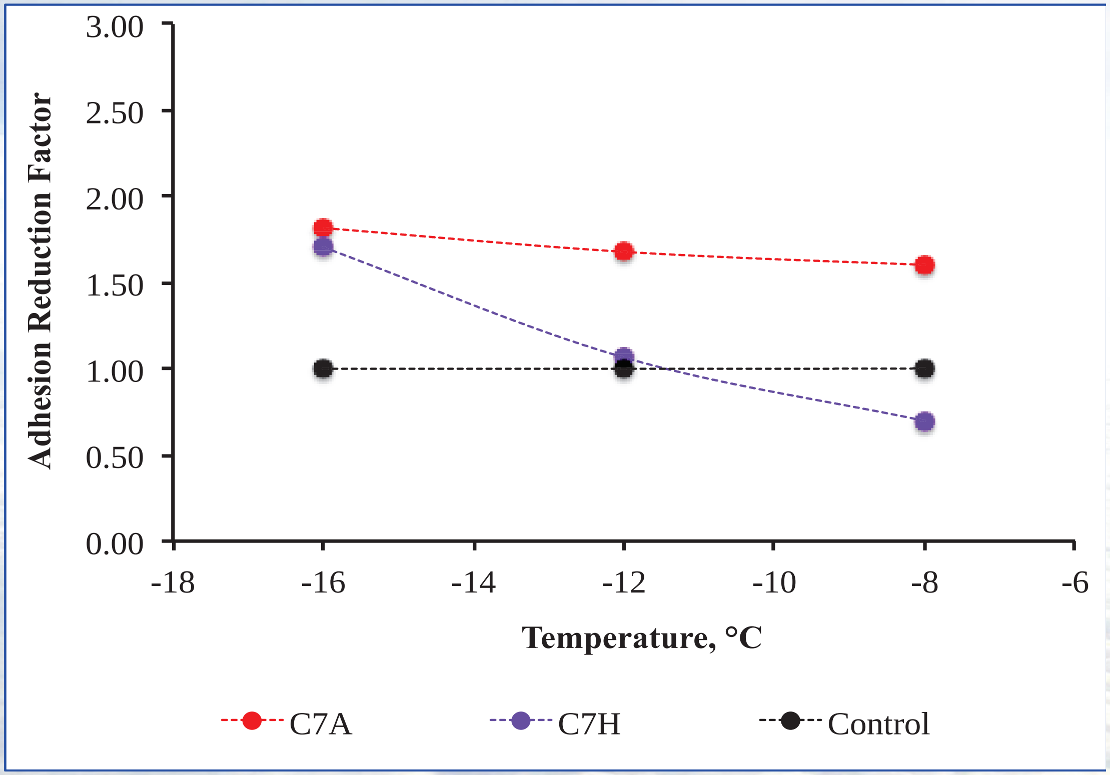
Comparison of non-Hydrogen Bonding & Hydrogen Bonding (Donor/Acceptor) Coatings

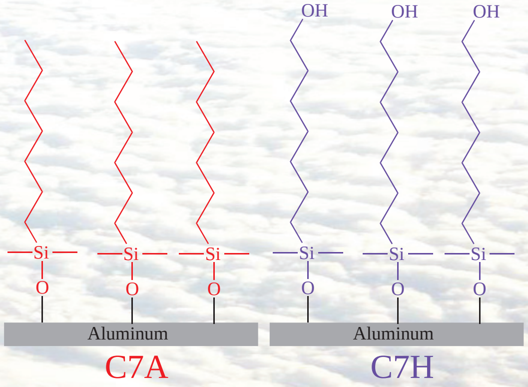




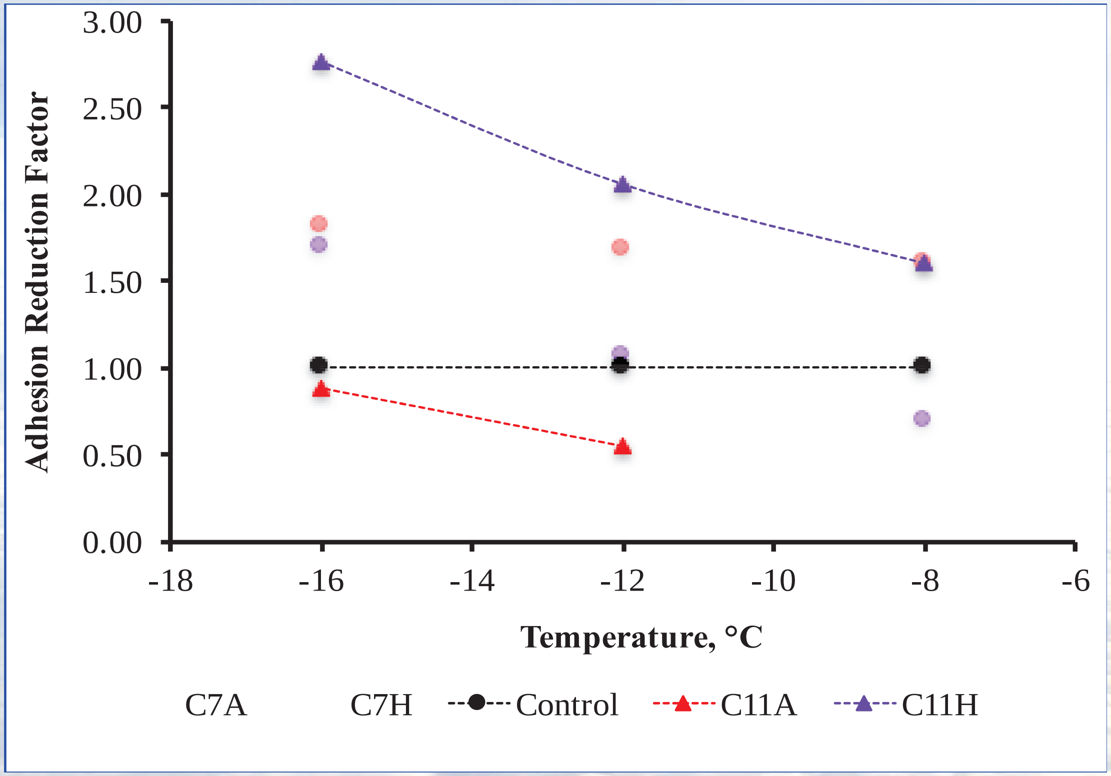
C11A C11H

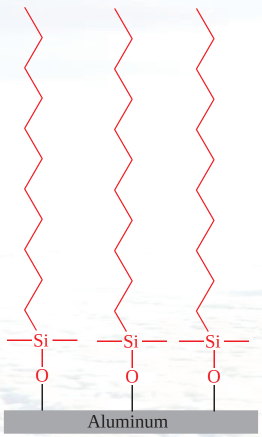
Comparison of non-Hydrogen Bonding & Hydrogen Bonding (Donor/Acceptor) Coatings





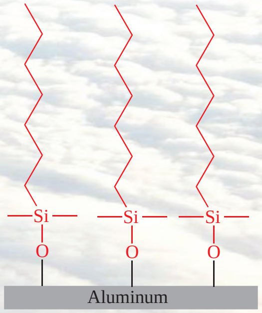
Comparison of non-Hydrogen Bonding & Hydrogen Bonding (Donor/Acceptor) Coatings



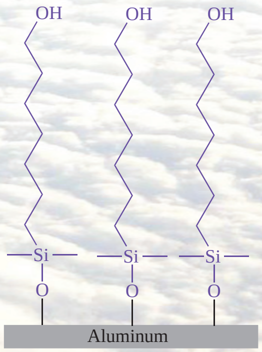


C11A

C11H

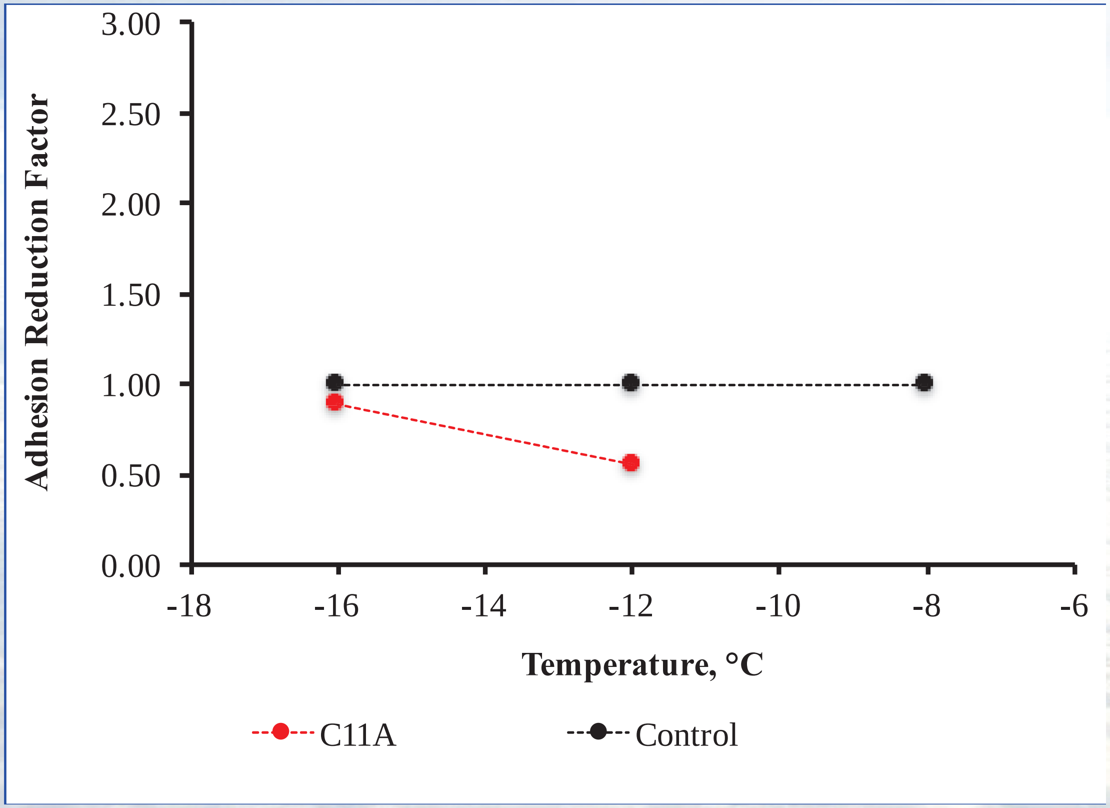


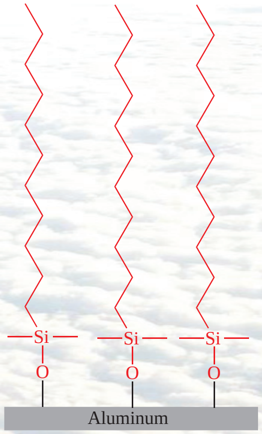
C7A



C7H

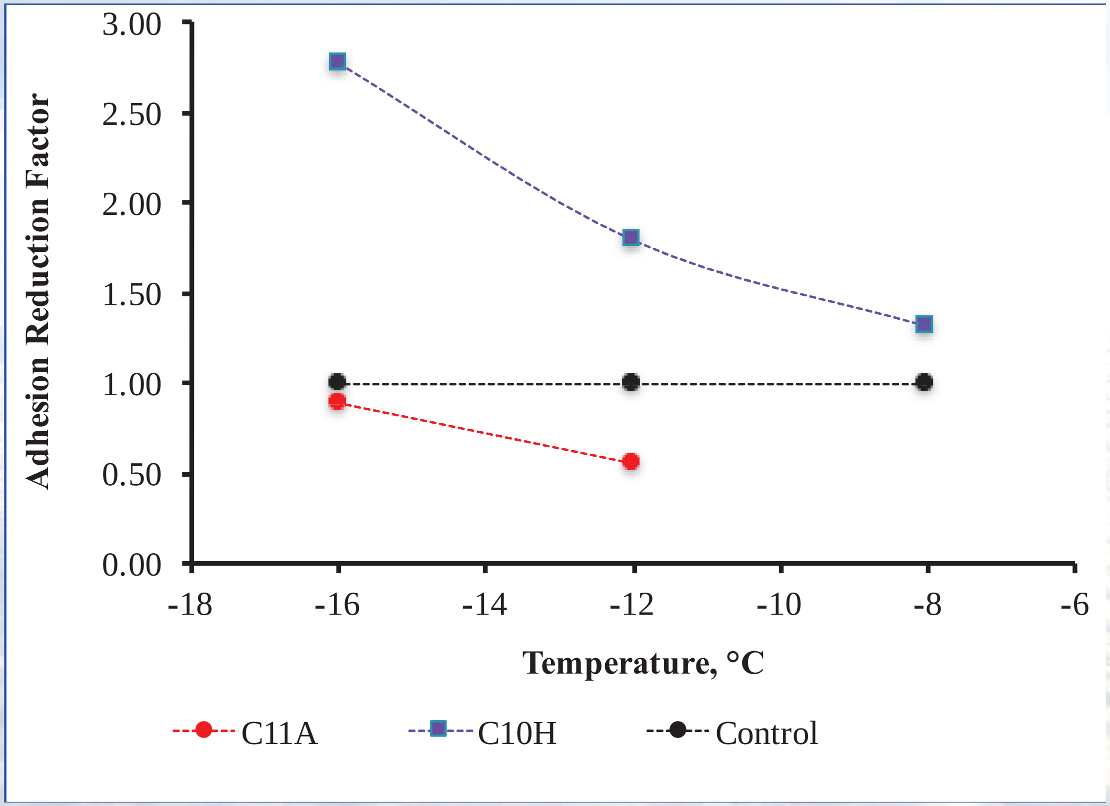
Comparison of non-Hydrogen Bonding, Hydrogen Bonding (Donor/Acceptor), & Hydrogen Bonding (Acceptor) Coatings

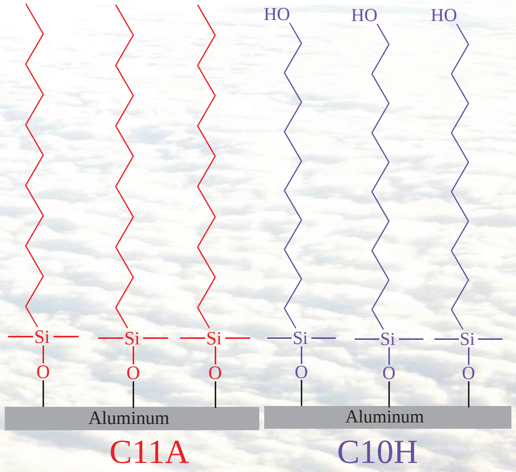




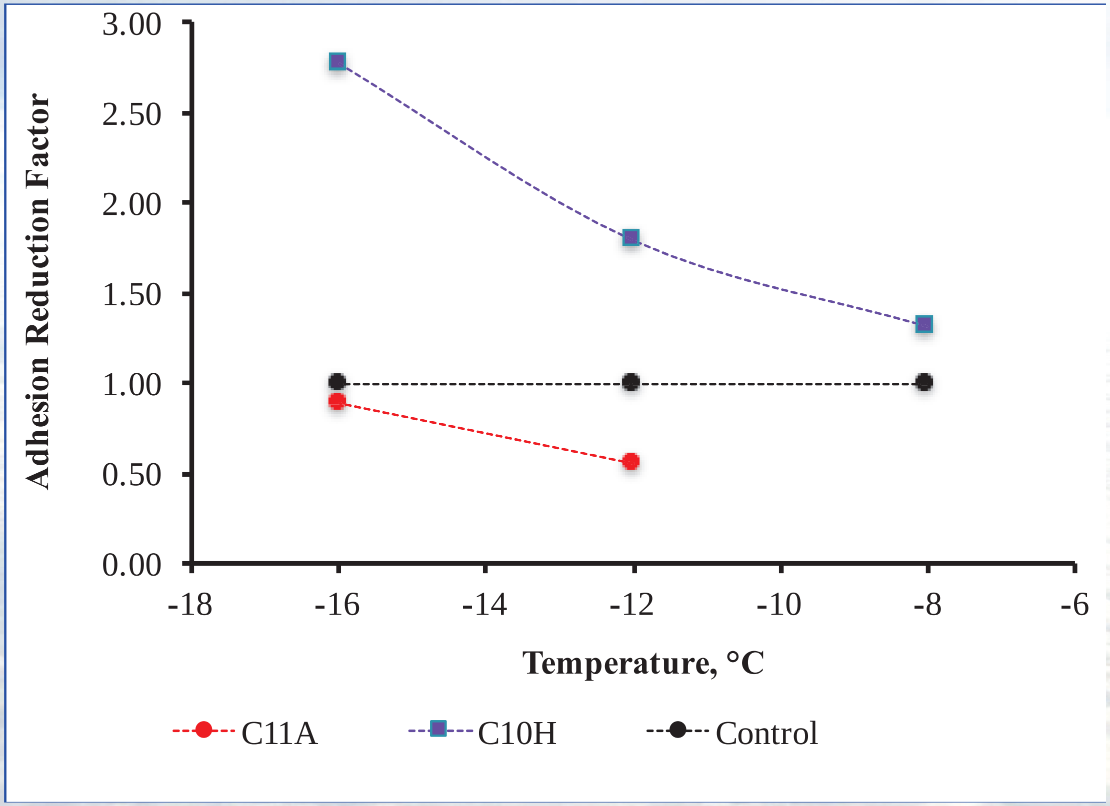
C11A

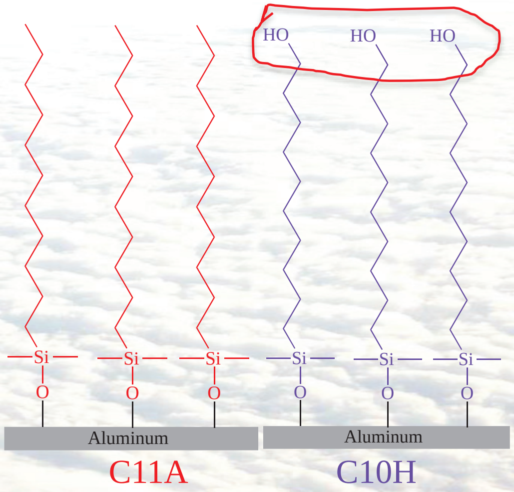
Comparison of non-Hydrogen Bonding, Hydrogen Bonding (Donor/Acceptor), & Hydrogen Bonding (Acceptor) Coatings



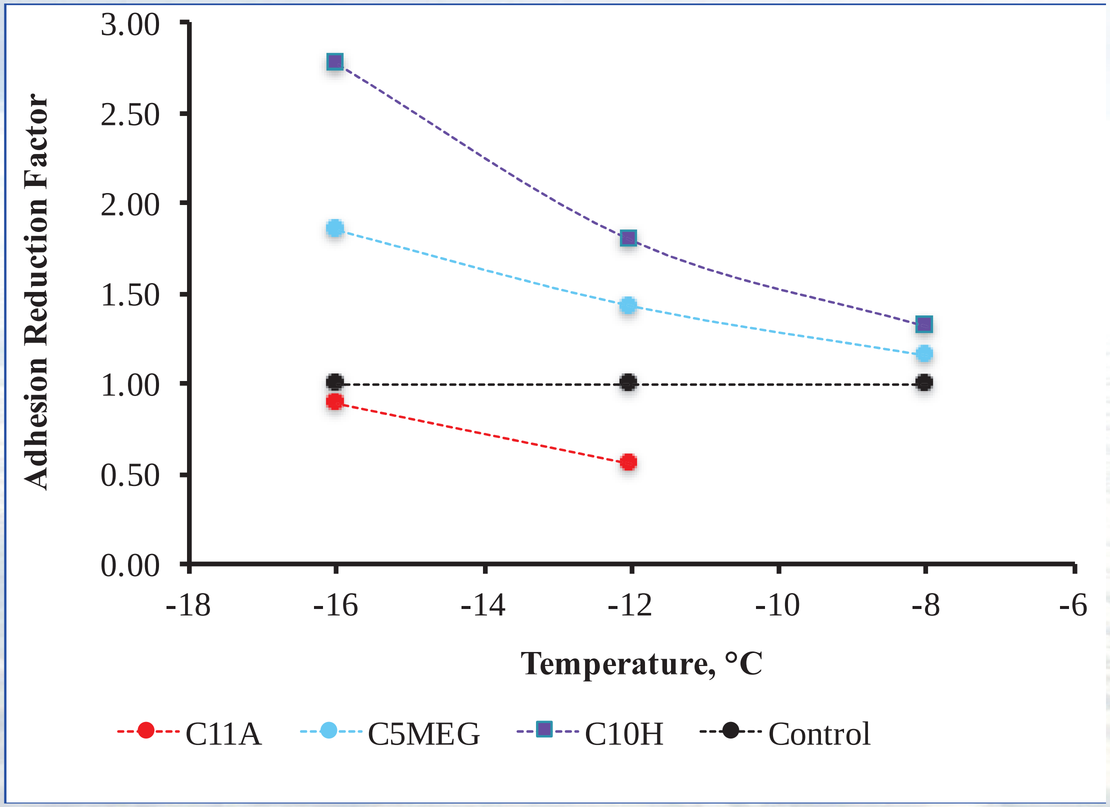


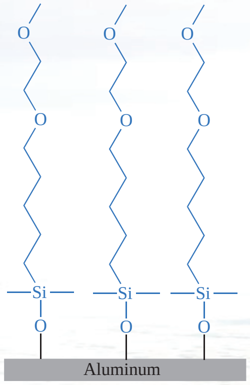
Comparison of non-Hydrogen Bonding, Hydrogen Bonding (Donor/Acceptor), & Hydrogen Bonding (Acceptor) Coatings



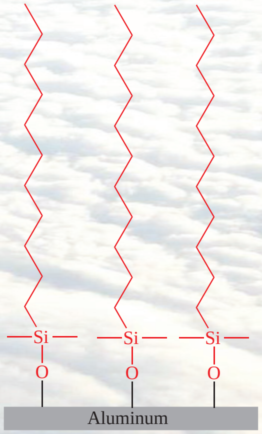


Comparison of non-Hydrogen Bonding, Hydrogen Bonding (Donor/Acceptor), & Hydrogen Bonding (Acceptor) Coatings





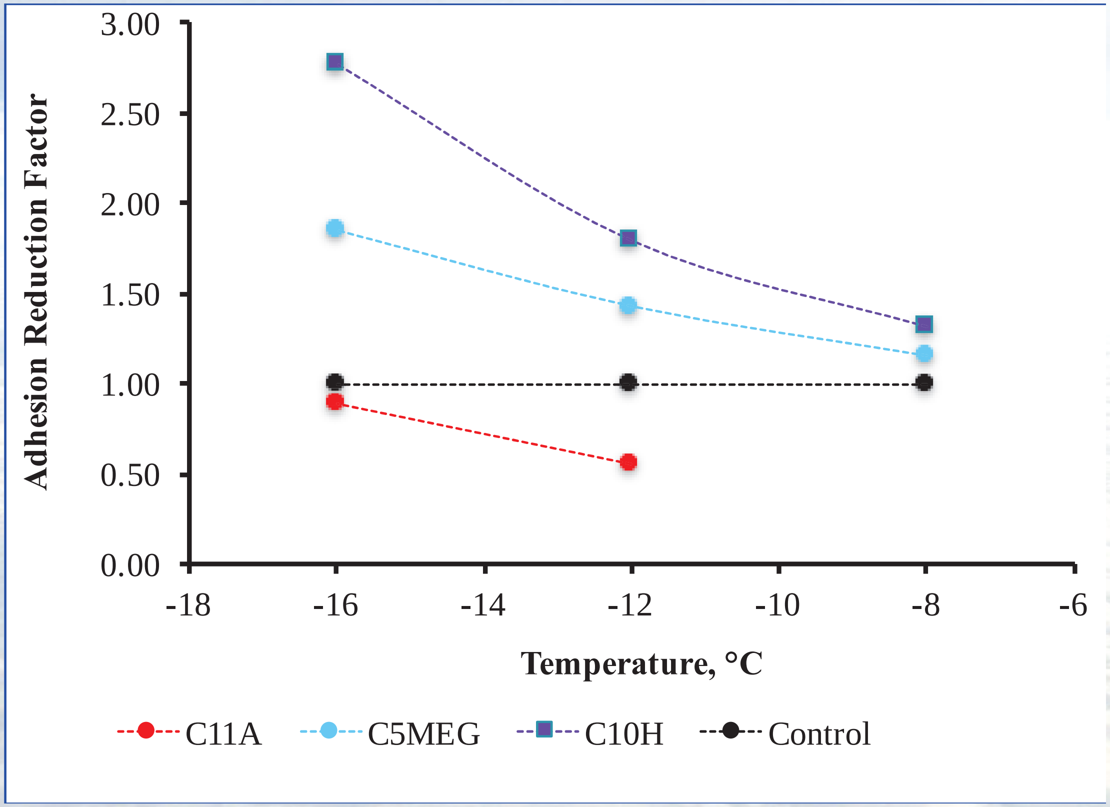
C5MEG

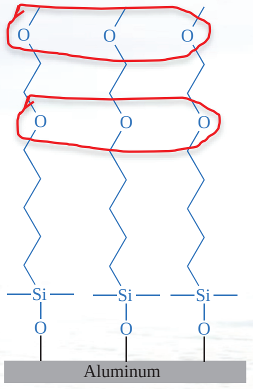


C11A

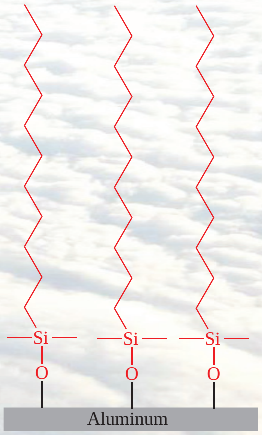
C10H

Comparison of non-Hydrogen Bonding, Hydrogen Bonding (Donor/Acceptor), & Hydrogen Bonding (Acceptor) Coatings





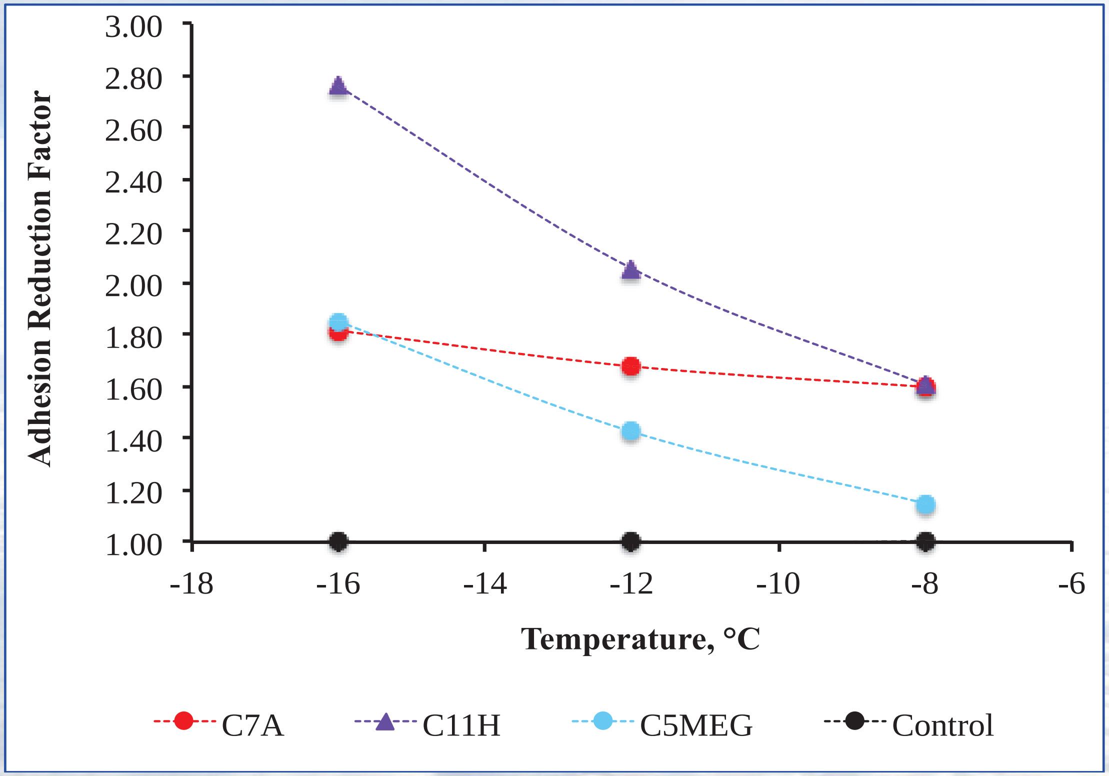
C5MEG



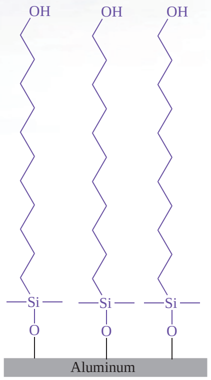
C11A

C10H

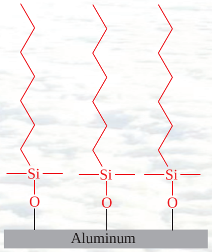
Best Performing Coating from Each Class



Hydrogen Bonding
(Donor/Acceptor)



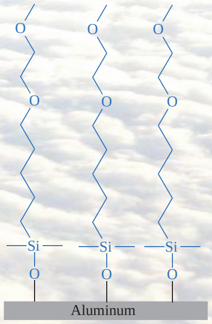
C11H



C7A

non-Hydrogen
Bonding

Hydrogen Bonding
(Acceptor)



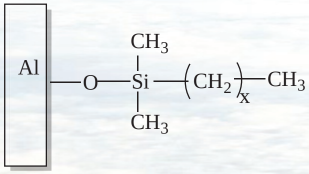
C5MEG

Neat Coating Summary

- General
  - Coating performance dependent upon functional group, chain length, and temperature
  - Performance related to surface energy change (i.e., non-polar and polar) during phase change of water to ice
  - Trend (based on the best performer of each series)
    - HB (D/Ac) > non-HB > HB (Ac)

Neat Coating Summary

- General
  - Coating performance dependent upon functional group, chain length, and temperature
  - Performance related to surface energy change (i.e., non-polar and polar) during phase change of water to ice
  - Trend (based on the best performer of each series)
    - HB (D/Ac) > non-HB > HB (Ac)

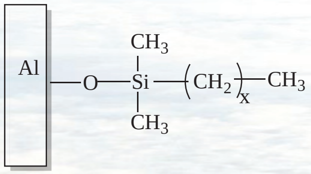


$$\text{Al}-O-\overset{CH_3}{\underset{CH_3}{Si}}-(CH_2)_x-CH_3$$

- non-Hydrogen Bonding
  - Performance dependent upon alkyl chain length
  - Moderate alkyl chain length (C7A) exhibited the best performance

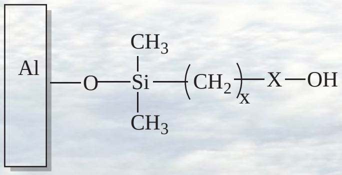
Neat Coating Summary

- General
  - Coating performance dependent upon functional group, chain length, and temperature
  - Performance related to surface energy change (i.e., non-polar and polar) during phase change of water to ice
  - Trend (based on the best performer of each series)
    - HB (D/Ac) > non-HB > HB (Ac)



$\text{Al}-\text{O}-\underset{\text{CH}_3}{\overset{\text{CH}_3}{\text{Si}}}-(\text{CH}_2)_x-\text{CH}_3$

- non-Hydrogen Bonding
  - Performance dependent upon alkyl chain length
  - Moderate alkyl chain length (C7A) exhibited the best performance

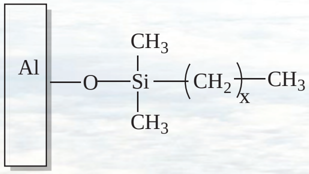


$\text{Al}-\text{O}-\underset{\text{CH}_3}{\overset{\text{CH}_3}{\text{Si}}}-(\text{CH}_2)_x-\text{X}-\text{OH}$

- Hydrogen Bonding (Donor/Acceptor)
  - Performance improved as temperature decreased
  - Long alkyl chain (C10H, C11H) exhibited best performance as opposed to non-hydrogen bonding analogs

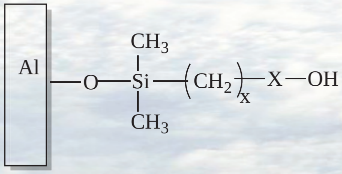
Neat Coating Summary

- General
  - Coating performance dependent upon functional group, chain length, and temperature
  - Performance related to surface energy change (i.e., non-polar and polar) during phase change of water to ice
  - Trend (based on the best performer of each series)
    - HB (D/Ac) > non-HB > HB (Ac)



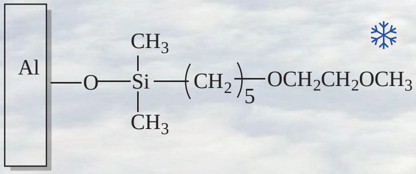
$\text{Al}-O-\overset{CH_3}{\underset{CH_3}{Si}}-(CH_2)_x-CH_3$

- non-Hydrogen Bonding
  - Performance dependent upon alkyl chain length
  - Moderate alkyl chain length (C7A) exhibited the best performance



$\text{Al}-O-\overset{CH_3}{\underset{CH_3}{Si}}-(CH_2)_x-X-OH$

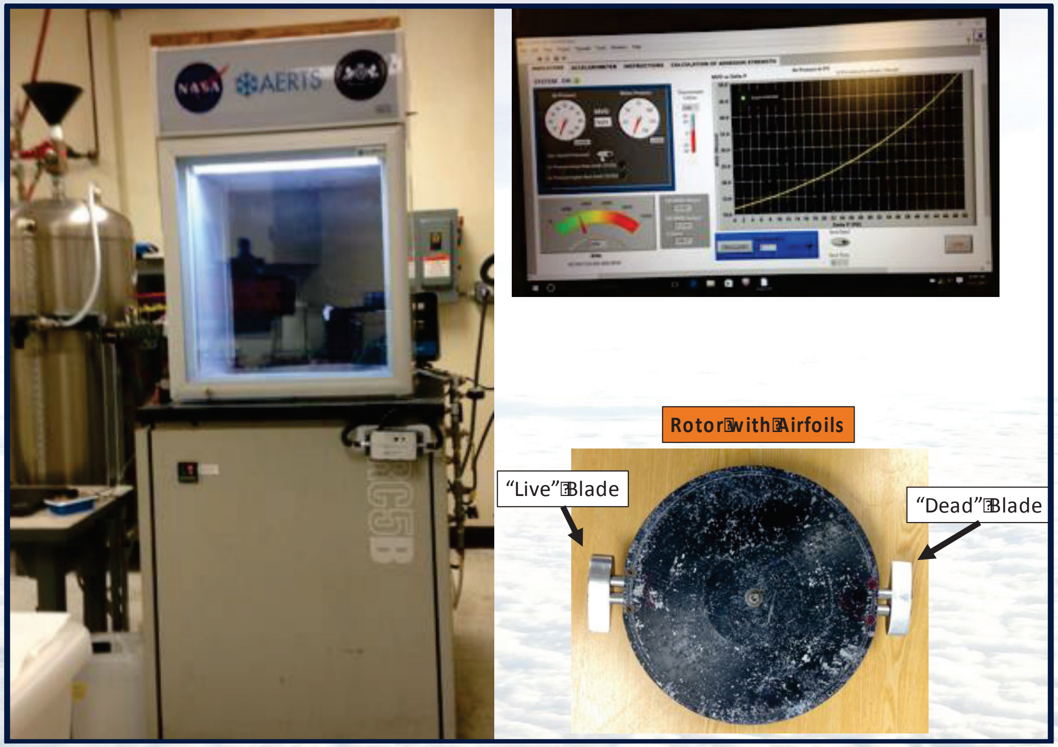
- Hydrogen Bonding (Donor/Acceptor)
  - Performance improved as temperature decreased
  - Long alkyl chain (C10H, C11H) exhibited best performance as opposed to non-hydrogen bonding analogs



$\text{Al}-O-\overset{CH_3}{\underset{CH_3}{Si}}-(CH_2)_5-OCH_2CH_2OCH_3$

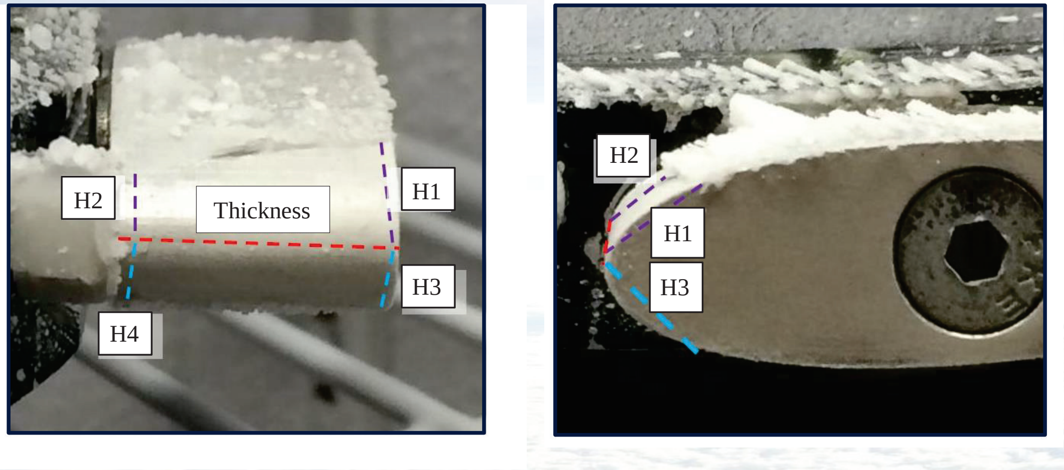
- Hydrogen Bonding (Acceptor)
  - Inclusion in aliphatic chain improved performance relative to non-hydrogen bonding composition of similar length (C11A)
  - Performance with respect to hydrogen bonding (donor/acceptor) composition (C10H) decreased with respect to decreasing temperature

Lab-Scale Evaluation of Icephobic Surfaces



- Screening of experimental surfaces is conducted on a lab-scale version of Adverse Environment Rotor Test Stand (AERTS) called AERTS Jr
- Designed and fabricated by Dr. Jose Palacios (The Pennsylvania State University, PSU)
- Equipped with one NASA MOD 2 Nozzle
- Promising surfaces are tested in AERTS at PSU

Ice Adhesion Shear Strength



Area = [Thickness x (H2 + H4)] + [0.5 x Thickness x (H1 – H2)] + [0.5 x Thickness x (H3 – H4)]

Ice Adhesion Shear Strength (IASS) = F_c/Area

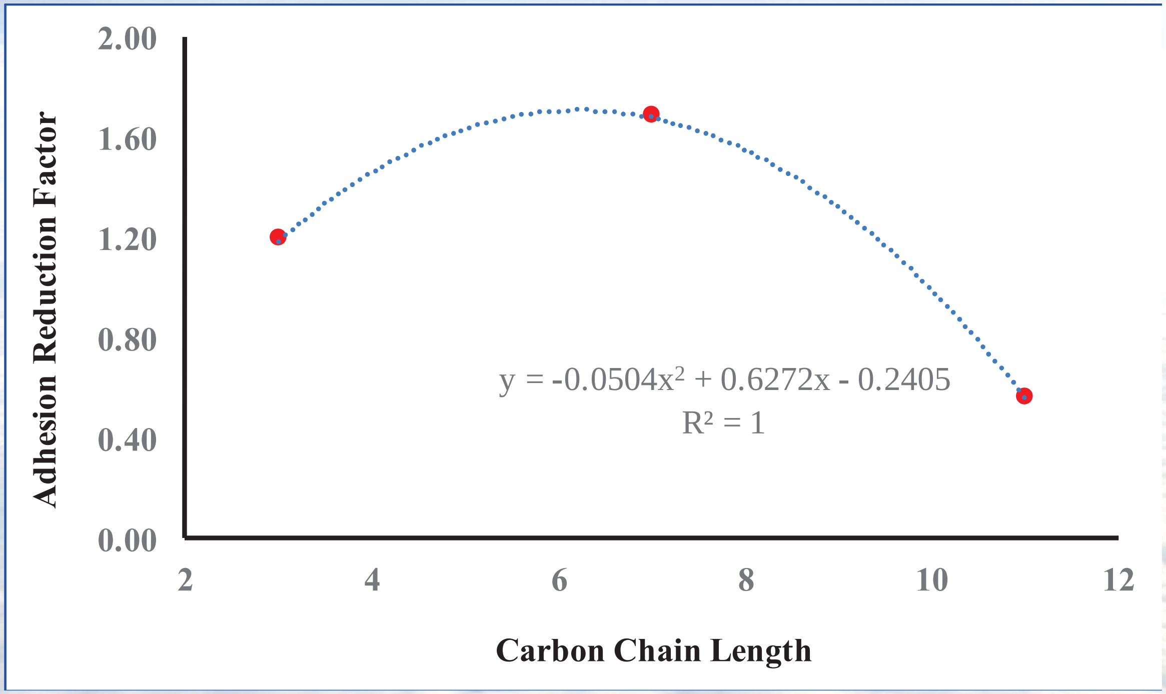
$$F_c = m_{ice} r \omega^2 = \frac{m_{ice} v^2}{r}$$

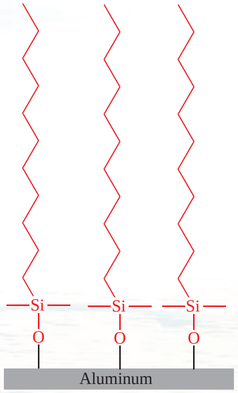
v = rω = r x rpm x 2π/60 s

$$\text{ARF} = \frac{\text{IASS of uncoated surface}}{\text{IASS of coated surface}}$$

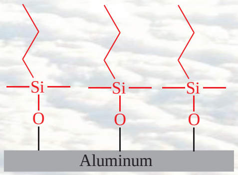
Adhesion Reduction Factor (ARF)

Non-Hydrogen Bonding ARF at -12°C

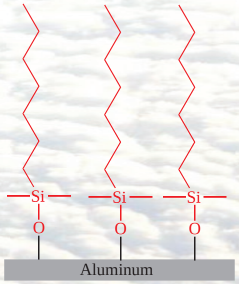




C11A



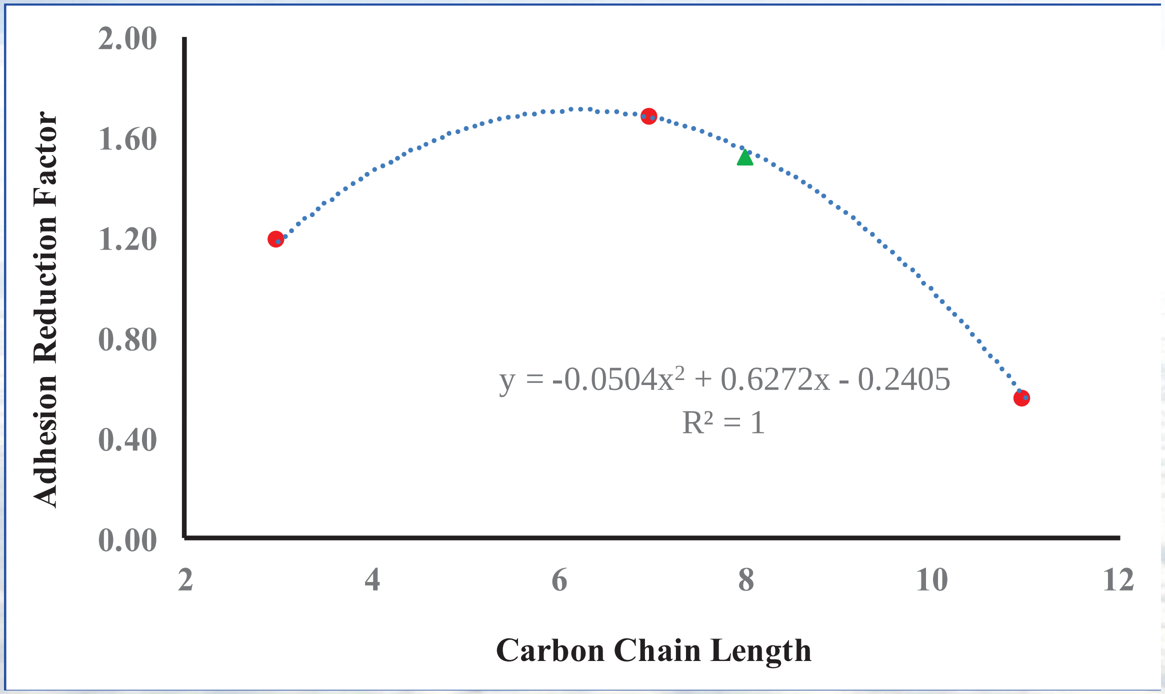
C3A

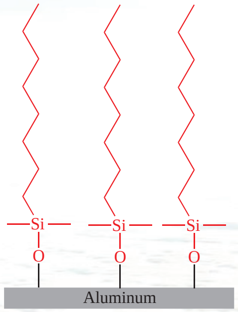


C7A

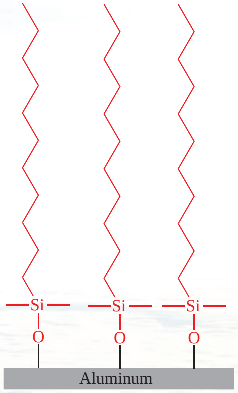
NASA Workshop on Low Ice Adhesion Materials, 10 August 2017

Non-Hydrogen Bonding ARF at -12°C

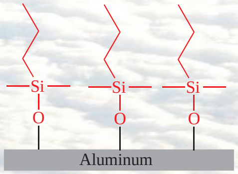




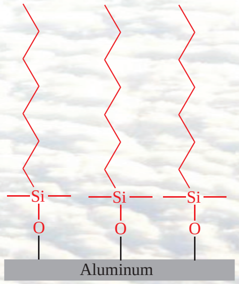
C8A



C11A

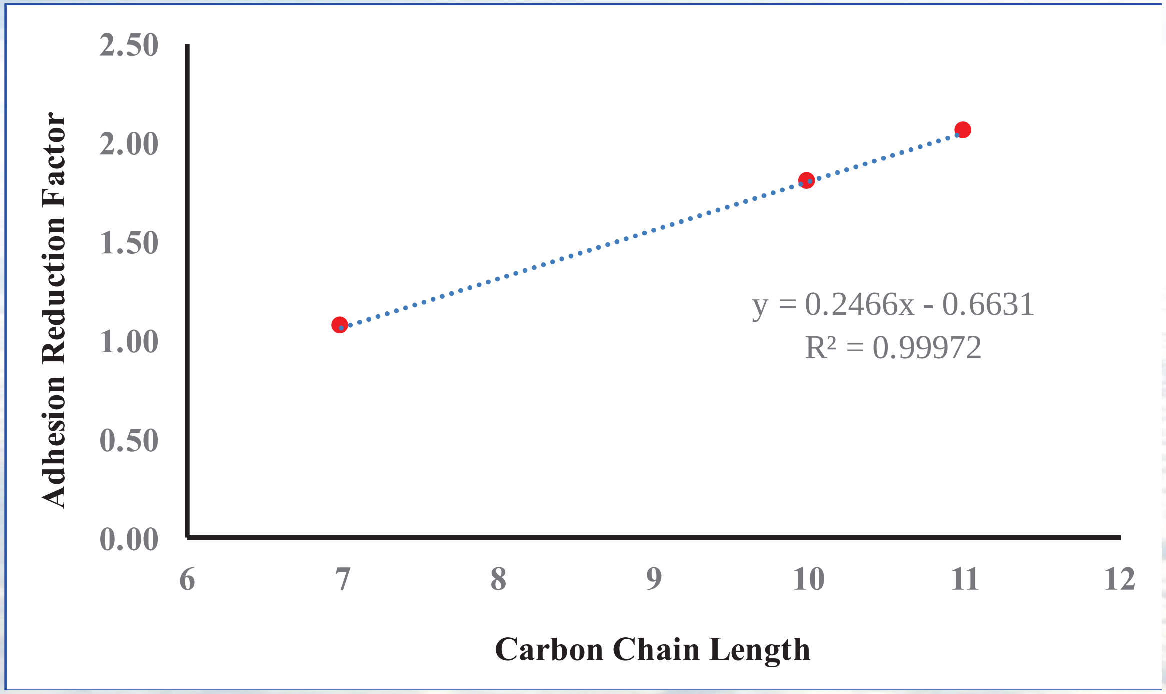


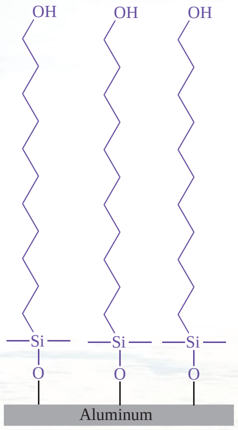
C3A



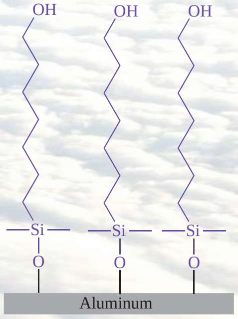
C7A

Hydrogen Bonding (Donor/Acceptor) ARF at -12°C

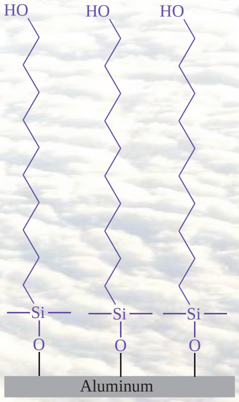




C11H

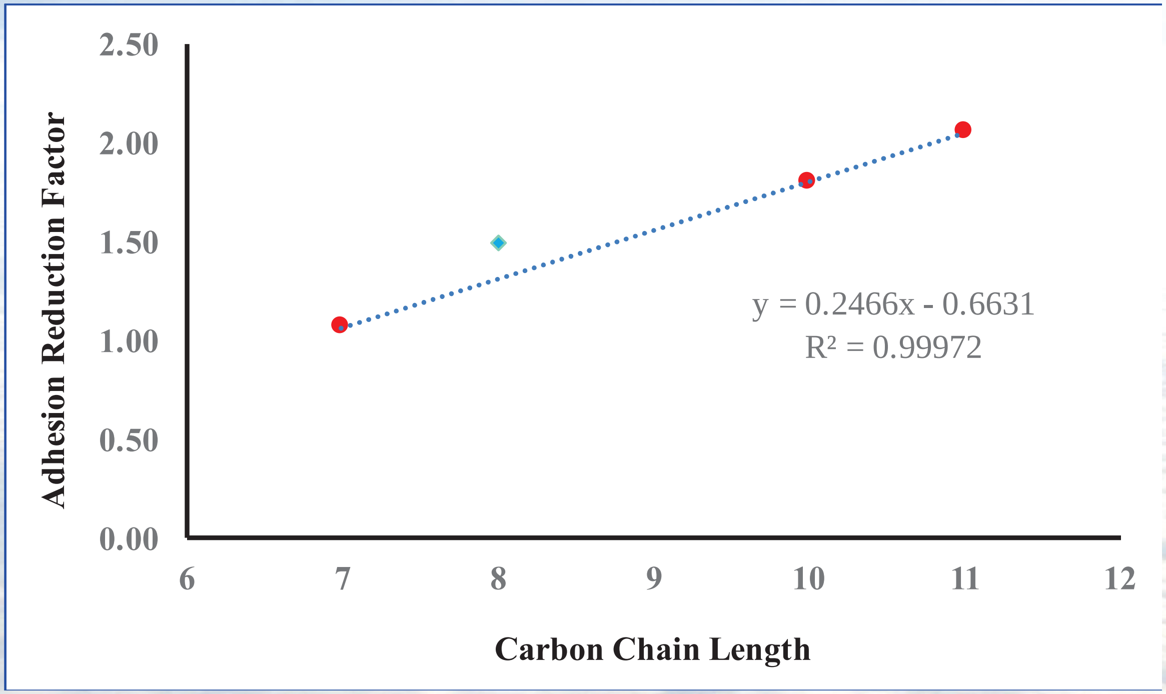


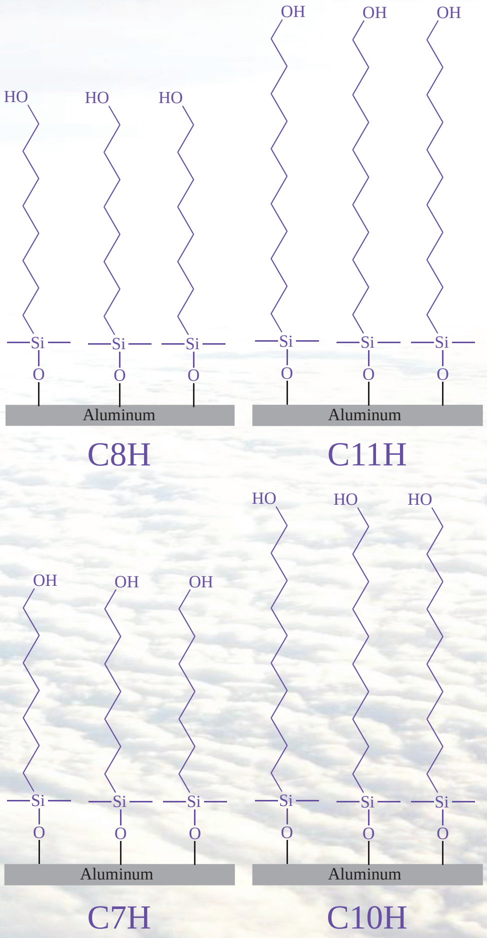
C7H



C10H

Hydrogen Bonding (Donor/Acceptor) ARF at -12°C





Acknowledgments

- NASA Glenn Research Center
  R.Eric Kreeger
- The Pennsylvania State University
  Jose Palacios, Taylor Knuth, Bryce Connelly
- South Dakota School of Mines and Technology
  Kevin Hadley, Nicholas McDougall
- The Governor’s School for Science and Technology
  Patrick Torchia
- NASA Internships, Fellowships, and Scholarships
  Rachel Brooks, Samuel Robbins
- Aeronautics Research Mission Directorate
  NASA Aeronautics Research Institute (2012 – 2015)
  Advanced Air Transport Technology (2015 – present)

Multiscale Design of Low Ice Adhesion Materials

Yan Wang (Georgia Tech), Ali Dhinojwala (Univ. of Akron), Mario Vargas (NASA Glenn)

NASA Workshop on Low Ice Adhesion Materials

August 10, 2017

Icephobic Material Design Research Plan

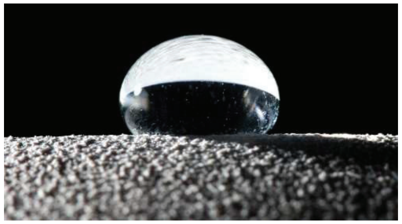
- We are developing a research plan for the multiscale design of a low ice adhesion coating material
  - The research plan will describe the computational, theoretical, and experimental research tasks needed to develop the coating material with the desired properties
- Document to be completed by December 1, 2017

Icephobic Materials

- Search for a external surface material which minimize wetting, accelerate water run-off, repel the ice and minimize ice adhesion has been ongoing since the first icing encounters during flight
- Many materials and coatings have been considered over the years, including: paint, polymers, nano-fluorocarbon, silicone coating, and slippery, liquid infused porous surfaces (SLIPS)
- The search still continues today with many materials and coatings being developed

Existing Research Efforts - Icephobic Materials

- Super Hydrophobic Surface (SHS)
  - Delayed ice nucleation and propagation



- Biomimetic Icephobic Material (Anti-Freezing Protein)
  - Thermal hysteresis
  - Recrystallization inhibition
- Slippery Liquid-Infused Porous Surface
  - Low adhesion

Desired Characteristics of Icephobic Materials

1. The icephobic material has to **withstand erosion, wear, corrosion** and other weathering conditions in terms of its structural integrity
2. The material has to be tested in a **realistic and dynamic environment** such as inside an icing tunnel at high velocity impact droplet, analogous to the conditions encountered during flight or in-situ test during actual flight
3. The material has to be **inexpensive to manufacture** and coherent with native structural materials, and environmentally friendly

Research Challenges in Physical Experiments

- Test standardization
  - Adhesion
  - Durability

- In-situ measurement
  - High-speed impacting droplet
  - High-fidelity high-throughput
  - Nanoscale characterization

Existing Research Efforts – Simulation

- Molecular Dynamics
  - Ice-water transition [Stillinger & Rahman 1972; Weber & Stillinger 1983; Kroes 1992; and MORE]
  - Hydrophobicity [Lee & Rossky 1994; Koishi 2009; and MORE]
  - Anti-freezing protein mechanism [Wen & Laursen 1992; Haymet & Kay 1992; Jorgensen et al. 1993; Madura et al. 1996; Chen & Jia 1999; and MORE]
  - Quasi-liquid layer structure [Nada & Furukawa 2000; Hayward & Haymet 2001]
  - Homogeneous nucleation [Matsumoto et al. 2002; Pluhařová et al. 2010; Zaragoza et al. 2015; Li et al. 2011; and MORE]
  - Heterogeneous nucleation [Cox et al. 2015; and MORE]
  - Calculation of surface adhesion [Landman et al. 1992; Miesbauer et al. 2003; Song et al. 2006; Kisin et al. 2007; and MORE]

Existing Research Efforts – Simulation

- Quantum Mechanics
  - Surface energy [Cheng et al. 2002; Liu et al. 2005; and MORE]
- Monte Carlo
  - Wettability [Pangali et al. 1979; Swaminathan & Beveridge 1979; Kumar et al. 2011; and MORE]
  - Ice growth [Dong et al. 2017]
- Computational Fluid Dynamics / Lattice Boltzmann Method
  - Droplet impact [Zu & Yan 2016; Yuan & Zhang 2017; Yao et al. 2017]
  - Droplet coalescence on SHS [Wang et al. 2017]

Research Challenges in Modeling & Simulation

- Size and time scales
  - Time scale mismatch
- Prediction credibility
  - Lack of confidence
- Integration between simulation and experiment
  - Computational simulation itself is NOT **design**

Existing Research Efforts – Data-Driven Modeling

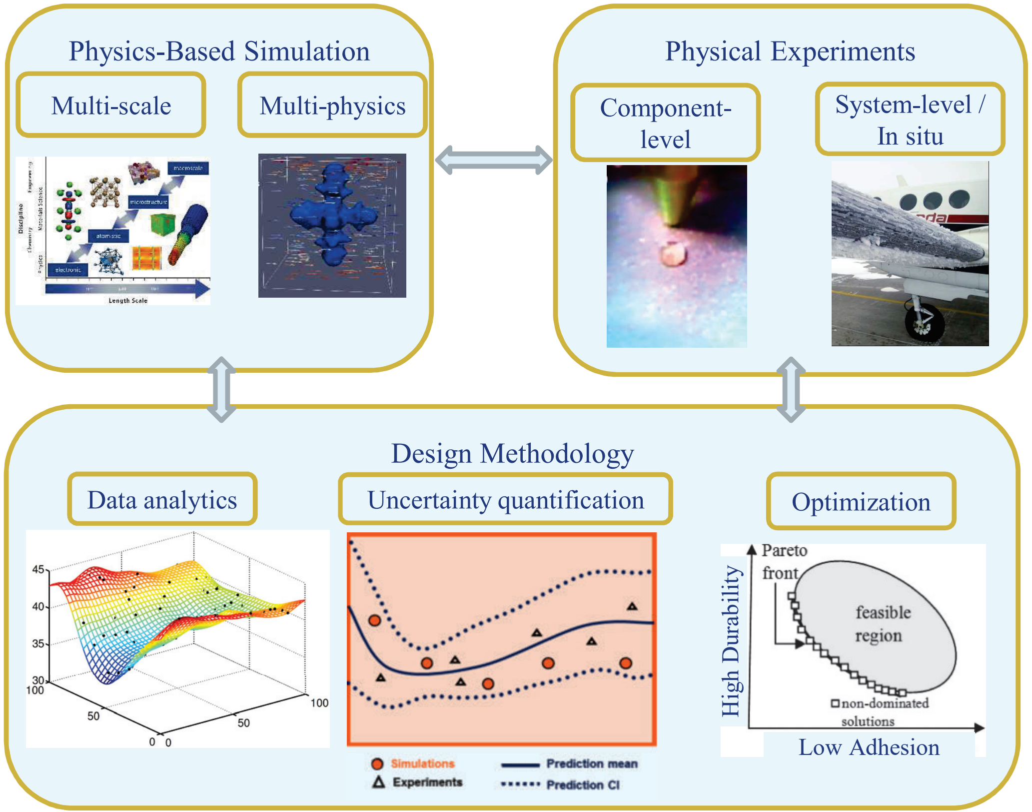
- Structure-Property Classification of Anti-Freezing Proteins (AFPs)
  - ***Classification based on machine learning*** (random forest, support vector machine, etc.) [Kandaswamy et al. 2011; and MORE]
  - ***Quantitative structure activity relationship*** classification [Briard et al. 2016]
- Feature Identification of AFPs
  - Dimensionality reduction based on principal component analysis

Research Objective

- To systematically design and implement a coating material with low ice adhesion and high durability based on materials design principles and methodology

A New Physics-Based Data-Driven Materials Design Framework

- ***Design*** is a systematic searching process to enumerate feasible solutions that meet the requirements and find the optimum from the feasible ones.



Physics-Based Simulation

Multi-scale

Multi-physics

Physical Experiments

Component-level

System-level / In situ

Design Methodology

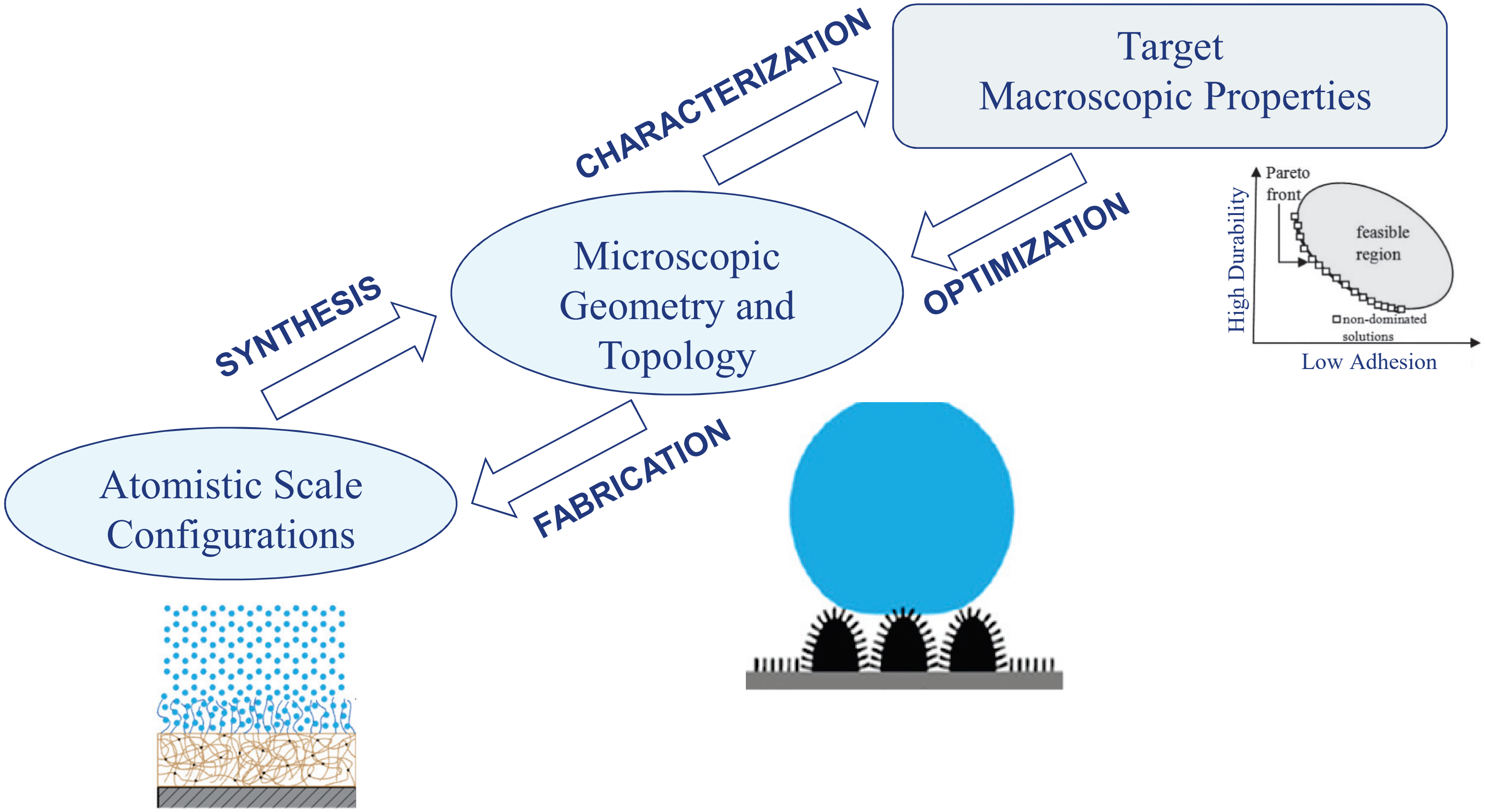
Data analytics

Uncertainty quantification

Optimization

Multiscale Materials & Process Design

- Establishment of **P**rocess-**S**tructure-**P**roperty relationship



Atomistic Scale Configurations

SYNTHESIS

Microscopic Geometry and Topology

CHARACTERIZATION

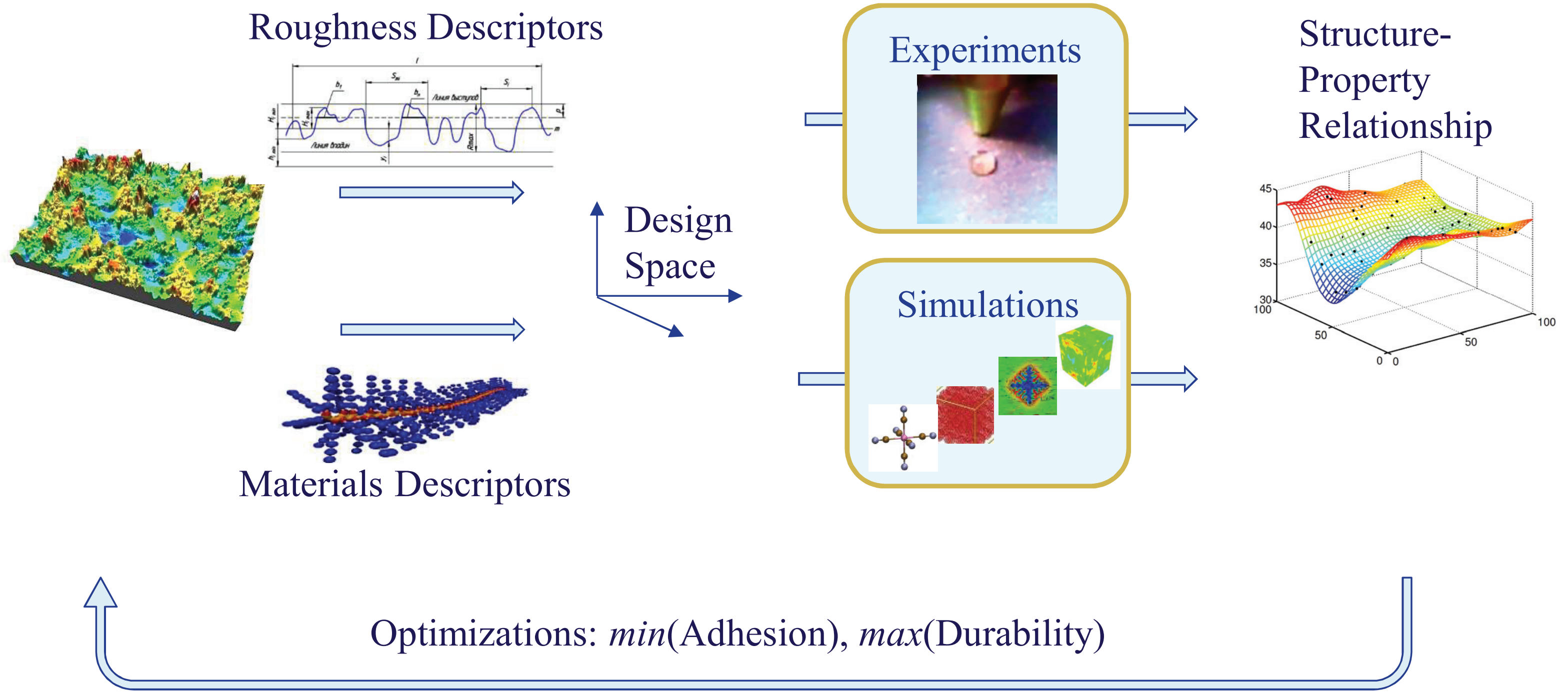
Target Macroscopic Properties

OPTIMIZATION

FABRICATION

Materials Design Process

- *Roughness* (**S**) − *Adhesion* (**P**) relationship establishment



Roughness Descriptors

Materials Descriptors

Design Space

Experiments

Simulations

Structure-Property Relationship

Optimizations: *min*(Adhesion), *max*(Durability)

Materials Design Process – Research Tasks

Macroscopic Test

- Ice adhesion
- Frost inhibition
- Durability
    - impact resistance
    - abrasion

Materials

- Characterization
    - chemistry
    - surface roughness
    - mechanical property
- Surface modification

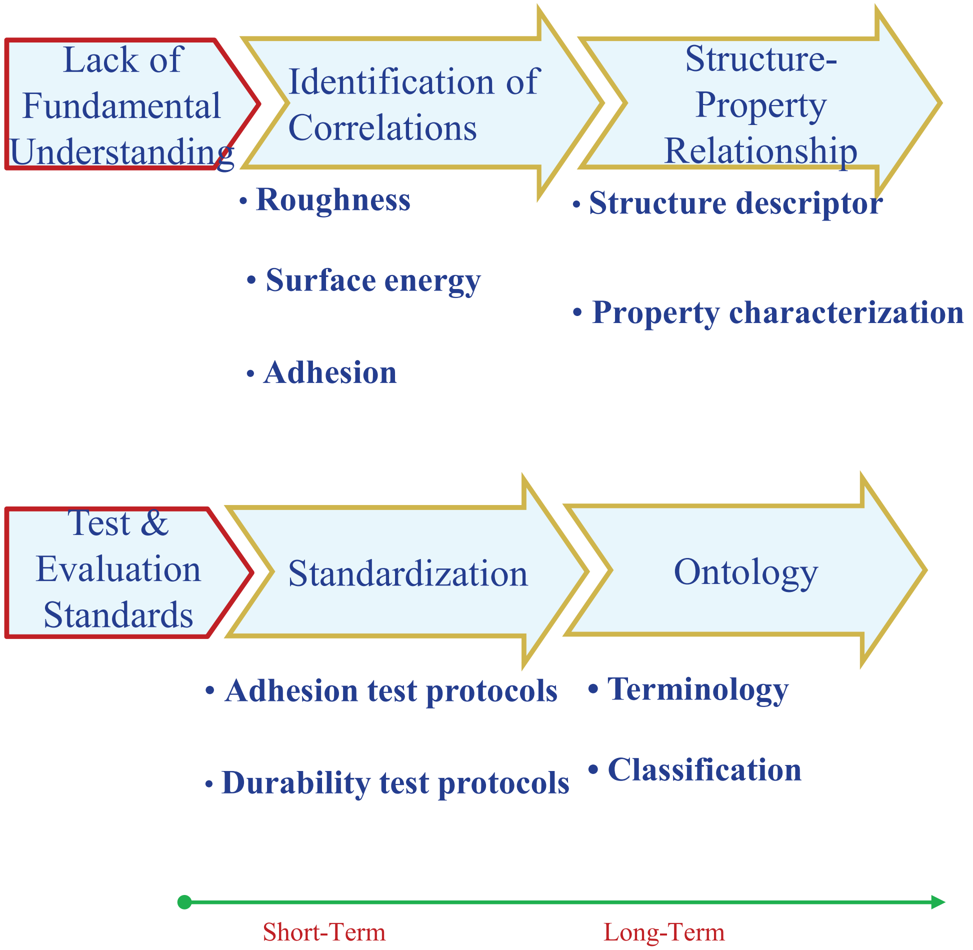
Simulation

- Atomistic
    - nucleation
    - adhesion
- Mesoscale
    - phases
- Macroscale
    - heat transfer
    - droplet impact

Structure-Property Relationship

- Identify materials and roughness descriptors
- Metamodeling and statistical machine learning
- Multi-objective optimization

Research Challenges & Opportunities – Design Parameter Identification



Lack of Fundamental Understanding → Identification of Correlations → Structure-Property Relationship

Identification of Correlations:
- Roughness
- Surface energy
- Adhesion

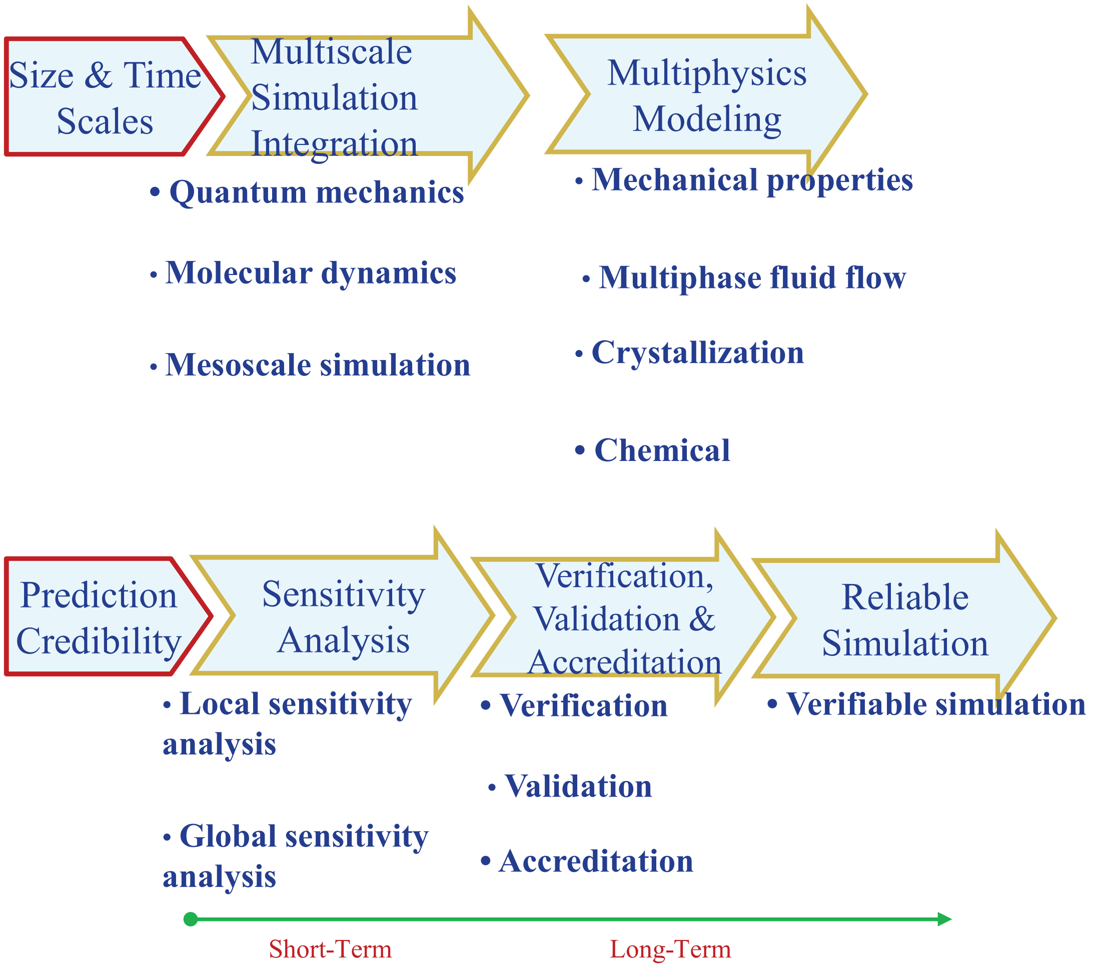
Structure-Property Relationship:
- Structure descriptor
- Property characterization

Test & Evaluation Standards → Standardization → Ontology

Standardization:
- Adhesion test protocols
- Durability test protocols

Ontology:
- Terminology
- Classification

Research Challenges & Opportunities – Modeling & Simulation



Size & Time Scales

Multiscale Simulation Integration

- Quantum mechanics
- Molecular dynamics
- Mesoscale simulation

Multiphysics Modeling

- Mechanical properties
- Multiphase fluid flow
- Crystallization
- Chemical

Prediction Credibility

Sensitivity Analysis

- Local sensitivity analysis
- Global sensitivity analysis

Verification, Validation & Accreditation

- Verification
- Validation
- Accreditation

Reliable Simulation

- Verifiable simulation

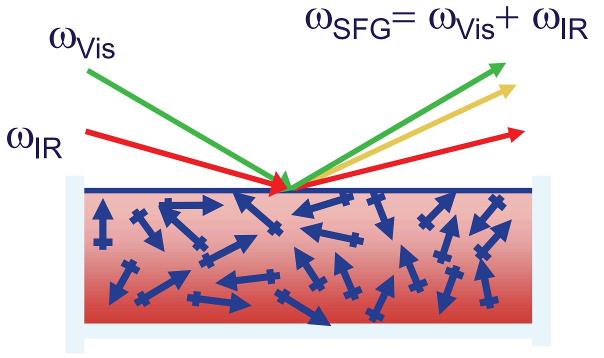
Short-Term

Long-Term

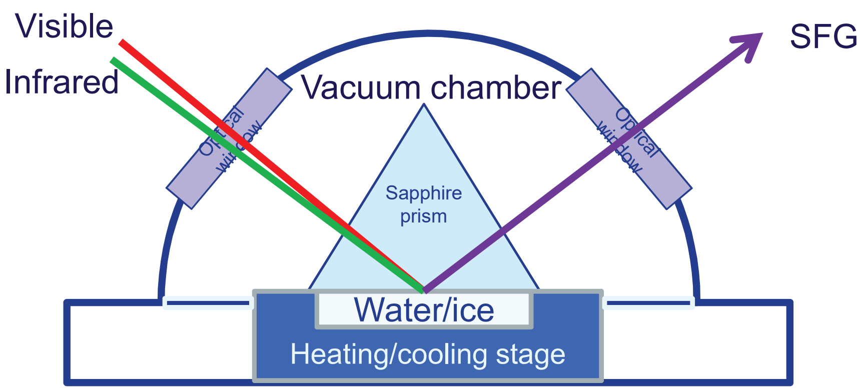
National Aeronautics and Space Administration

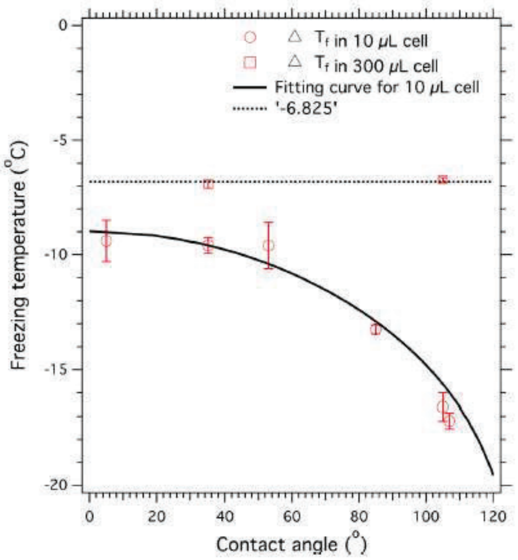
Direct Probe of Surface Nucleation

[Dhinojwala et al. 2012]



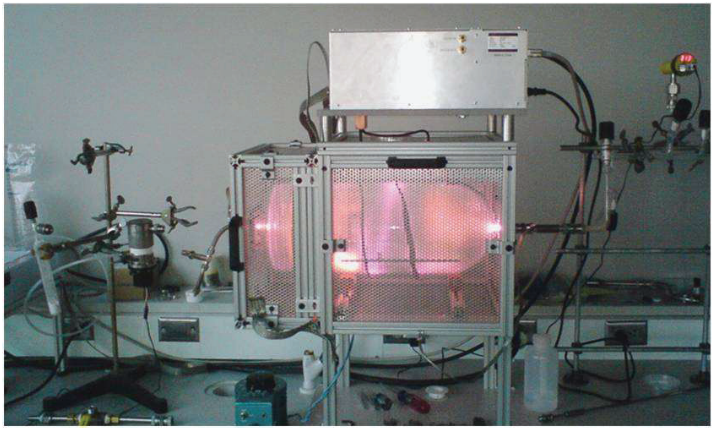
$$\chi^{(2)}_{eff} = \chi^{(2)}_{eff,NR} + \chi^{(2)}_{eff,R} = \chi^{(2)}_{eff,NR} + \sum_q \frac{A_q}{\omega_2 - \omega_q - i\Gamma_q}$$



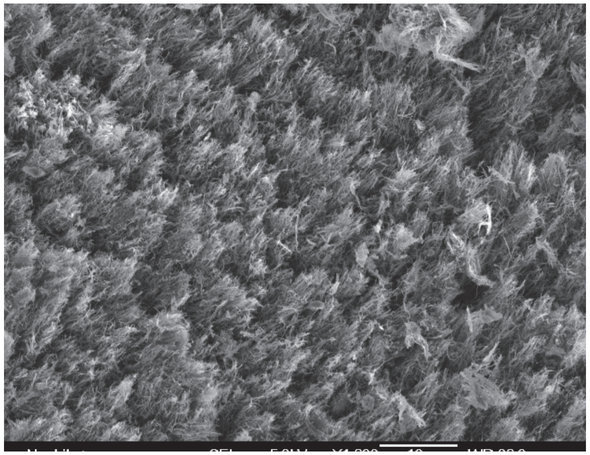


On-going: Inhibiting Frost Formation

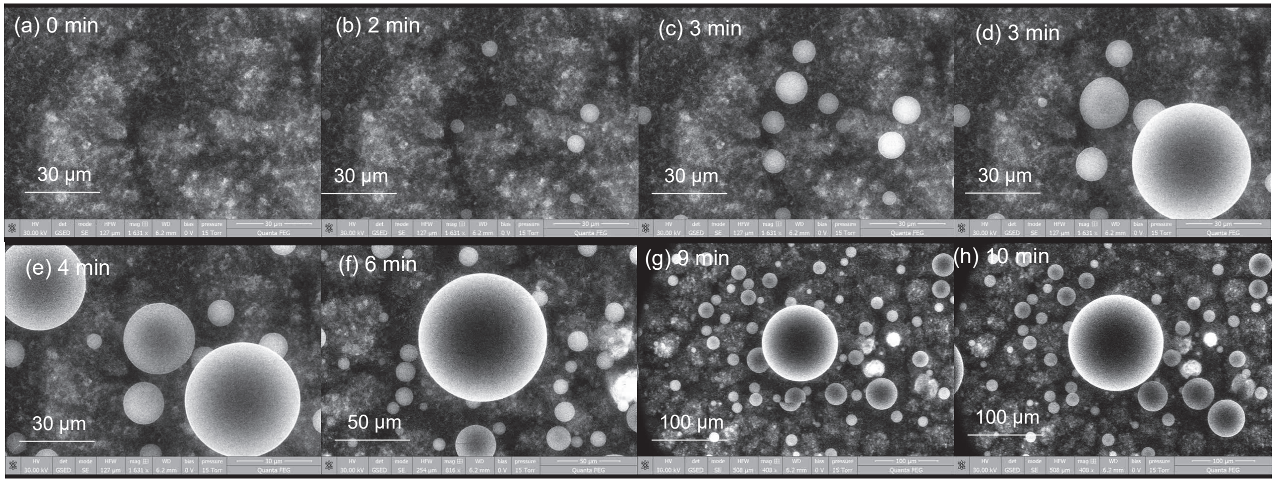
@Univ. of Akron



Plasma Modification of Surfaces



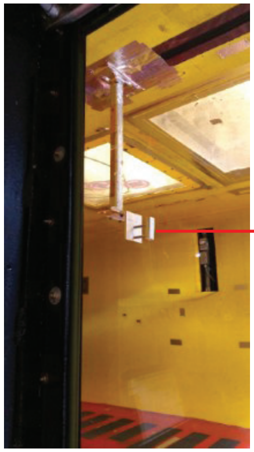
Carbon Nanotubes Growth on Steel Surfaces

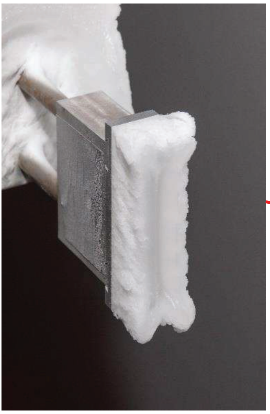


Stable Cassie State on Plasma Coated Surfaces

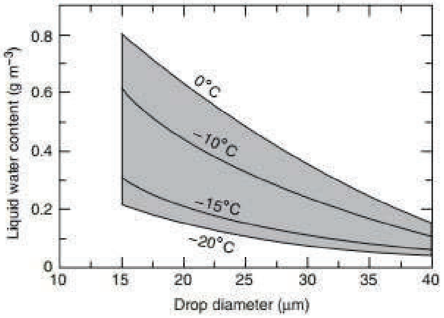
On-going: Ice Adhesion Testing of Coating Materials

@NASA Glenn





Coating exposed to Icing Conditions
FAA Part 25 Appendix C

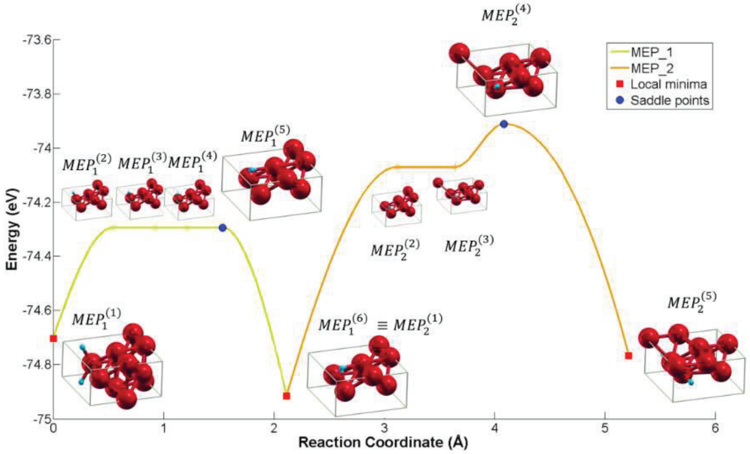


Adhesion Force Measured
in environmental chamber
using the newly developed
NASA Glenn methodology

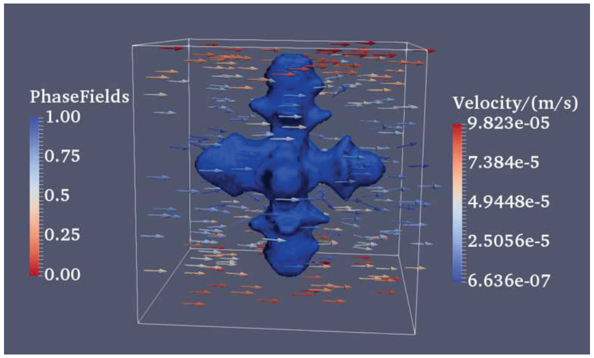
On-going: Process-Structure-Property Prediction based on Multiscale Multi-physics Simulations

@Georgia Tech

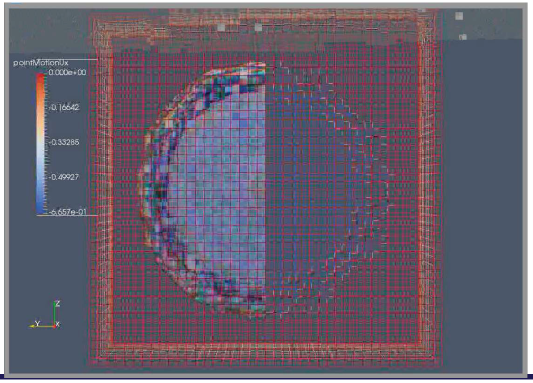
Scalable metamodeling and first-principles DFT phase transition prediction



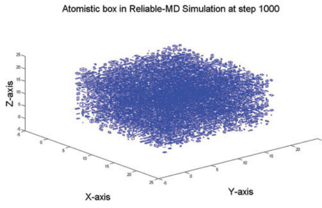
Mesoscale multi-physics simulation of fluid flow + thermal + phase change

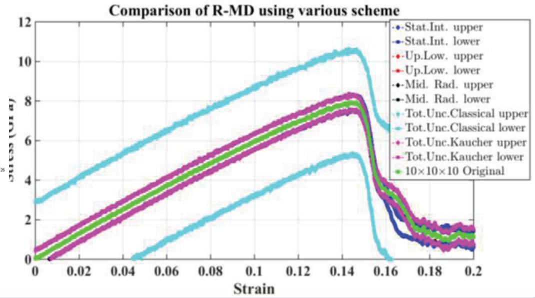


Macroscale simulation of droplet impact and ice formation



Reliable molecular dynamics simulation





Expected Research Outcomes of the Proposed Research

- A detailed research plan will be developed
  - Research tasks for each step of design process
  - Detailed experimental and computational activities
- Expected research outcomes
  - A generic icephobic materials design framework
    - Experimental and simulation data integration tool
    - Metamodeling and design optimization tools
  - A demonstrative new coating material by design
    - Synthesis and surface modification guideline
    - Adhesion and durability test results
  - Research reports and publications

Short Term Plans

- Begin collaboration with Glenn personnel already doing design of materials
  - Participate in completing the plan and future activities
  - Initial contact and conversations started
- Evolve the plan into a NASA Glenn initiative in collaboration with other NASA Centers and Academia
  - Explore funding sources (ex: CAS)

Questions?

Measurement of Impact Ice Adhesion Strength

Jose Palacios

Assistant Professor Aerospace Engineering

The Pennsylvania State University, University Park, PA

NASA Workshop on Low Ice Adhesion

8-10 2017

Presentation Outline

- Background & Objectives
- Testing Facility & Procedure
- Results
  - Material and Icing Parameter Effects
  - Evaluation of erosion resistant materials
  - Evaluation of superhydrophobic materials
- Conclusions

Background & Motivation

PennState AERTS

- Aircraft encounter adverse weather conditions, including icing events.
- Ice accretion severely degrades aerodynamic performance and introduces vibration.
- Active ice protection systems are costly, introduce complexity, and weight.
- Glaze ice
  - Characterized by water droplets splashing on impact & running before freezing
  - Forms clear Ice
  - Large particle size
  - Warmer temperatures
- Rime ice
  - Characterized by water droplets Freezing on impact and trapping air in the ice
  - Forms opaque Ice
  - Small particle size
  - Colder temperatures
- Mixed ice
  - Characterized by glaze main ice shape with rime feathers

Would not it be great to have a passive coating that prevents ice accretion for all varying icing conditions?!
Yes, an ICEPHOBIC COATING!!!

Maybe an ICE PROTECTIVE COATING
(Low Ice Adhesion Strength)

Glaze

Mix

Rime

Background & Motivation

PennState AERTS

Author Date (Reference)	Mechanical Test Type	Aluminum Shear Adhesion Strength		Ice Type
		psi	kPa	
Loughborough 1946	Pull	81	558	Freezer
Stallabrass and Price 1962	Rotating Instrumented Beam	14	97	Impact
Itagaki 1983	Rotating Rotor	4 - 23	27 - 157	Impact
Scavuzzo and Chu 1987	Shear Window	13 - 42	90 - 290	Impact
Reich 1994	Pull	130	896	Freezer
Brouwers 2011	Pull	76	526	Freezer

- Freezer ice tends to have higher adhesion strength over impact ice

- Moving ice from freezing camber to adhesion tester can damage the bond from unintended thermal changes and mechanical stress

- Surface roughness information is not published

PennState
AERTS

Objectives

- Experimentally determine what environmental and material surface properties contribute most significantly to ice adhesion strength
- Measure and compare ice adhesion strength of metallic erosion resistant materials used in aircraft manufacturing
- Explore the capability of superhydrophobic materials to reduce ice adhesion strength
- Initiate the development of a model that could predict ice adhesion strength

Presentation Outline

- Background & Objectives
- Testing Facility & Procedure
- Results
  - Material and Icing Parameter Effects
  - Evaluation of erosion resistant materials
  - Evaluation of superhydrophobic materials
- Conclusions

PennState
AERTS

Testing Requirements

Ice accretion must be representative of aircraft environments by controlling:

- Liquid Water Content
- Water Droplet Median Volume Diameter
- Temperature
- Impact Velocity

Material surface characteristics must be known:

- Surface Roughness
- Surface Temperature during Accretion

The ice shear adhesion strength should be quantified

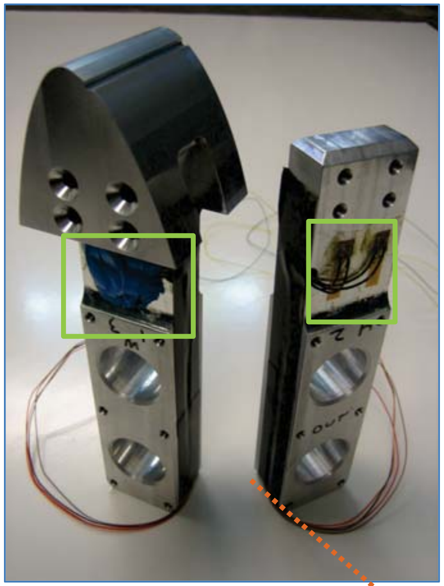
The accreted ice CANNOT BE TOUCHED, MOVED, OR EXCITED TRANSIENTLY:

- Must avoid undesired energy that could pollute shear ice adhesion strength data
- How?: CENTRIFUGAL TESTING SUBJECTED TO AN ICING CLOUD

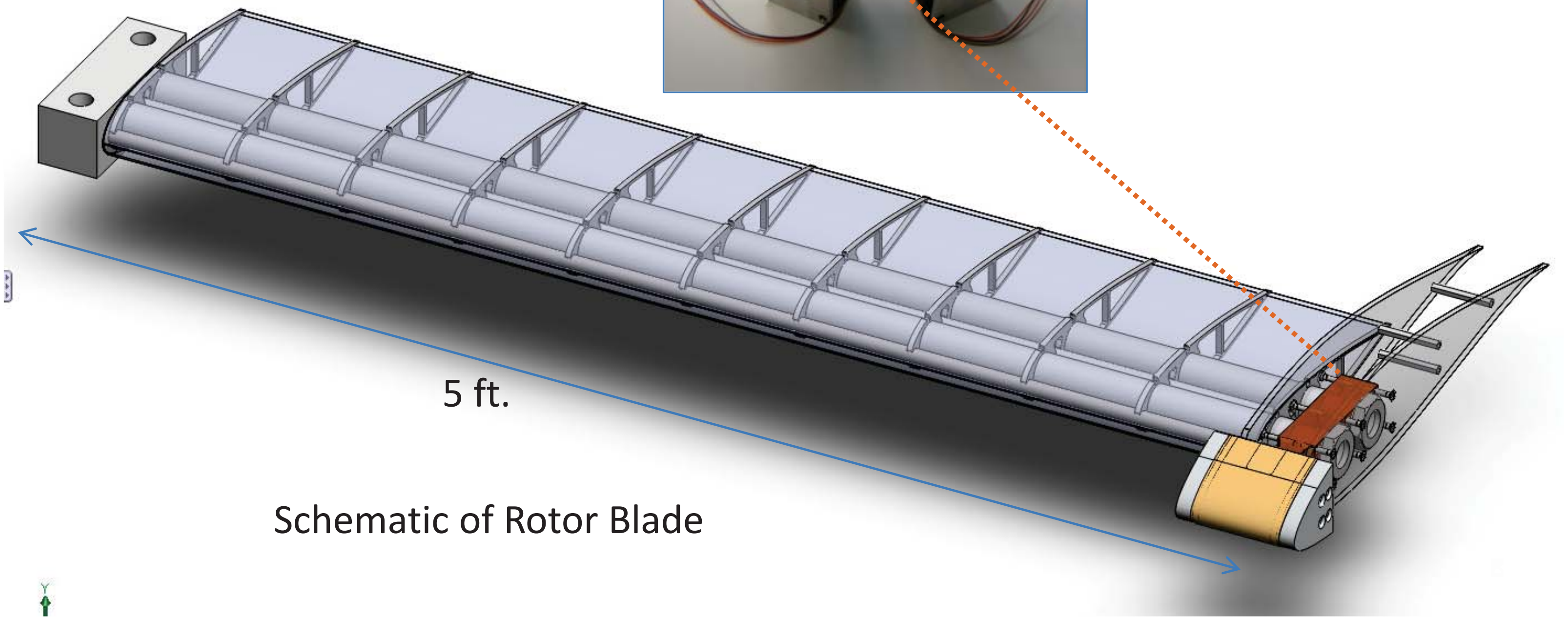
Adhesion Strength Measurements

Centrifugal Bending Beam
- Stallabrass 1962

Benefits
- Ice is accreted and shed without outside interaction

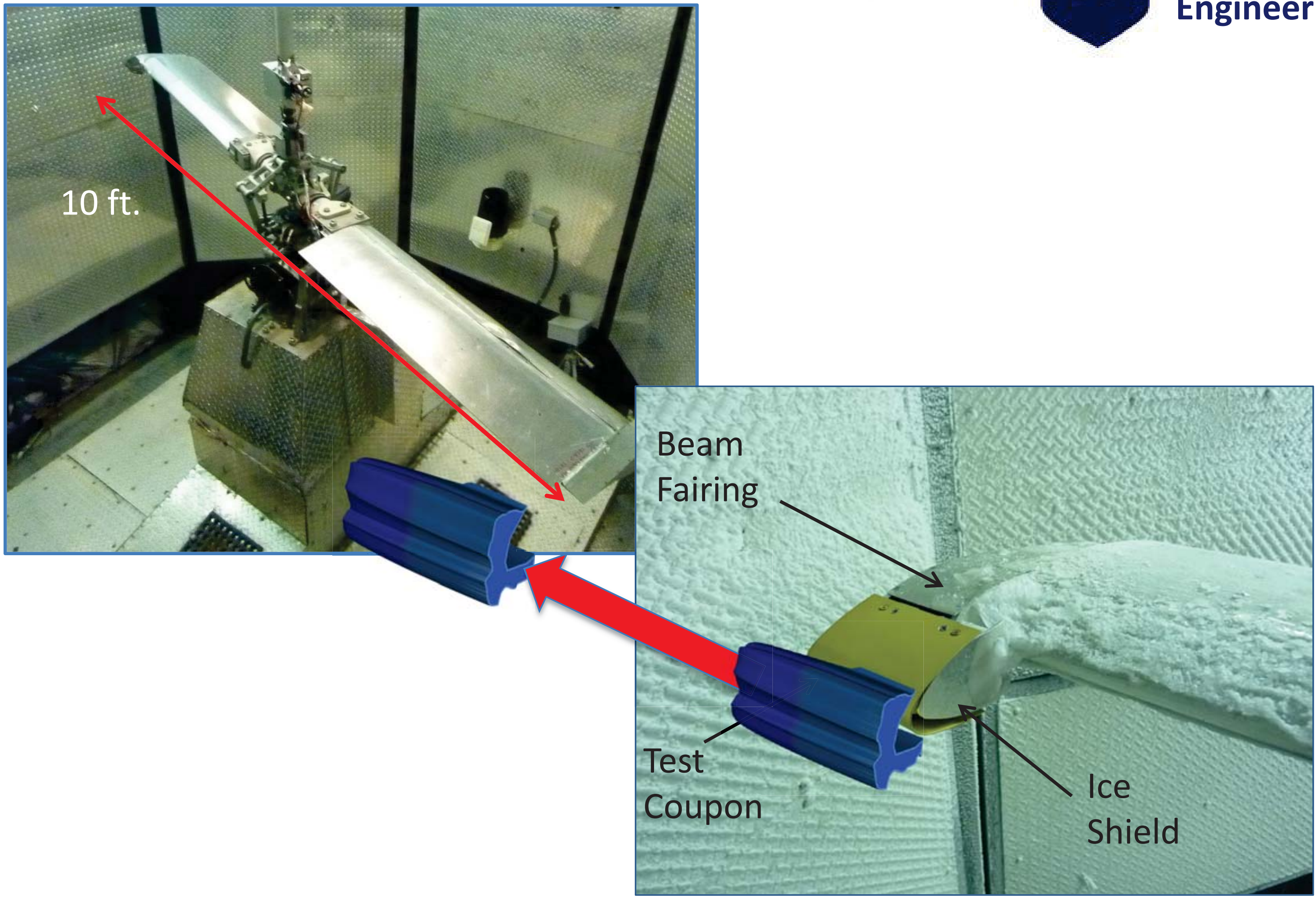


Strain Gauges
- Full Wheatstone Bridge
- Encapsulated for water proofing



Schematic of Rotor Blade

Adhesion Strength Measurements

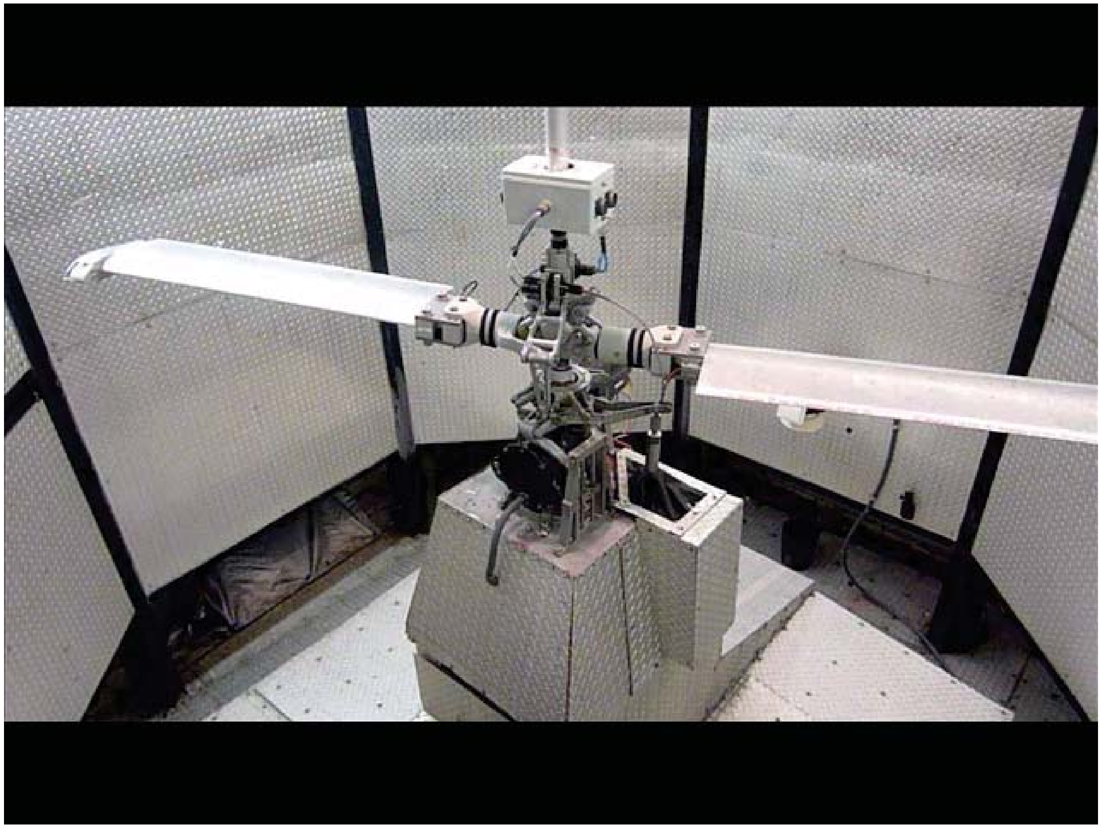


Adhesion Strength Measurements

PENNSTATE
1855
Aerospace
Engineering

As ice accretes, strain gauges sense an increase in bending due to ice load

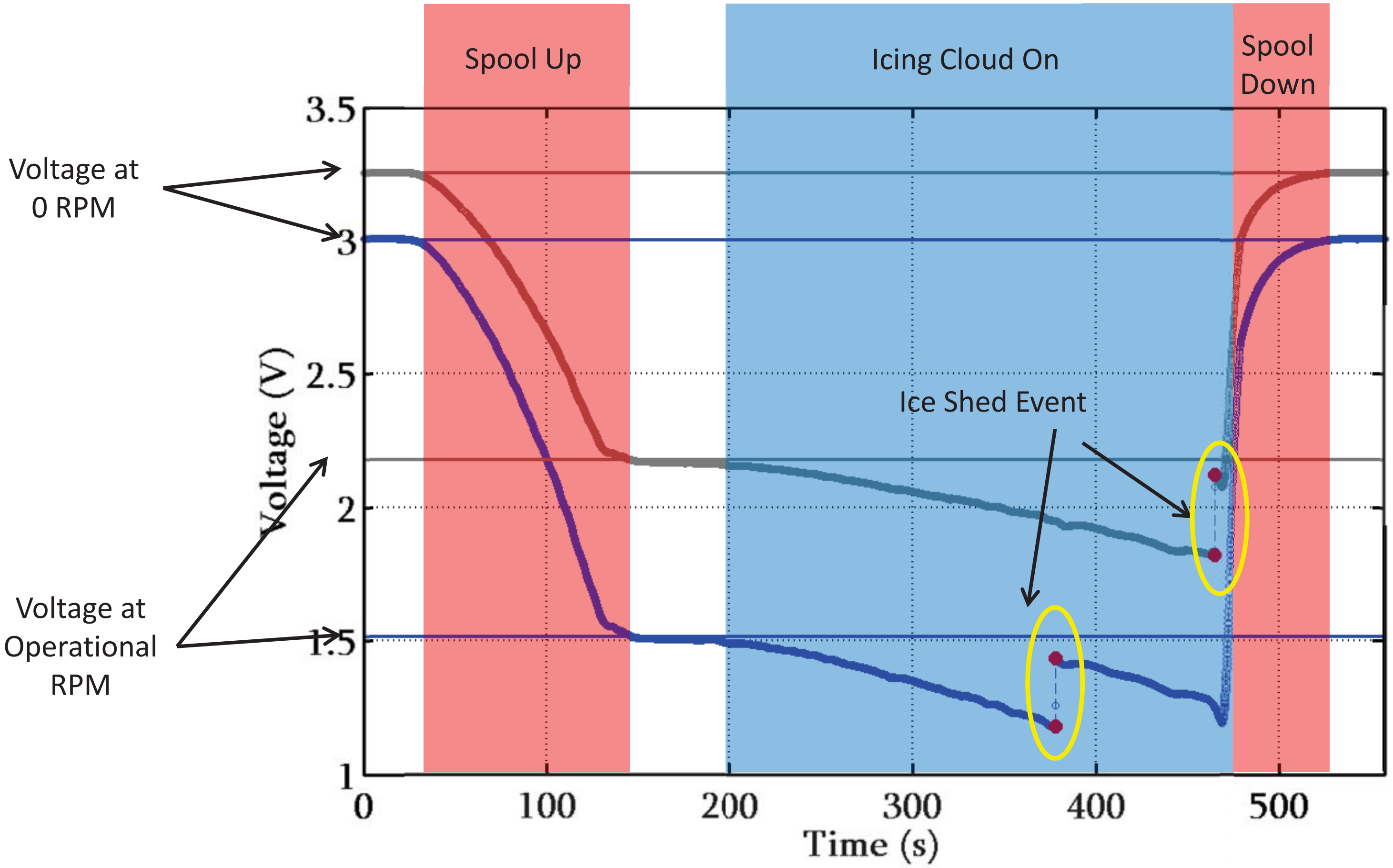
$$\tau = \frac{L_{ice}}{A_s}$$



Typical Voltage Output During Test

PENNSTATE

Aerospace
Engineering

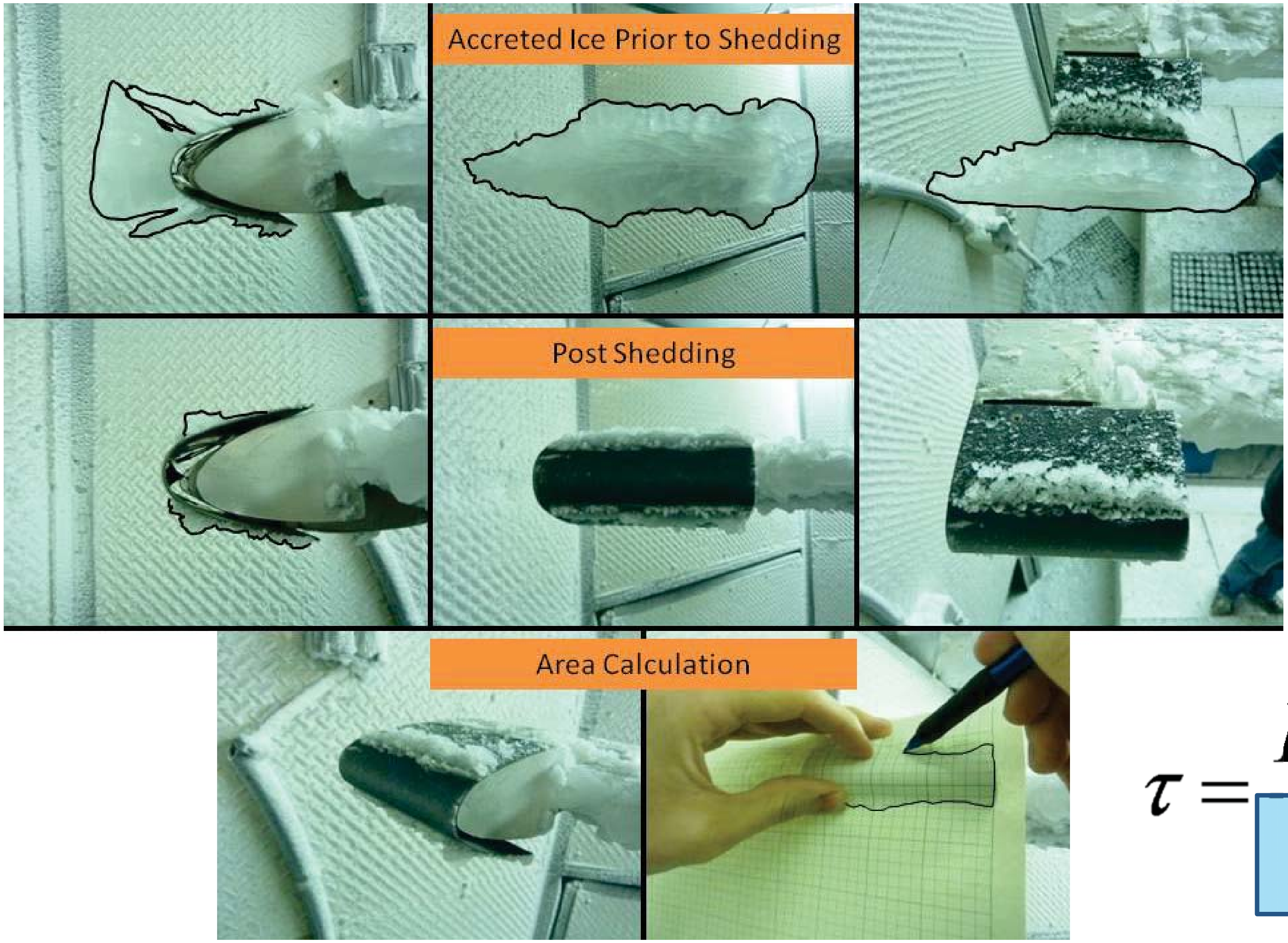


$$\tau = \frac{\boxed{L_{ice}}}{A_s}$$

Area Calculation

PENNSTATE

Aerospace Engineering



$$\tau = \frac{L_{ice}}{\boxed{A_s}}$$

Experimental Uncertainty

PENNSTATE
Aerospace Engineering

Researcher	Test Type	% deviation
Soltis	Rotor beam	20
Brouwers	Rotor beam	23
Hassan	Vibrating beam	40
Laforte	Centrifuge	18

NASA Glenn Icing Research Tunnel Error	
MVD	12
LWC	12

Presentation Outline

- Background & Objectives
- Testing Facility & Procedure
- Results
  - Material and Icing Parameter Effects
  - Evaluation of erosion resistant materials
  - Evaluation of superhydrophobic materials
- Conclusions

PennState AERTS

Environmental and Surface Parameters of Interest

Environmental Conditions

Cloud density (LWC)

0.5 g/m³, 2.0 g/m³, 5.0 g/m³

Particle size (MVD)

20 μm, 30 μm, 40 μm

Temperature

-5°C , -10°C , -15°C

Material Surface Characteristics

Surface roughness

20 μin Ra, 50 μin Ra, 100 μin Ra

Grain direction

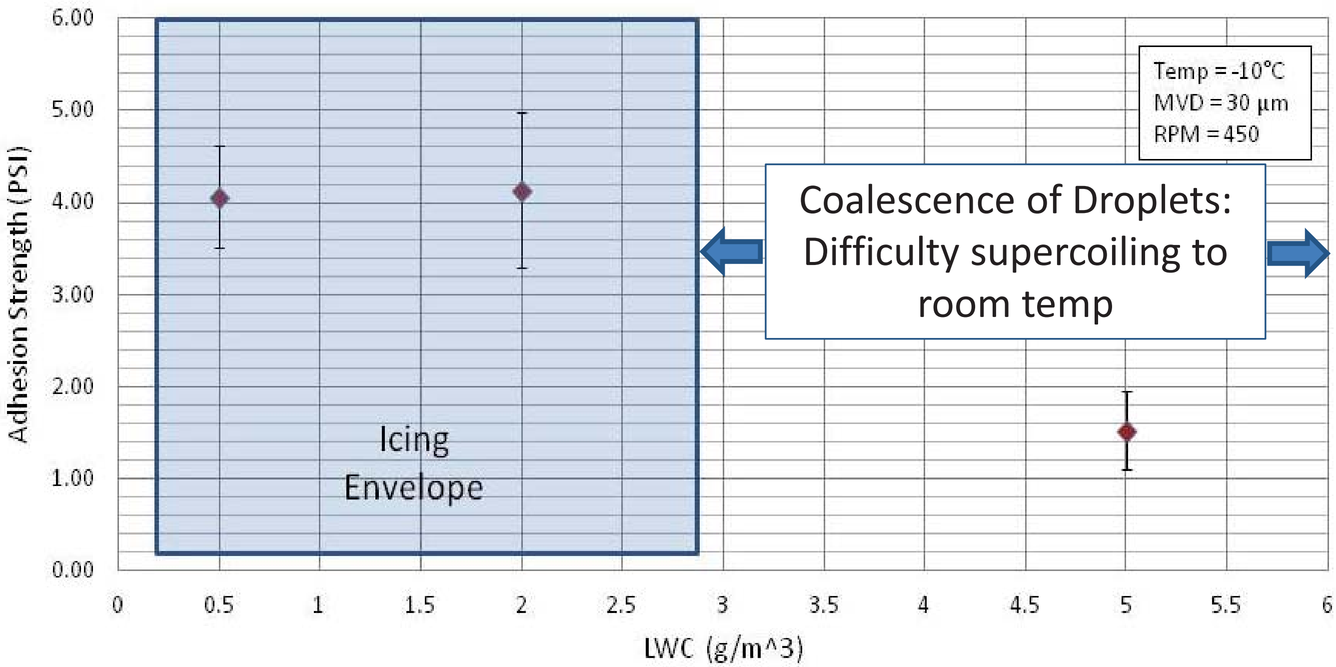
0°, 90°

Test Material

Stainless steel 430

Impact velocity 70 m/sec

Effect of Cloud Density



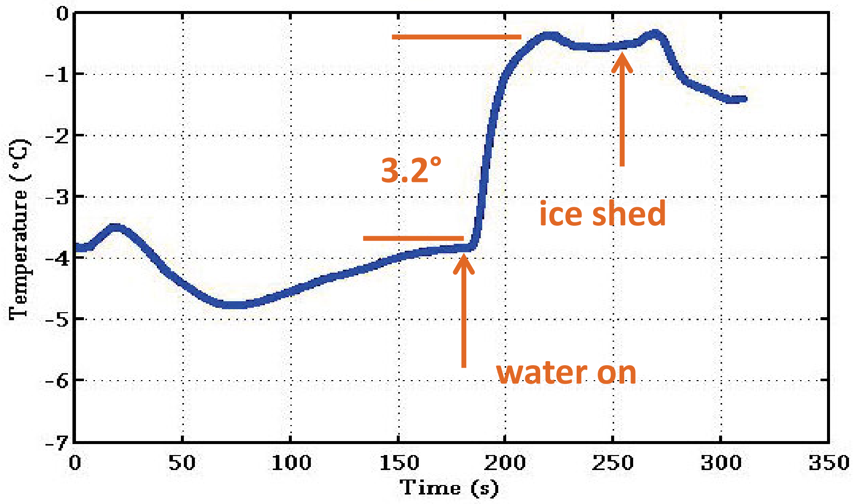
Coalescence of Droplets: Difficulty supercoiling to room temp

- Over the FAA LWC icing envelope, ice adhesion strength is constant
- Higher LWC (not to exceed 2 g/m^3) might be used to reduce testing time

Effect of Cloud Density

PENNSTATE
1855
Aerospace Engineering

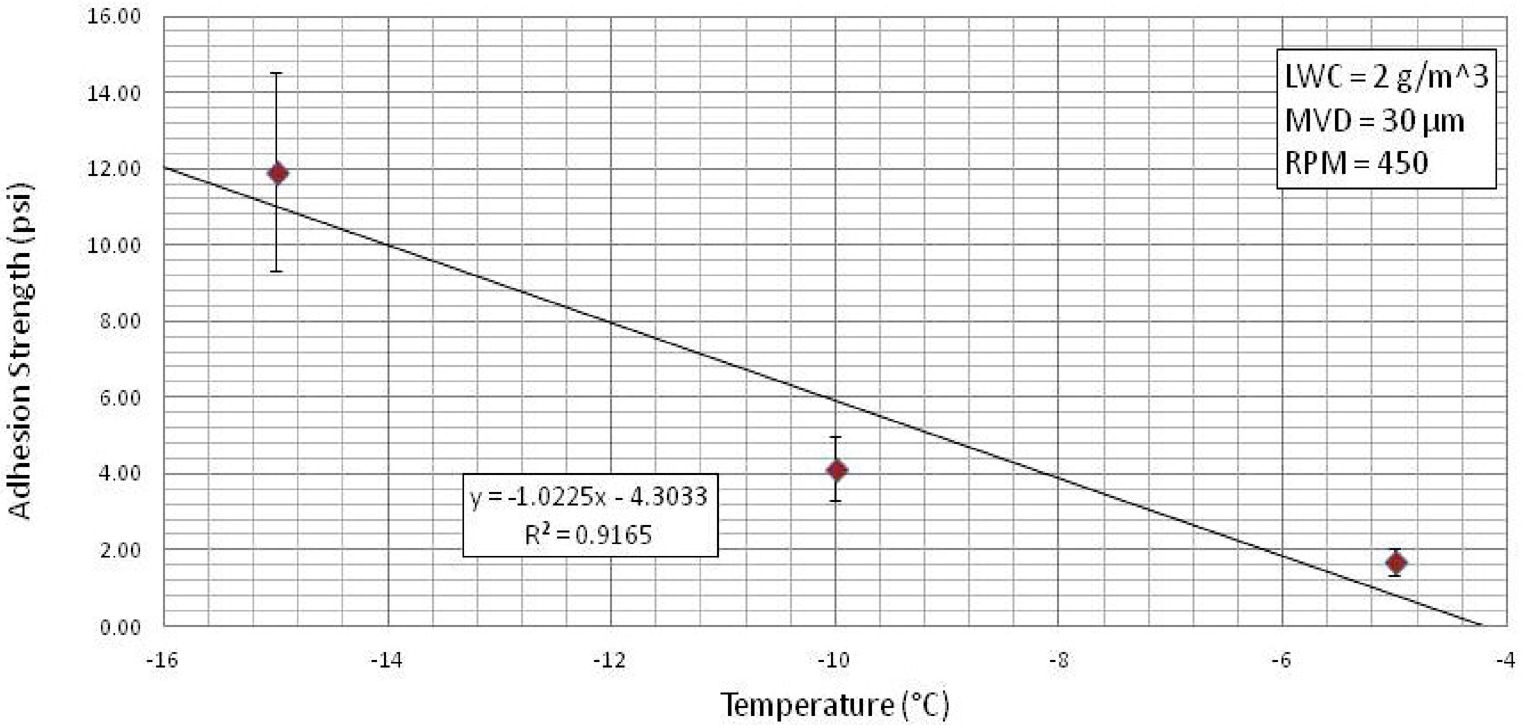
5.0 g/m^3 LWC



- At cloud densities above those specified in the FAA icing envelope, the super cooling of the drops is difficult due to coalescence.
- The surface temperature of the coupon increases causing a decrease in adhesion strength (see effects of temperature).

PENNSTATE
1855
Aerospace Engineering

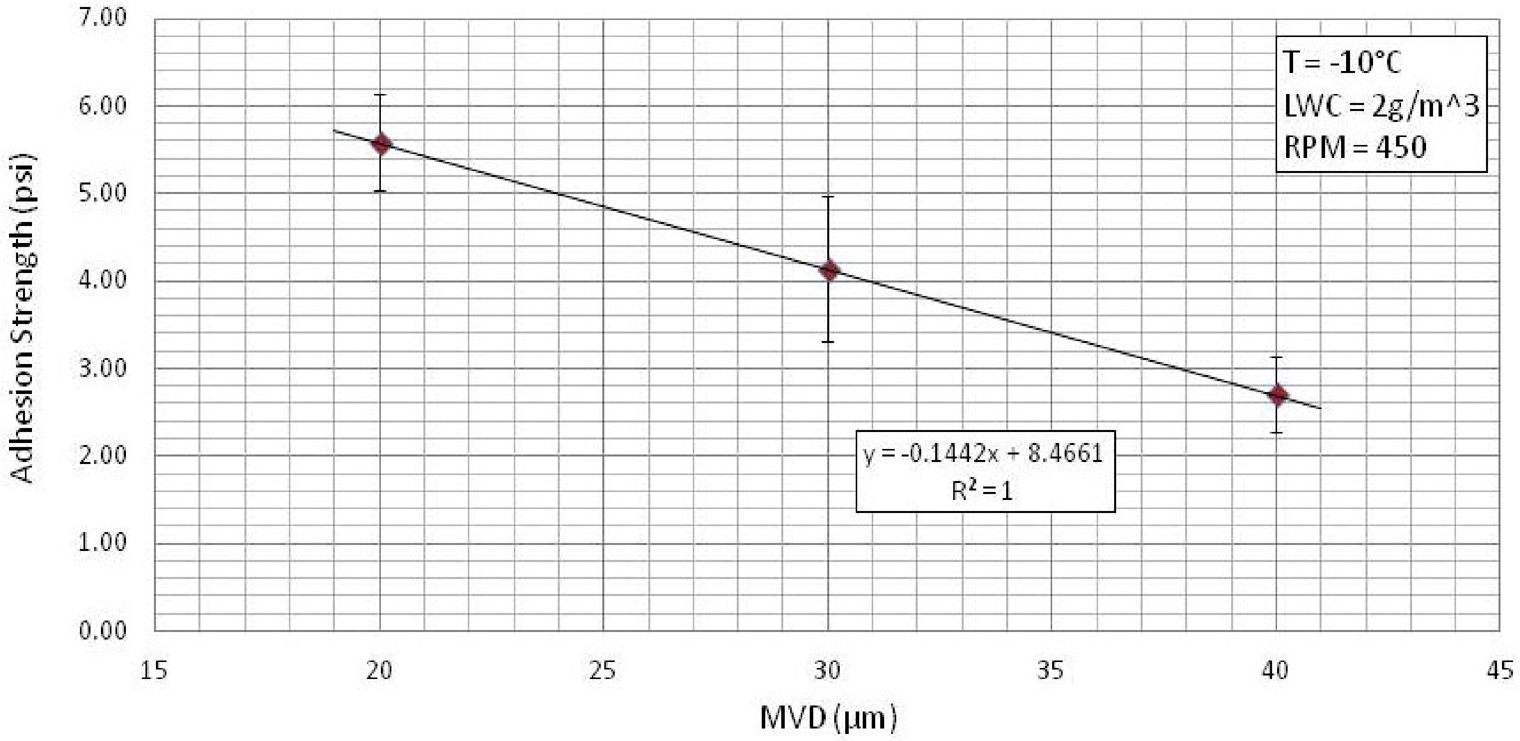
Effect of Ambient Temperature



- Ice adhesion strength is linearly dependant with ambient temperature
- 600% reduction in adhesion strength from -15 °C to -5 °C

PENNSTATE
Aerospace Engineering

Effect of Particle Size



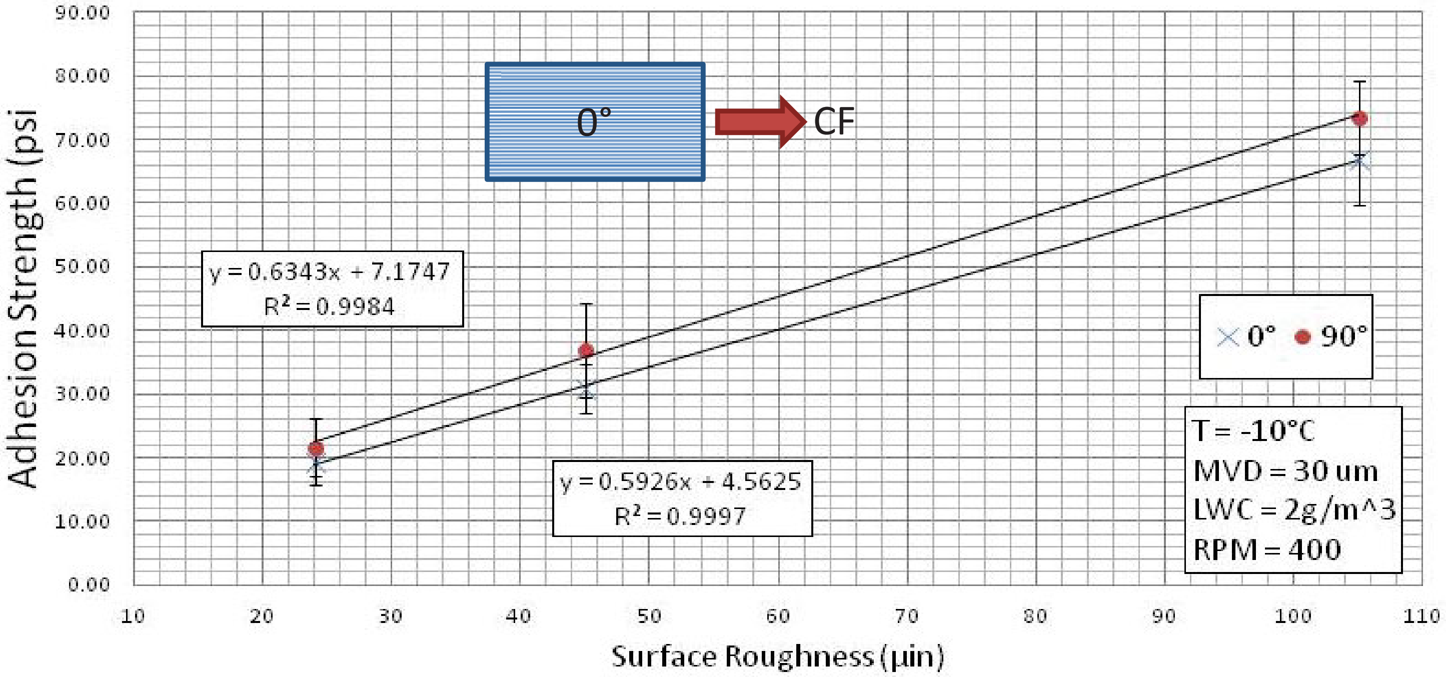
- Ice adhesion strength is linearly dependant with MVD
- 52% change in adhesion strength from 20 μm to 40 μm

PENNSTATE

1 8 5 5

Aerospace Engineering

Surface Roughness and Grain Direction



- Ice adhesion strength is linearly dependant with surface roughness
- 246% increase in adhesion strength from 24 µin *Ra* to 105 µin *Ra*
- 14% increase in adhesion strength from 0° to 90° grain direction

PENNSTATE
Aerospace Engineering

Environmental and Surface Effects Summary

Properties	% Change Over Range
LWC	2
MVD	52
Temperature	600
Surface Roughness	246
Grain Direction	14

The linear trends in adhesion strength with temperature and surface roughness could used to reduce test matrix size*.

*Soltis, J., Palacios, J., Eden, T., & Wolfe, D. (2014). Ice Adhesion Mechanisms of Erosion-Resistant Coatings. *AIAA Journal: 1-9, June 2014, 10.2514/1.J053208.*

Presentation Outline

PennState AERTS

- Background & Objectives
- Testing Facility & Procedure
- Results
  - Material and Icing Parameter Effects
  - Evaluation of erosion resistant materials
  - Evaluation of superhydrophobic materials
- Conclusions

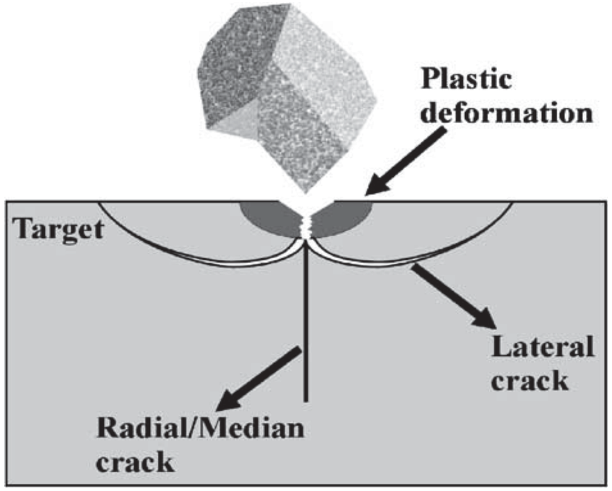
PENNSTATE

Aerospace Engineering

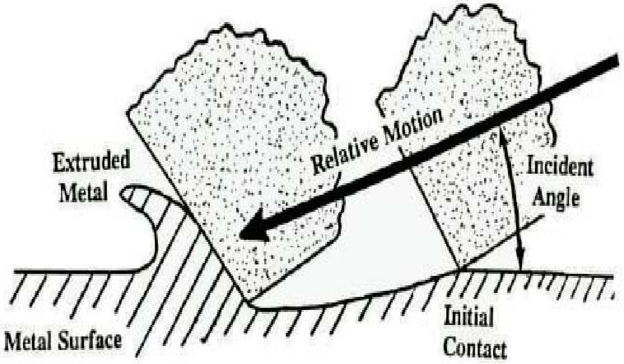
Erosion Background

- During takeoff and landing propellers & rotors ingest dirt and debris
- The particles impact the rotor blades and material is removed
- Life span of the blades is reduced


Shephard 3/16/12



Brittle erosion



Ductile erosion

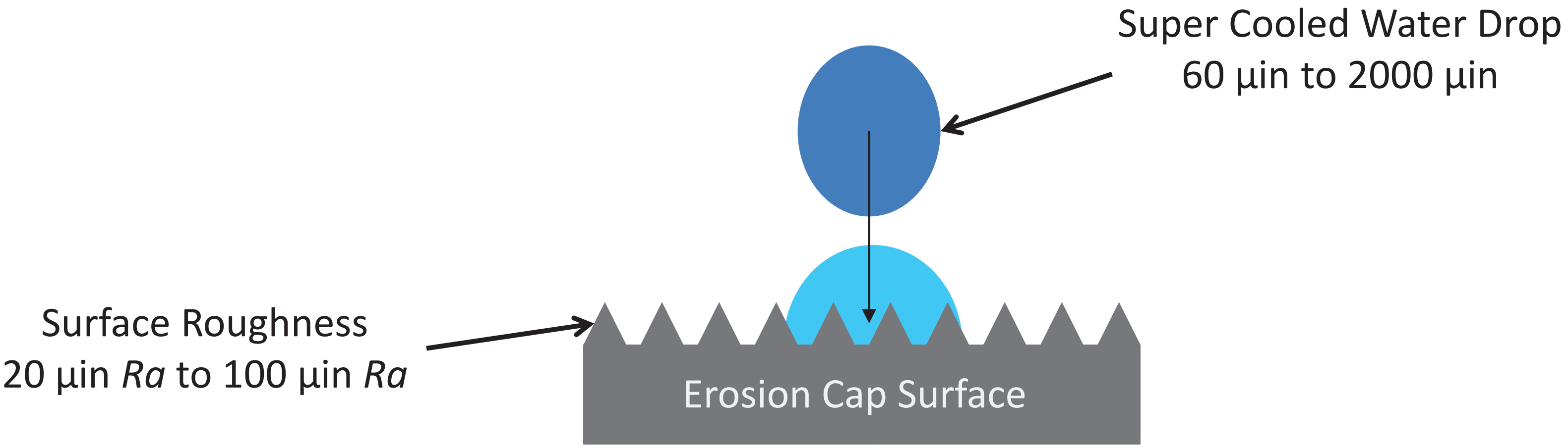
Motivation

PENNSTATE
1 8 5 5
Aerospace Engineering

Hypothesis

- Ice adheres to the substrate surface due to mechanical clamping

- Increasing surface roughness increases adhesion strength supporting the hypothesis



Erosion Resistant Material Testing

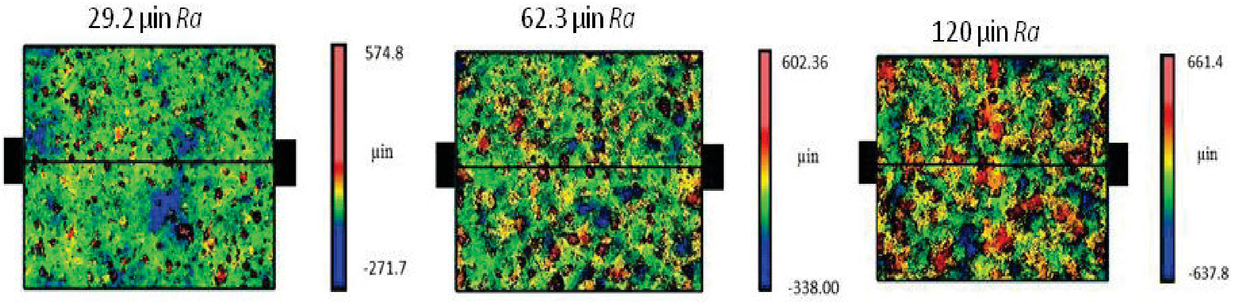
PENNSTATE

1855

Aerospace Engineering

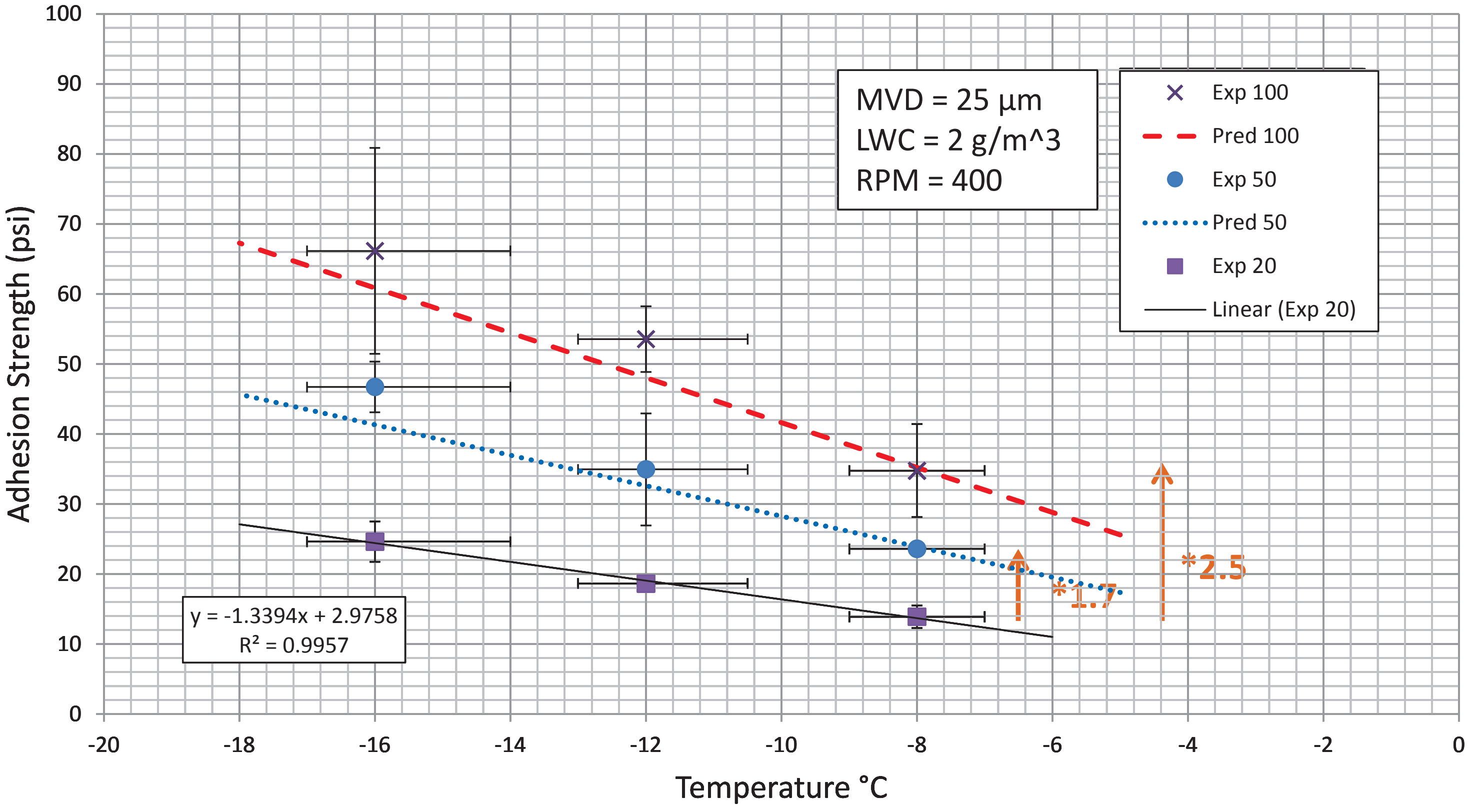
Goal 1: determine the impact ice adhesion strength of erosion resistant materials

- Materials: Stainless steel 430
  - Inconel 625
  - Titanium grade 2
  - Titanium nitride (TiN)
  - Titanium aluminum nitride (TiAlN)
- Surface Roughness (µin *Ra*): 20,50,100
- Temperature (°C) : -8 , -12 , -16
- MVD (µm): 25
- LWC (g/m^3): 2.0
- RPM: 400
- Tip speed: 58.7 m/s, 193 ft/s
- Cathodic arc physical vapor deposition
- Coating thickness: 15µm
- Titanium grade 2 substrate



Impact Ice Adhesion Strength for Stainless Steel 430

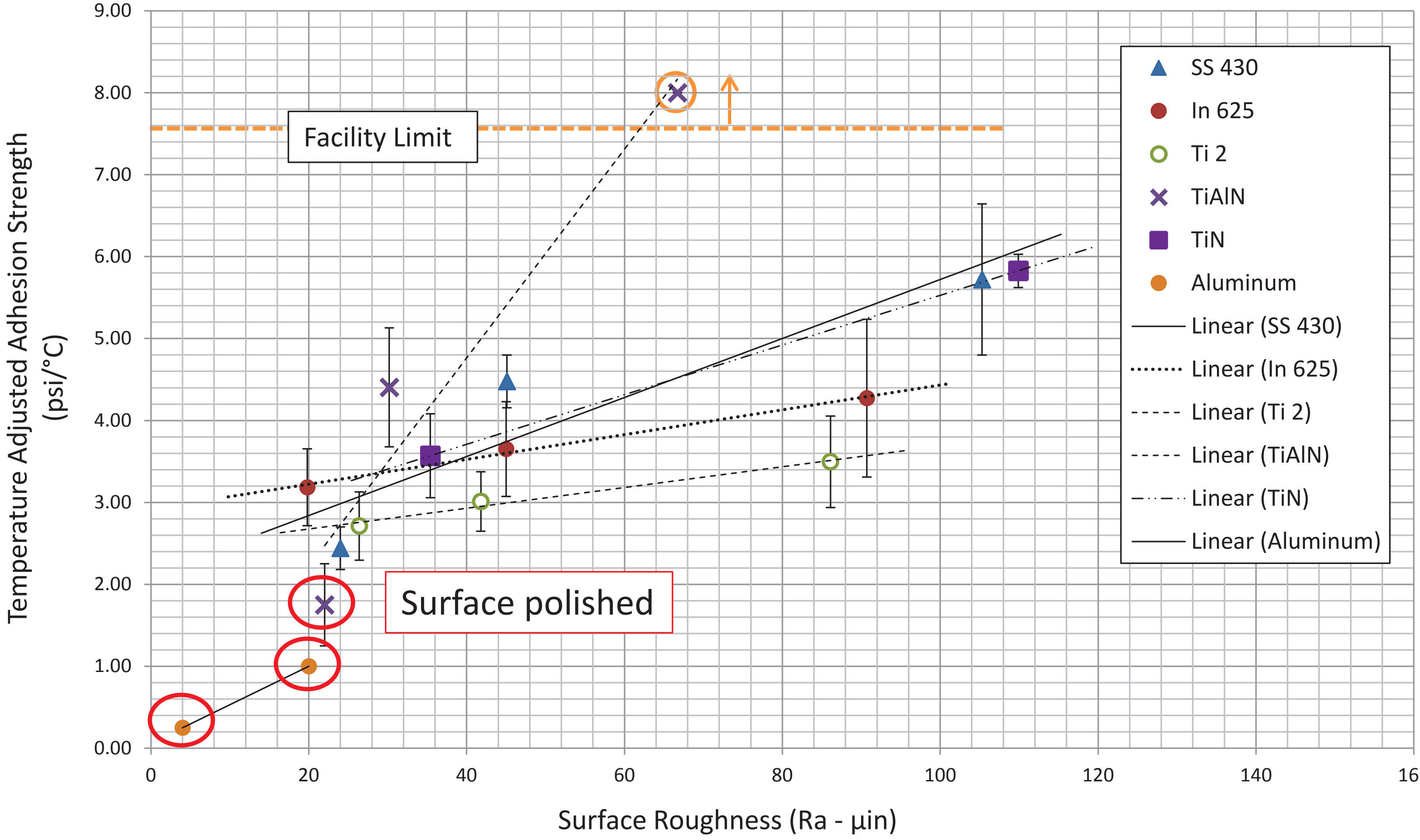
PENNSTATE
1 8 5 5
Aerospace Engineering



- Extrapolation predicts to within standard deviation of experiment

Ice Adhesion Comparison

PennState AERTS

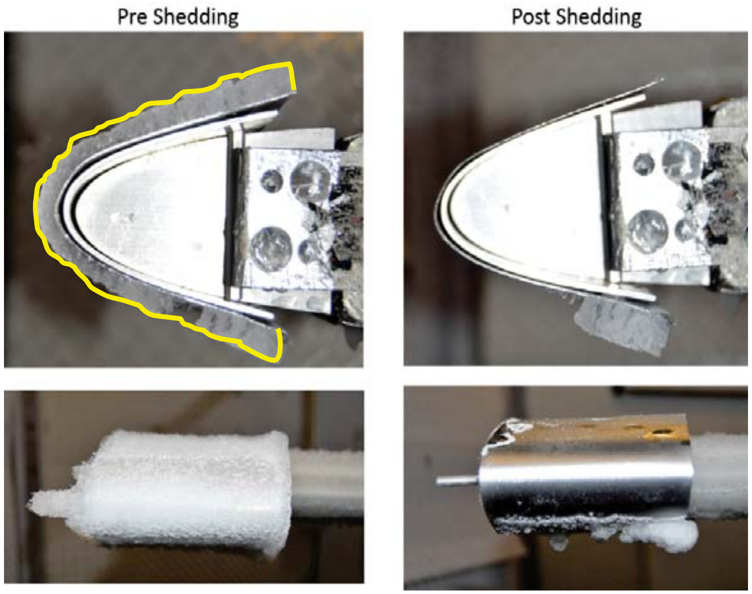


- Un-optimized coatings have higher adhesion strength than uncoated material
- Low surface roughness decreases ice adhesion…

Ice Adhesion Comparison

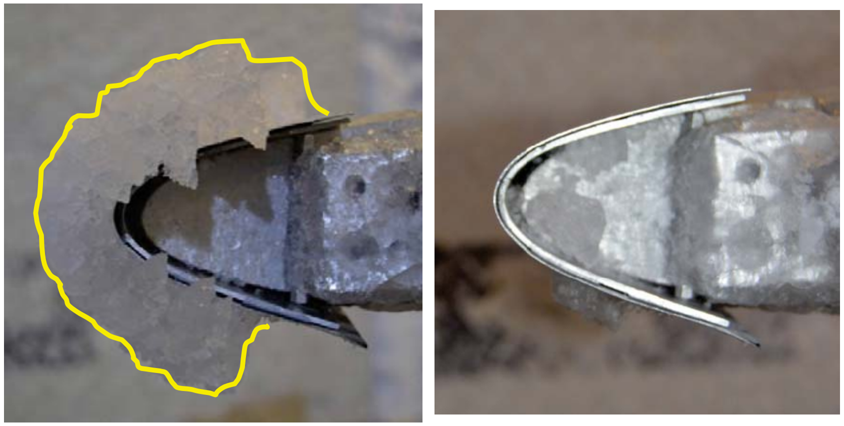
PennState AERTS

Aluminum 6061


Pre Shedding

Post Shedding

50 nm Ra @-8°C

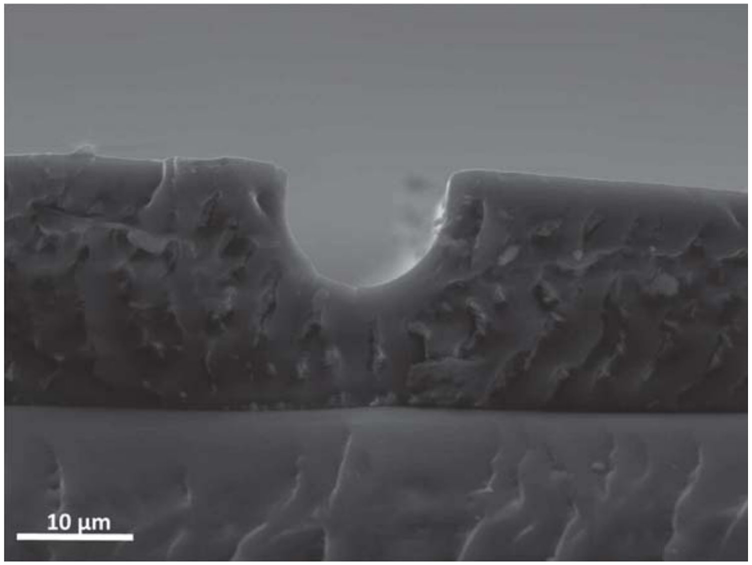

1700 nm Ra @ -8°C

Surface Roughness Effect: Further Exploration

•Four (4) coatings tested (polymer epoxy coat):

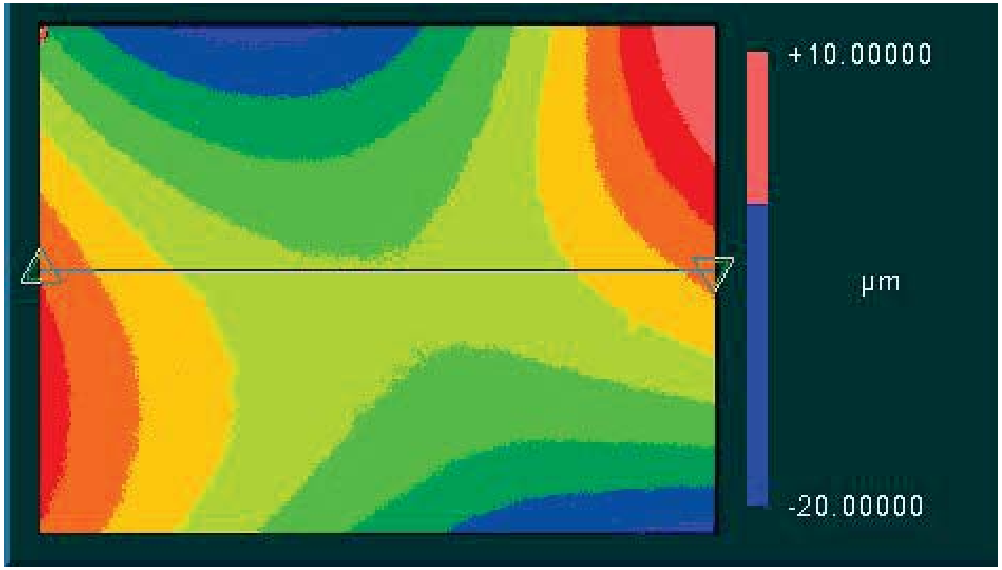
- Ra is the roughness average, or the arithmetic average of absolute values:
- $$R_a = \frac{1}{n}\sum_{i=1}^{n} |y_i|$$
- Baseline: Nanometer scale smooth coating (Ra 10 nanometer)
- Three (3) Slotted coatings: Valley carved coatings

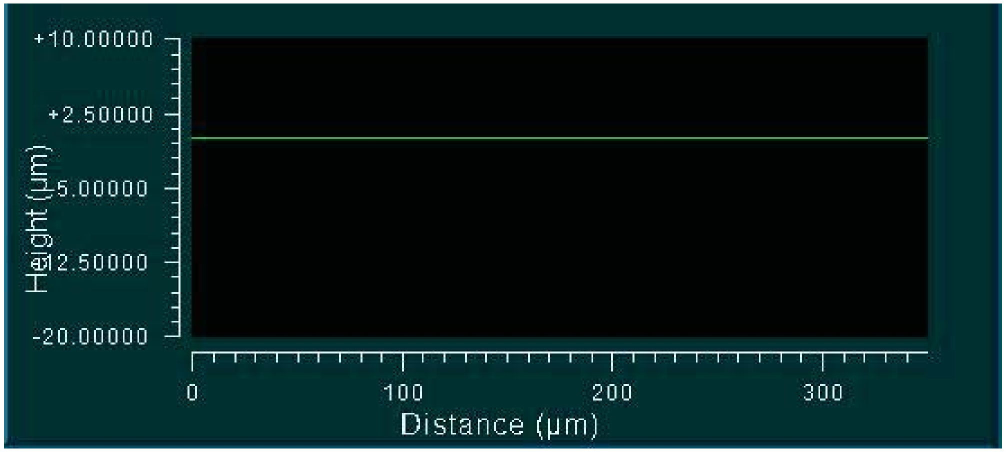
Laser Ablation Level	Ra (um)
0.35 W	1.13
0.6 W	1.95
1.2 W	5.11



Baseline Epoxy

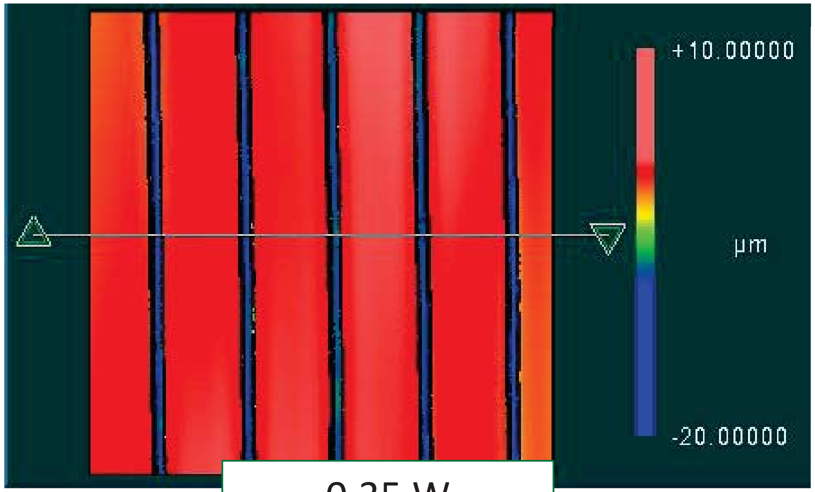
- Epoxy was applied to AL 3003 to create a coating with Ra≅0.
- After the baseline was tested, the coating was ablated with differing intensities of a laser to create a controlled surface topography.



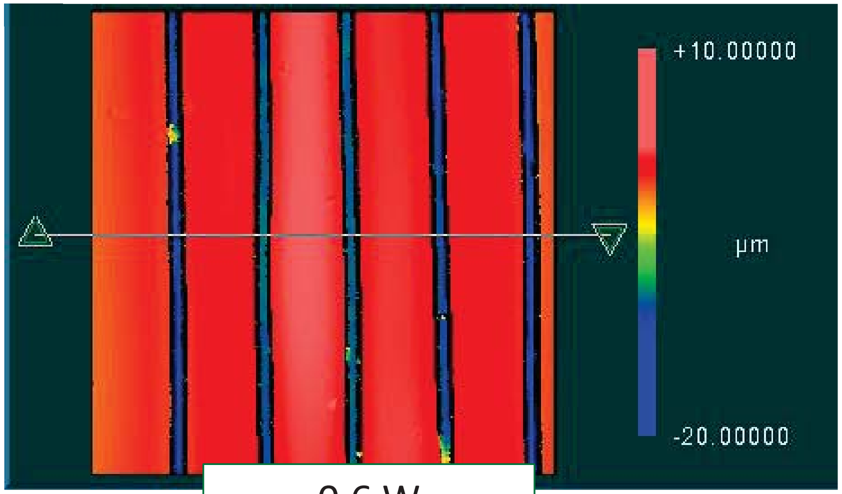


Varying Laser Ablation Intensities Create Varying Topographies

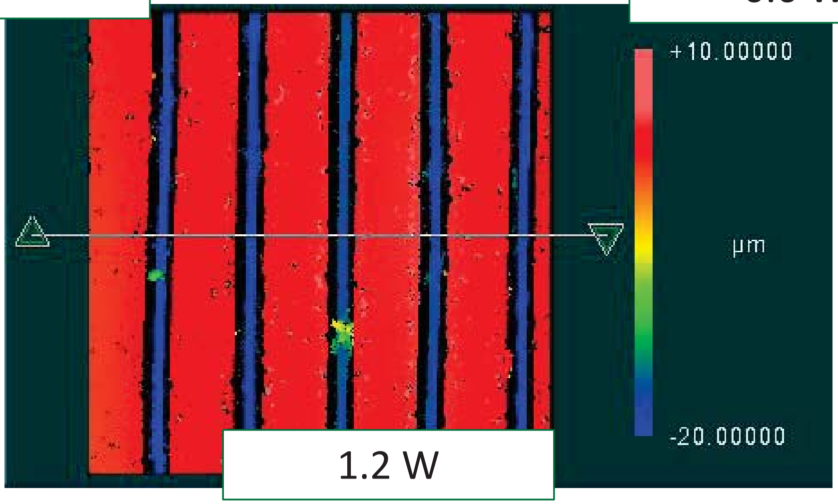
PennState AERTS



0.35 W

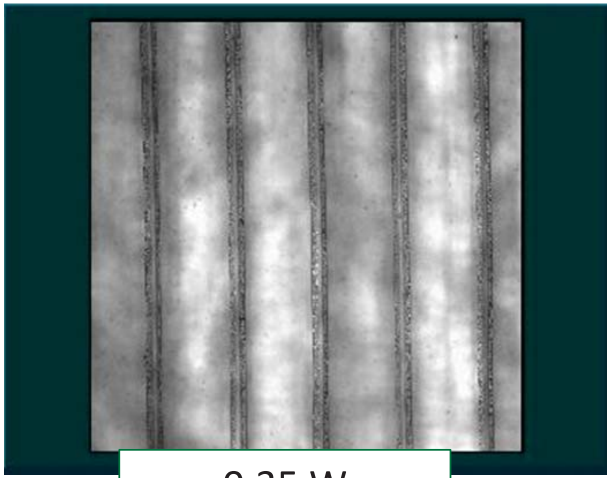


0.6 W

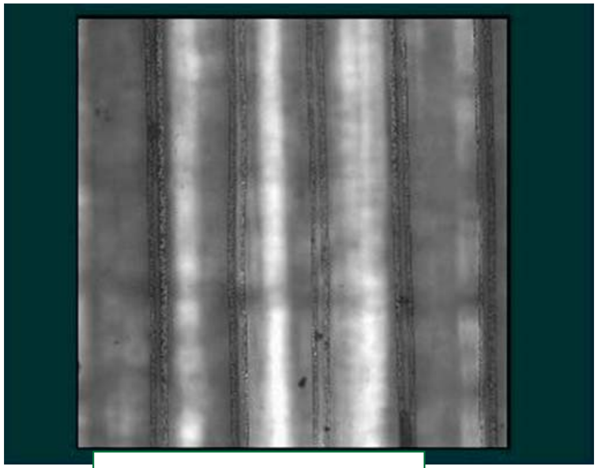


1.2 W

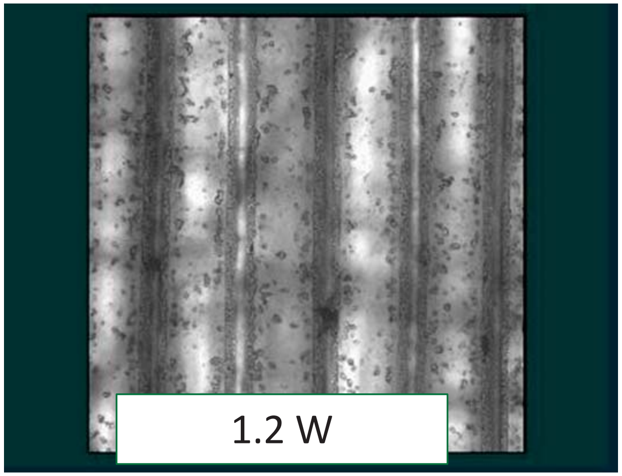
Varying Laser Ablation Intensities Create Varying Topographies



0.35 W

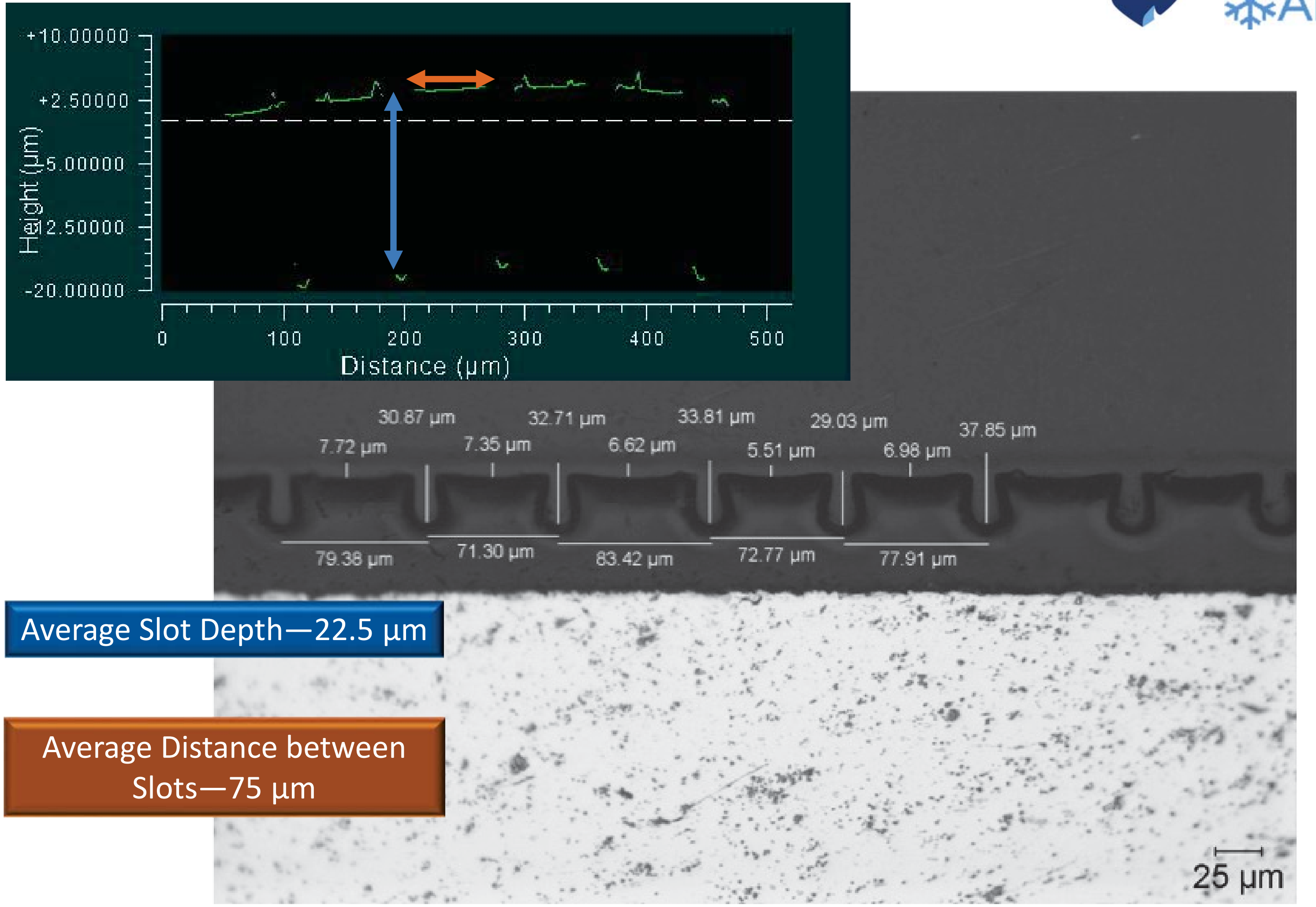


0.6 W



1.2 W

1.2 W Ablated Sample

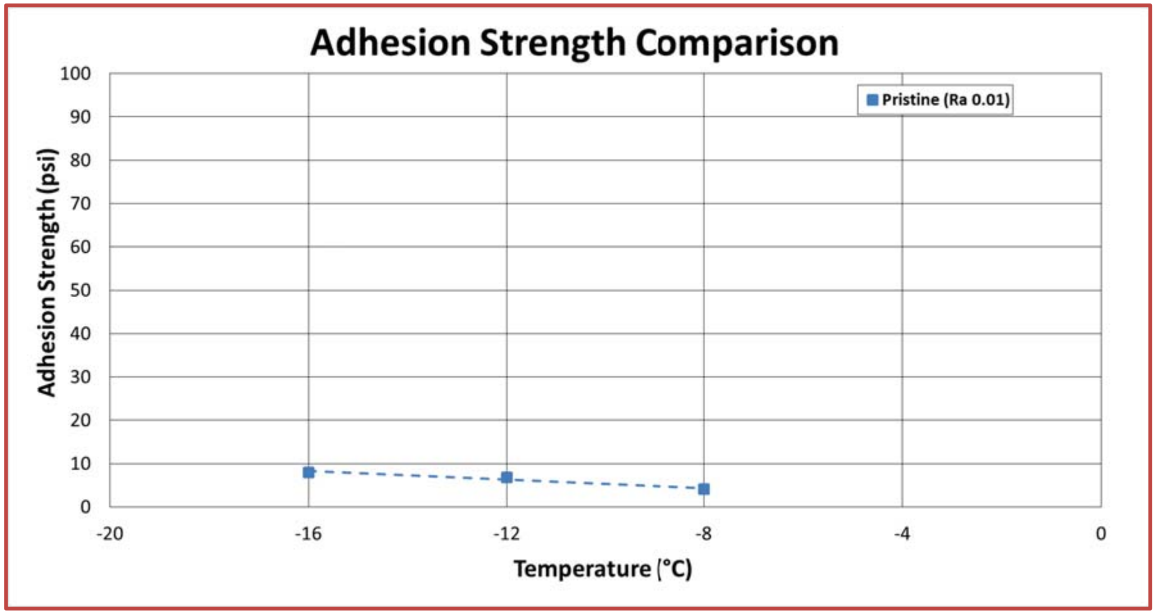


Average Slot Depth—22.5 µm

Average Distance between Slots—75 µm

Results for Varying Surface Substrates

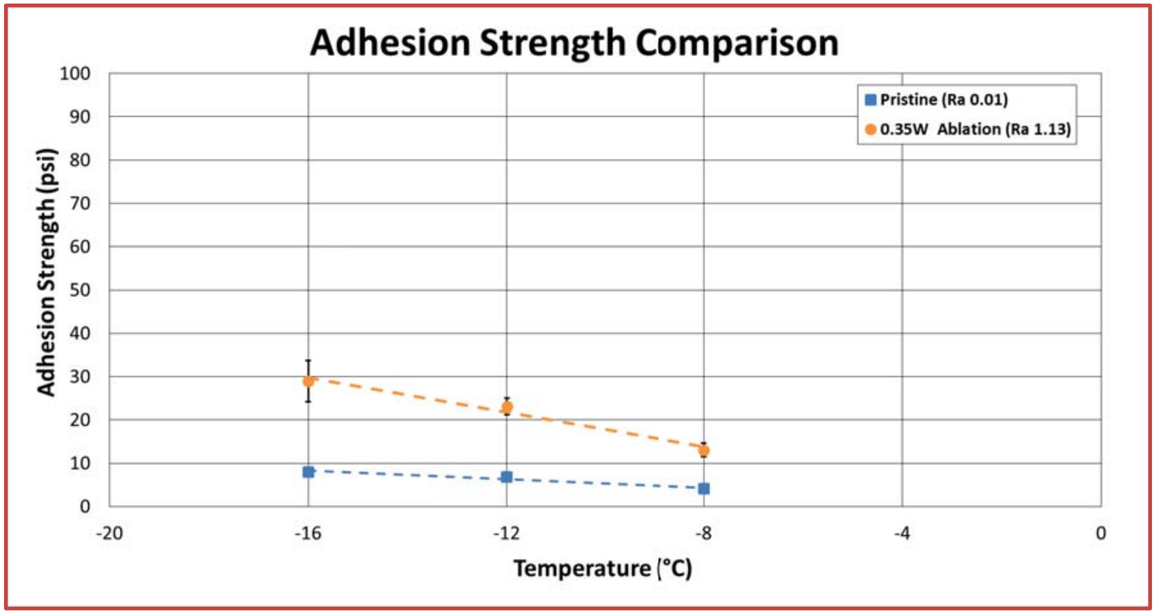
PennState
AERTS

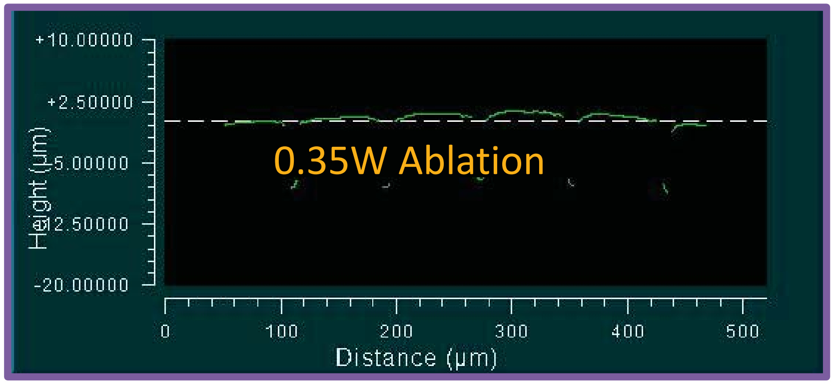


No Ablation

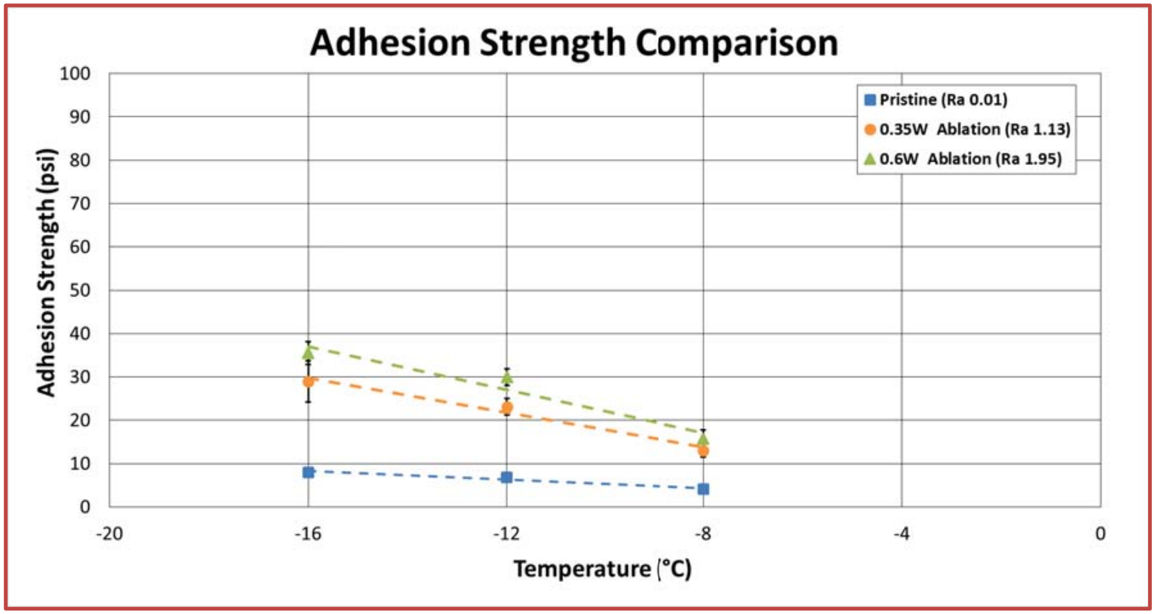
Results for Varying Surface Substrates

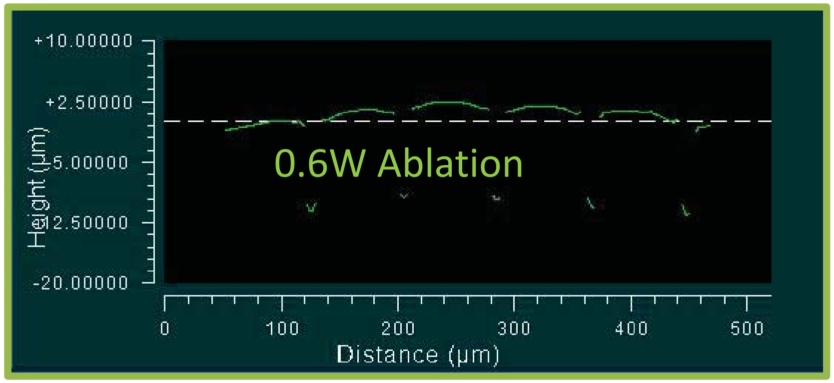
PennState AERTS





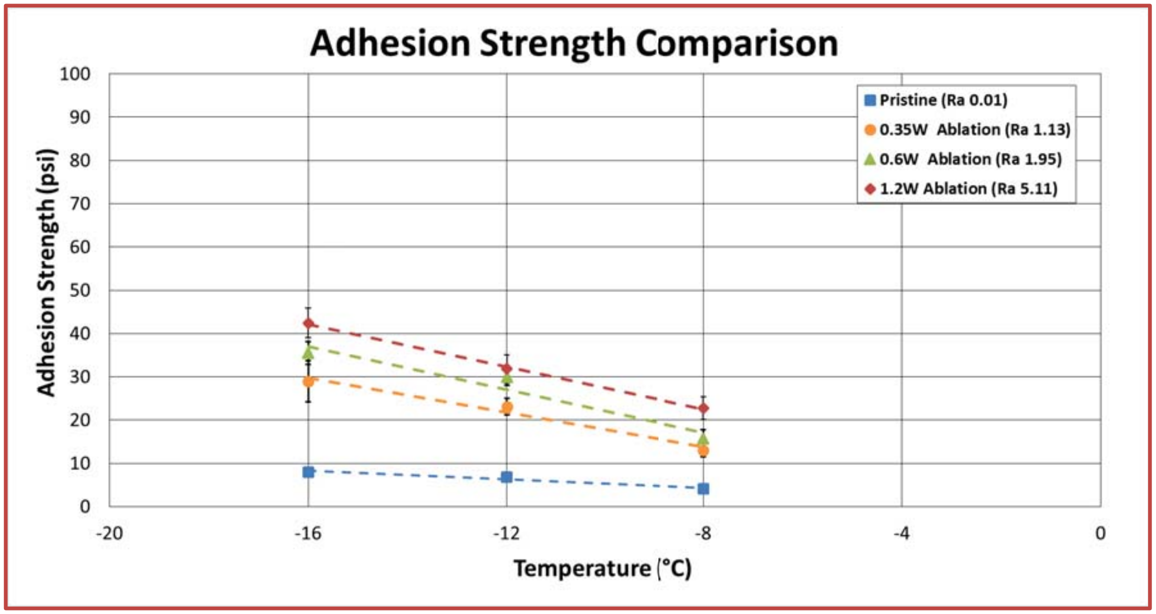
Results for Varying Surface Substrates

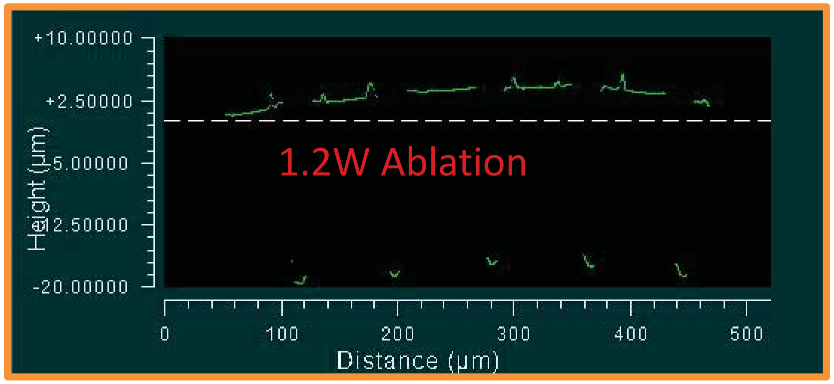




Results for Varying Surface Substrates

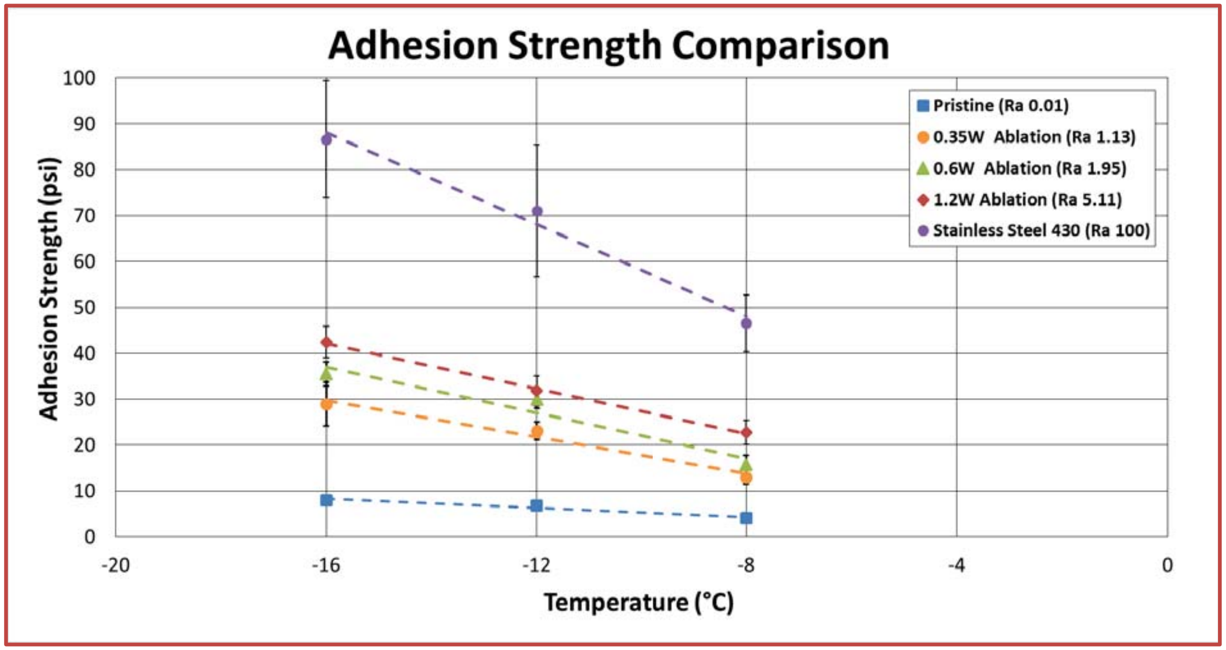
PennState AERTS





Results for Varying Surface Substrates

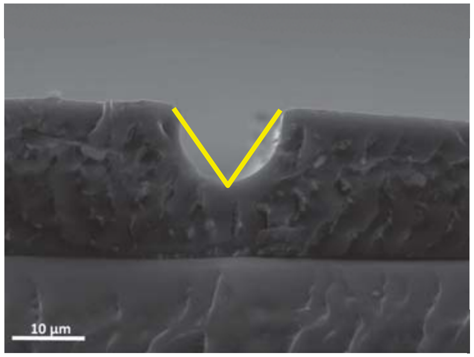
PennState
AERTS

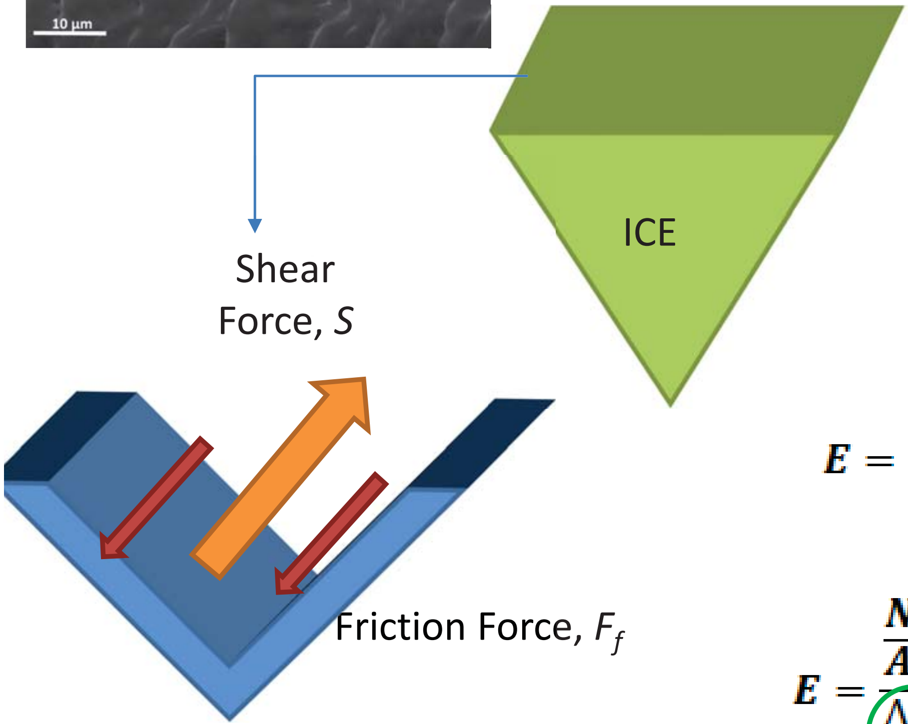


Increased Surface Roughness Corresponds to Increased Adhesion Strength

Modeling based on Surface Roughness?

PennState AERTS

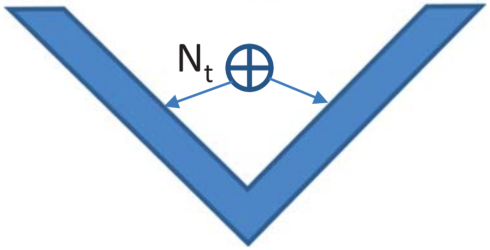




$$-S + F_f + F_f = 0$$

$$F_f = \mu N_t$$

$$S = 2\mu N$$



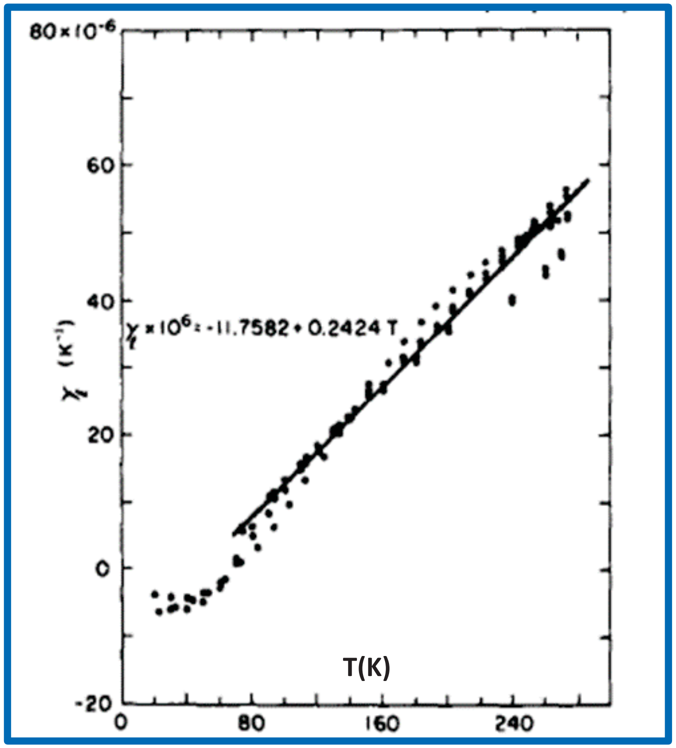
$$E = \frac{\sigma}{\varepsilon}$$ Stress strain relationship

$$E = \frac{\frac{N_t}{A_0}}{\boxed{\frac{\Delta L}{L_0}}} = \frac{N_t L_0}{A_0 \Delta L}$$

Thermal Coefficient of Thermal Expansion

$$\alpha = \frac{\frac{\Delta L}{L_0}}{\Delta T}$$

Temperature Dependency

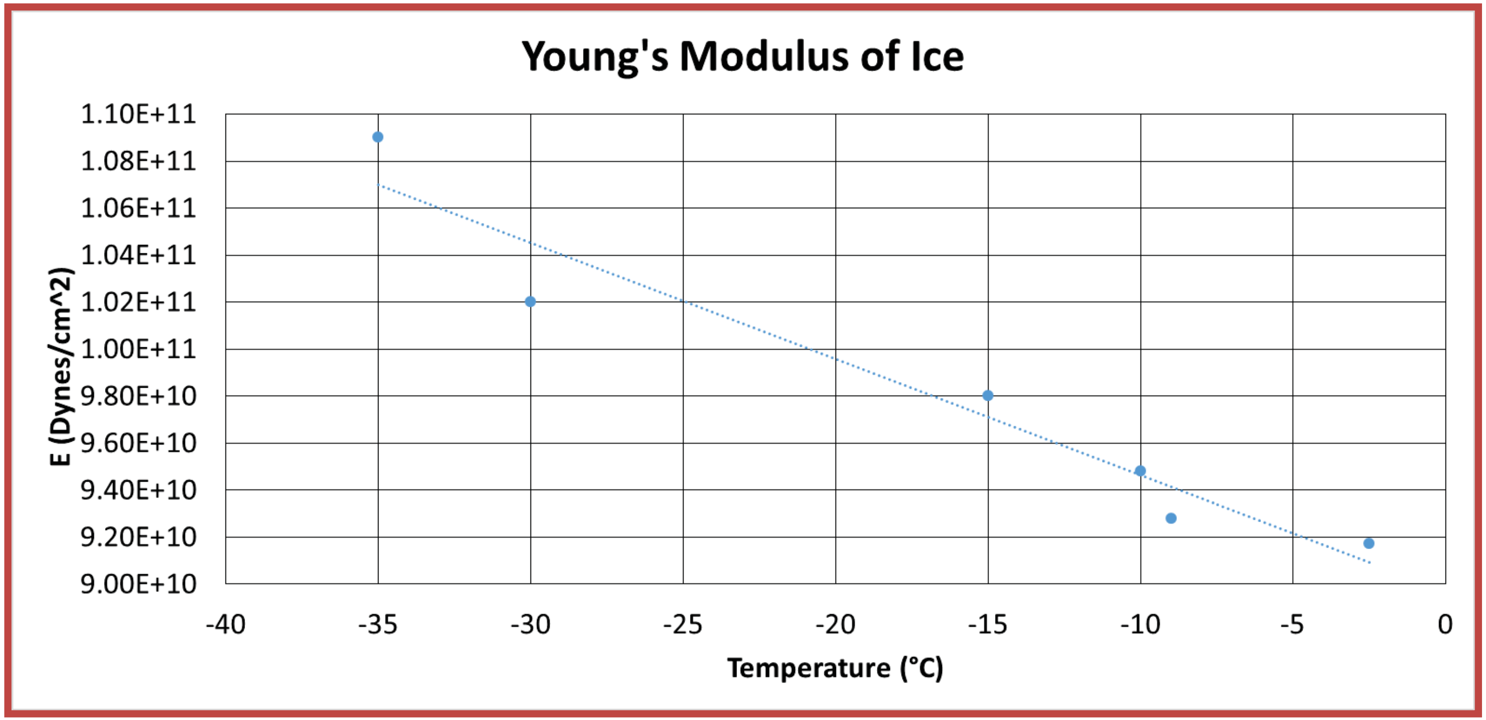


$$S = 2\mu E A_0 \boxed{\alpha} \Delta T$$

- **The Coefficient of Thermal Expansion for Ice is dependent on Temperature***

*Y. Yen, "Review of Thermal Properties of Snow, Ice, and Sea Ice," Vol. 81 Issue 10 CRREL Report, 1981.
†T. Northwood, "Sonic Determination of the Elastic Properties of Ice," Canadian Journal of Research Vol. 25, Sec A, 1947.

Temperature Dependency



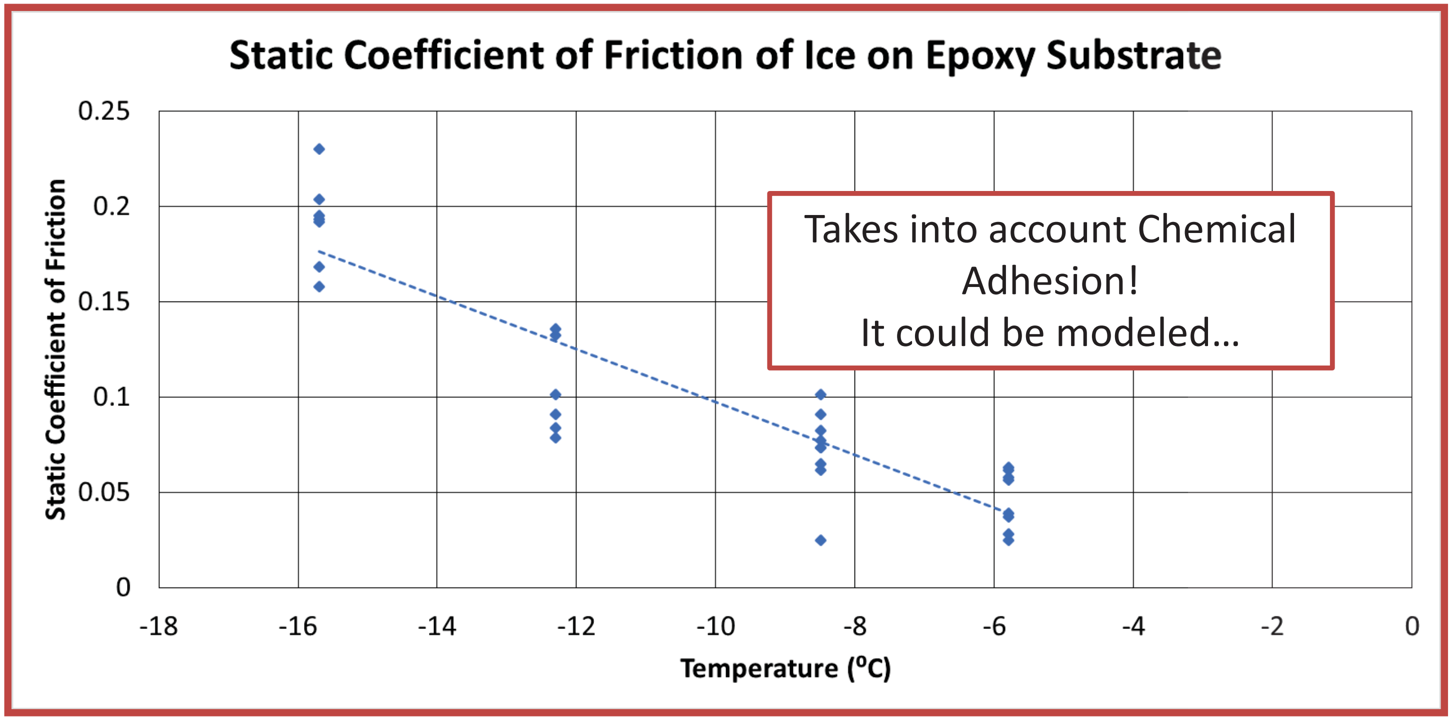
$$S = 2\mu \boxed{E} A_0 \alpha \Delta T$$

- **Young’s Modulus of Ice (Sea) is dependent on Temperature†**

*Y. Yen, “Review of Thermal Properties of Snow, Ice, and Sea Ice,” Vol. 81 Issue 10 CRREL Report, 1981.

†T. Northwood, “Sonic Determination of the Elastic Properties of Ice,” Canadian Journal of Research Vol. 25, Sec A, 1947.

Temperature Dependency


Static Coefficient of Friction of Ice on Epoxy Substrate

Takes into account Chemical Adhesion!
It could be modeled…

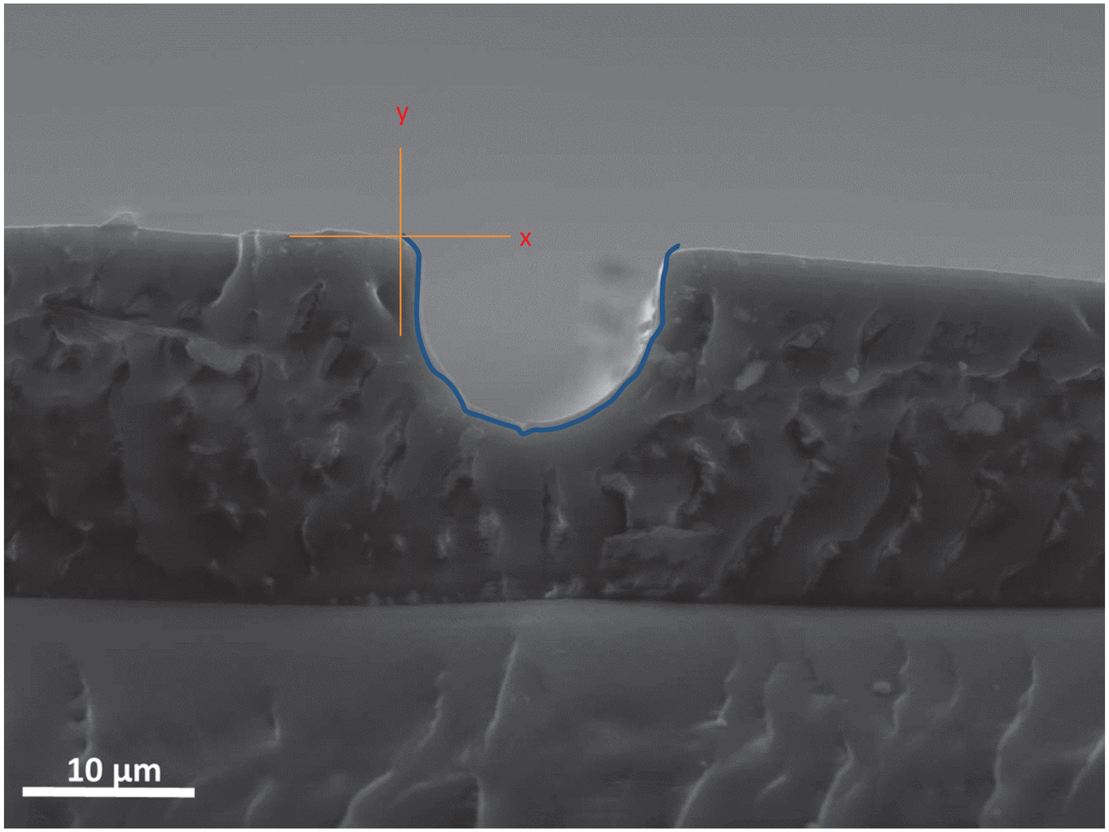
$$S = 2\boxed{\mu} E A_0 \alpha \Delta T$$

- **Coefficient of Static Friction also dependent on temperature, and measured in the AERTS facility.**

*Y. Yen, “Review of Thermal Properties of Snow, Ice, and Sea Ice,” Vol. 81 Issue 10 CRREL Report, 1981.
†T. Northwood, “Sonic Determination of the Elastic Properties of Ice,” Canadian Journal of Research Vol. 25, Sec A, 1947.

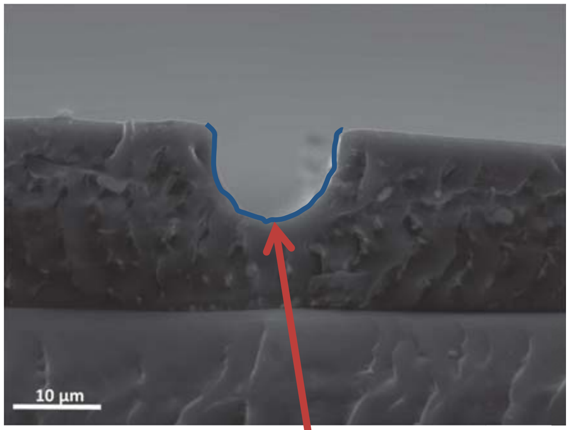
Ablation Digitization

- 1. Obtain cross-sectional view using Scanning Electron Microscopy (SEM)
- 2. Digitize ablated surface
- 3. Input (x,y) coordinates into computer model

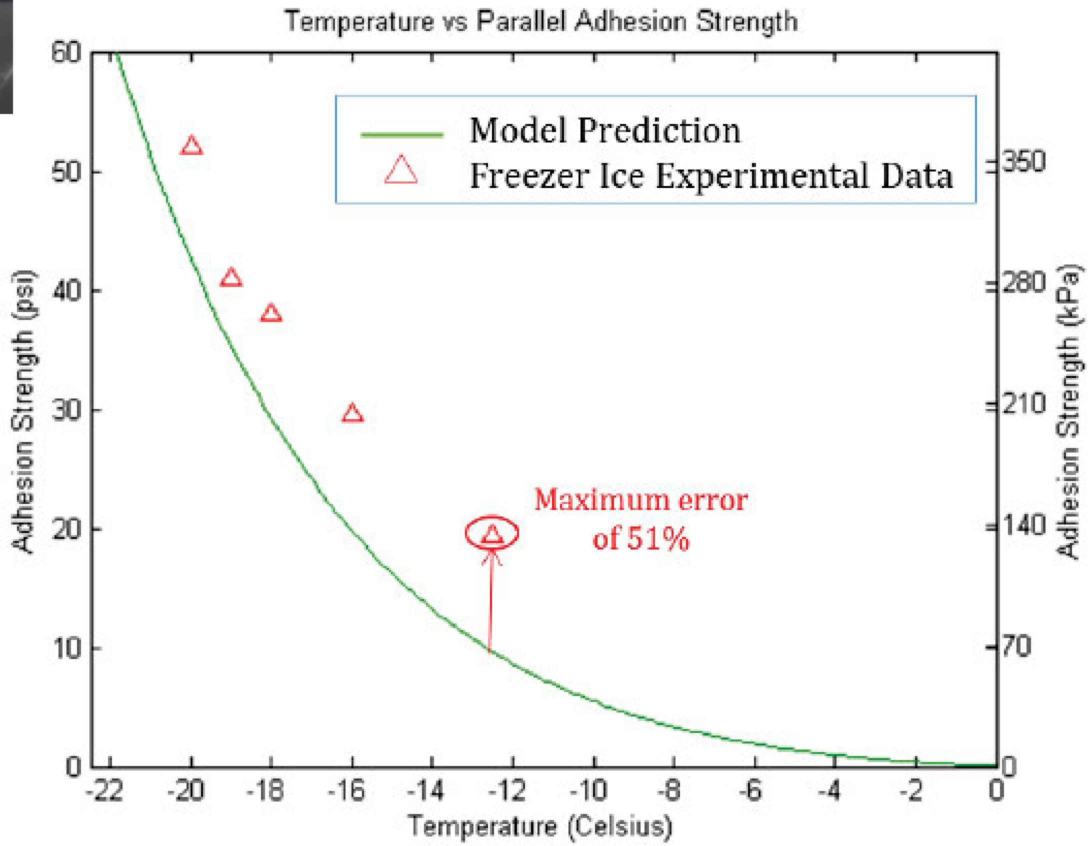


Results

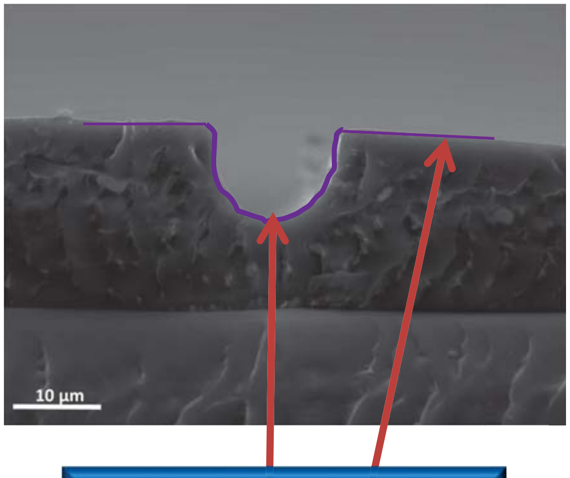
PennState AERTS



The digitized data is input into Matlab, and the slope is calculated between every point

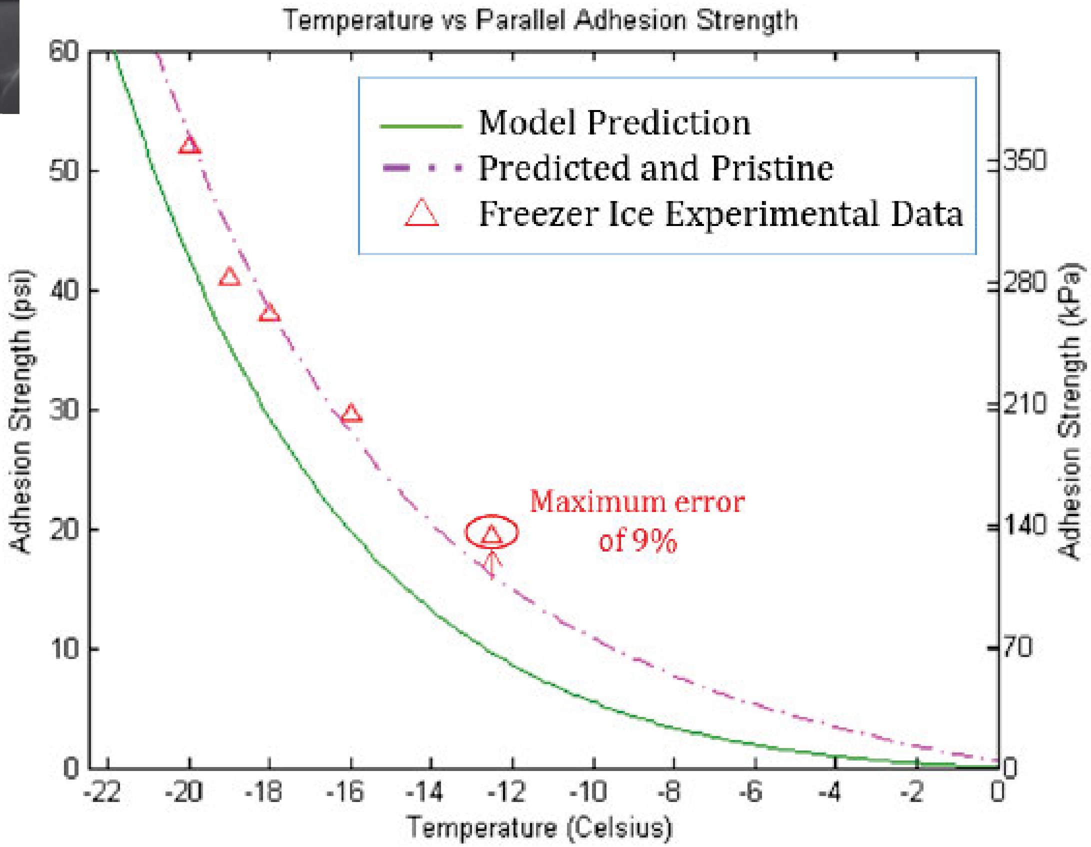


Results



Adhesion Strength is calculated for the valley and plateaus

An average error reduction to less than 9%



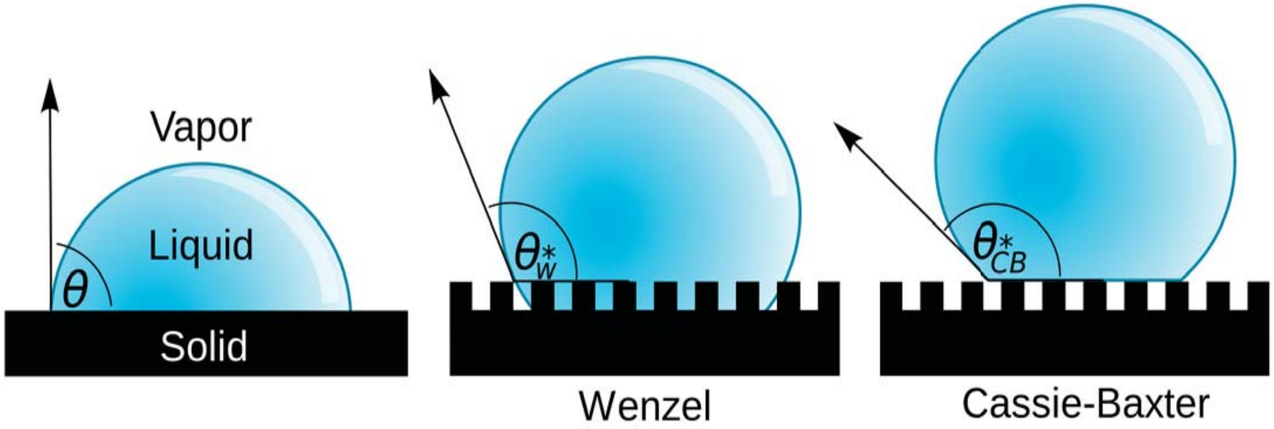
Presentation Outline

PennState AERTS

- Background & Objectives
- Testing Facility & Procedure
- Results
  - Material and Icing Parameter Effects
  - Evaluation of erosion resistant materials
  - Evaluation of superhydrophobic materials
- Conclusions

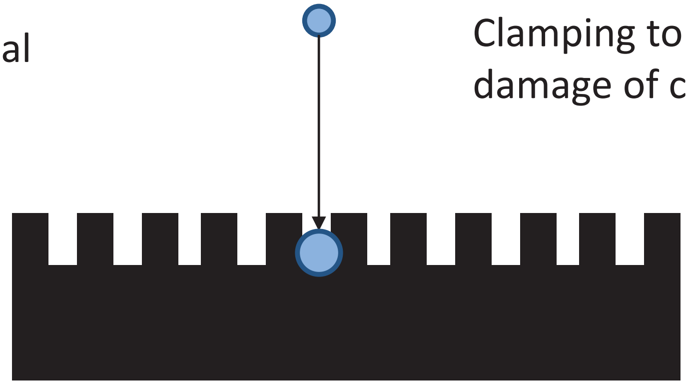
Superhydrophobic Test Results

Hoe do Hydrophobic coatings work?



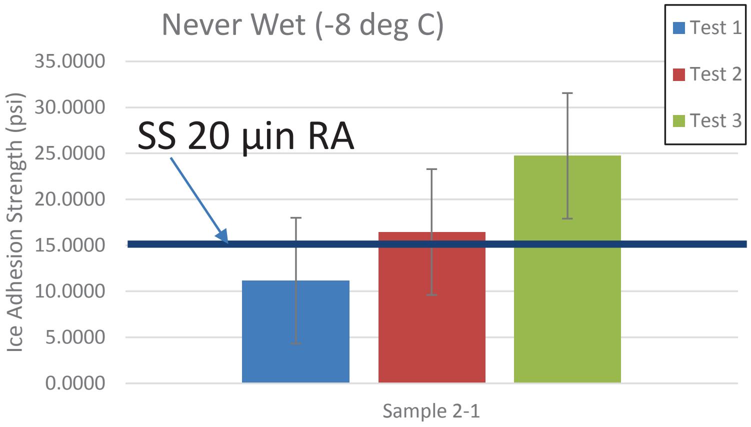
Aircraft Icing:
10 to 50 µm typical

Clamping to substrate, potential damage of coating

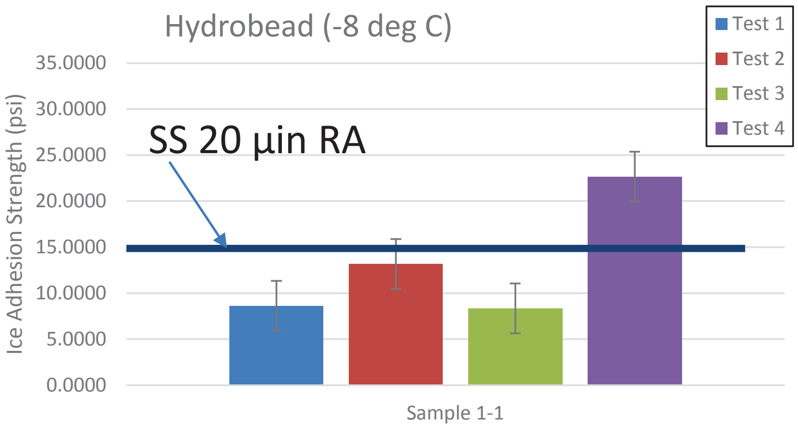


Superhydrophobic Test Results

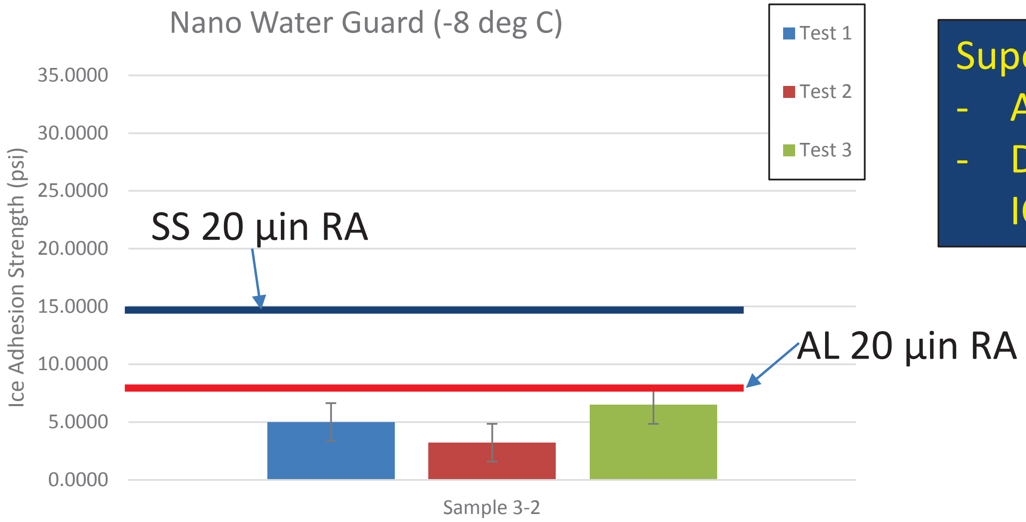
PennState AERTS



Never Wet (-8 deg C)



Hydrobead (-8 deg C)

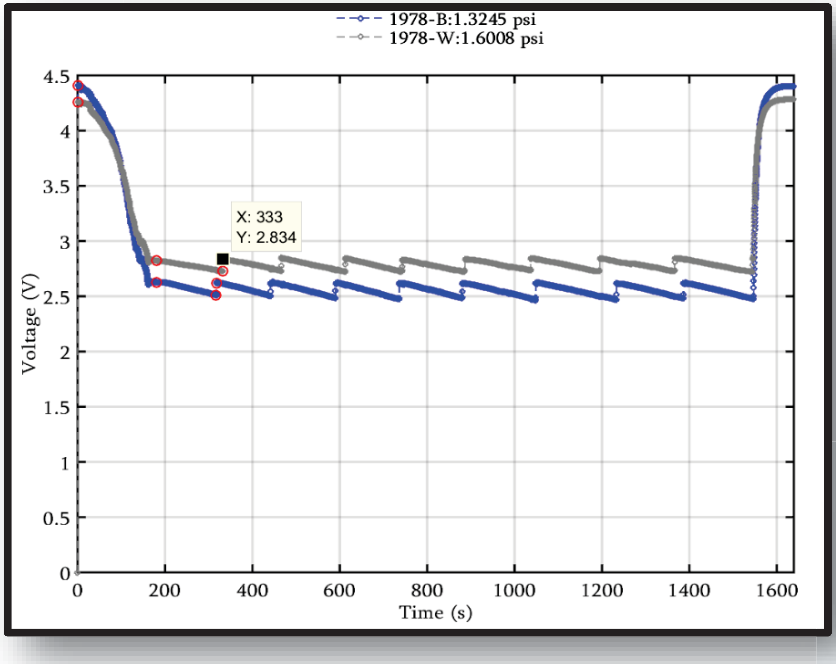


Nano Water Guard (-8 deg C)

Superhydrophobic coatings:

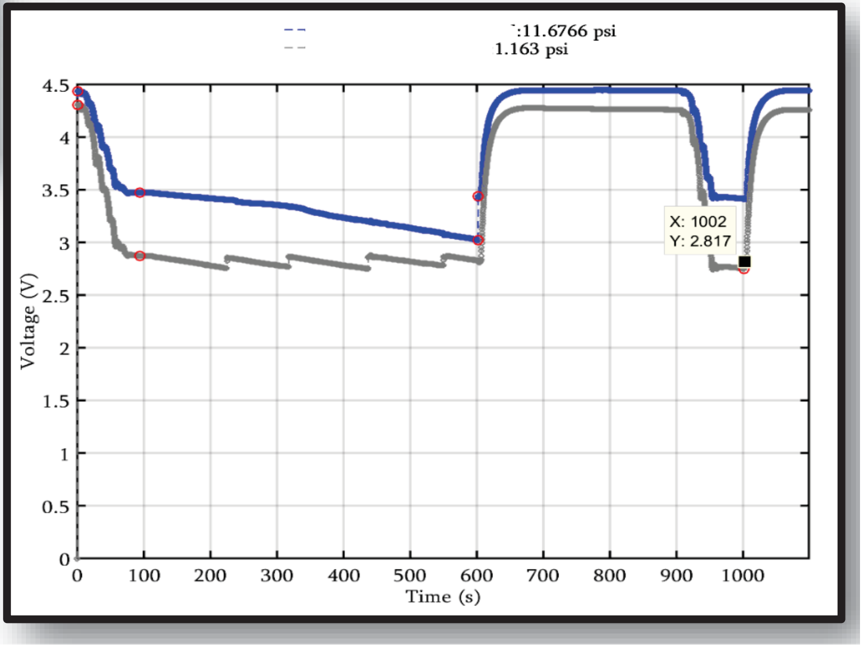
- ARE NOT ICE PROTECTIVE
- DEGRADE WITH CONSECUTIVE ICE SHEDDING EVENTS

Example of Ice Protective Coating



-12°C, 70 m/sec, 20 µm, 2 g/m^3

~1.5 psi at -12°C



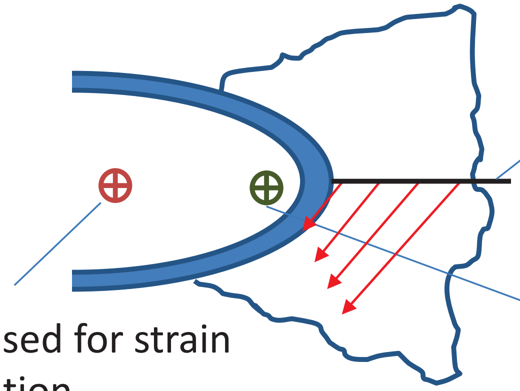
PennState AERTS

New Testing Technique Being Explored

Issues with the Technique

PennState AERTS

- Hand traced areas are not ideal: Discrepancies of 5% between readings
- Large ice accretions of varying thickness (i.e. on airfoils) introduce bending moments at the ice interface
- Large ice accretion displaces the center of gravity of the beam bending system, and the calculation of the load requires knowledge of the ice thickness to re-calculate the location of the center of gravity
- This effects are small for “ice protective coatings” but could provide shear ice adhesion strength values up to 30% for eroded surfaces.



Complex bending moment is hard to account for due to ice shape

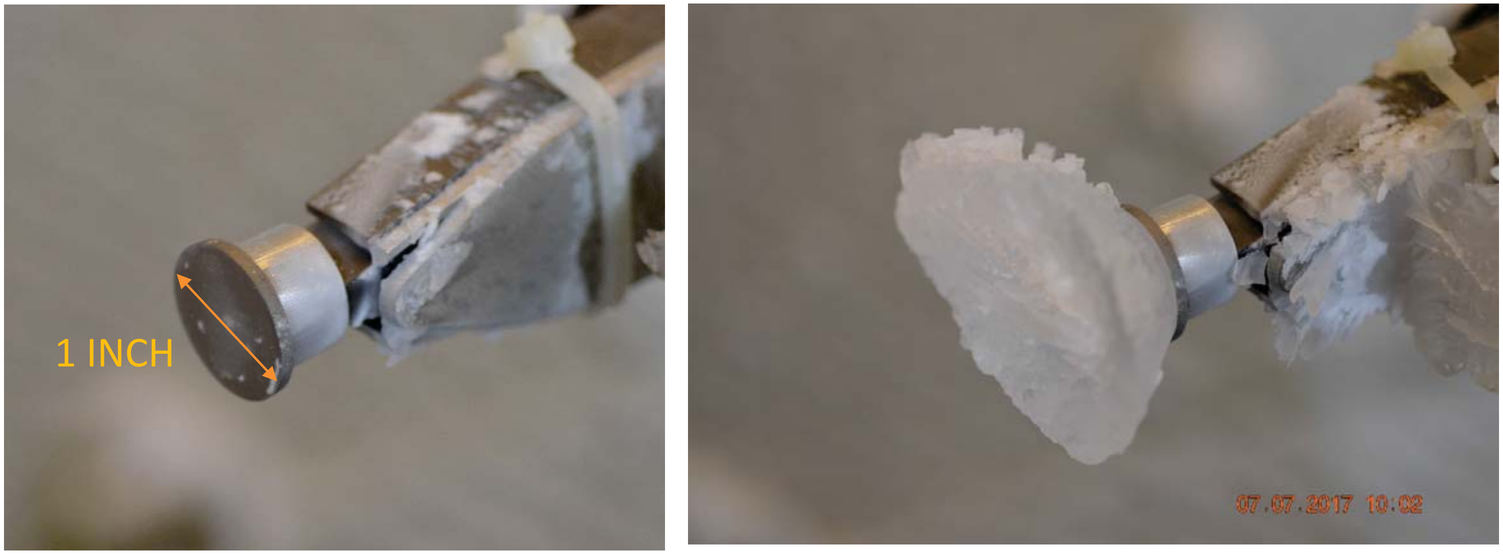
Original CG used for strain gauge calibration

New CG after ice accretion

Flat Surface Ice Accretion

PennState
AERTS

- Prevents ice bridging
- Sheds full surface (no need to measure)
- Provides similar ice adhesion strength values for equal icing conditions as airfoils shapes
- Eases taking into account CG motion due to ice accretion



Conclusions

1. The proposed rotor testing procedure is accurate with a standard deviation of less than 20% (consistent with other ice adhesion measurement techniques).

2. Erosion resistant materials must be optimized with low surface roughness to be effective ice icing conditions.
   - TiAlN has a 47% higher ice adhesion strength than the average of uncoated metallic materials
   - TiN has a 31% higher ice adhesion strength than the average of uncoated metallic materials

PENNSTATE

Aerospace Engineering

Conclusions

3. Ambient temperature and surface roughness are the two most influential parameters for impact ice adhesion strength.
   - 600% reduction in adhesion strength from -15 °C to -5 °C
   - 246% increase in adhesion strength from 24 µin *Ra* to 105 µin *Ra*

4. It is possible to extrapolate adhesion strength over ambient temperature for a given surface roughness for metallic substrates.

3. Modeling ice adhesion strength by taking into account surface roughness/morphology is needed (not only chemical adhesion)

PennState
AERTS

Conclusions

Superhydrophobic coatings:

ARE NOT ICE PROTECTIVE

Questions?

Multiscale Modeling of Ice Adhesion

NASA Workshop on Low Ice Adhesion Materials
August 11, 2017

Dr. David Thompson, Mississippi State University
Professor and Airbus Helicopters, Inc. Professor
Department of Aerospace Engineering

MISSISSIPPI STATE UNIVERSITY

AIRBUS

Acknowledgements

Project team

- Dr. Dong Meng, MSU Swalm School of Chemical Engineering
- Dr. Jing Zhong, MSU Swalm School of Chemical Engineering
- Dr. Elmar Bonnacurso, Airbus Group Innovations (AGI)
- Amir Afshar, PhD student, MSU Swalm School of Chemical Engineering
- Randa Bassou, PhD student, MSU Department of Aerospace Engineering
- Alex Laroche, PhD student, AGI
- Vittorio Vercillo, PhD student, AGI

The support of NASA Glenn Research Center through the Advanced Air Transport Technology (AATT) Project is gratefully acknowledged.

Title: Multiscale, Physics-Based Modeling of Impact Ice Adhesion
Cooperative agreement: NNX16AN20A
NASA TM: Eric Kreeger

MISSISSIPPI STATE UNIVERSITY

AIRBUS

Outline of Presentation

1. Overview
2. Experimental Method
3. Ice Adhesion Stress: Empirical Model
4. Ice Adhesion Stress: Predictive Models
5. Summary

Outline of Presentation

1. **Overview**
2. Experimental Method
3. Ice Adhesion Stress: Empirical Model
4. Ice Adhesion Stress: Predictive Models
5. Summary

Multiscale Modeling of Ice Adhesion

Motivation

Goal: To facilitate prediction of icing mitigation on engineered surfaces using a physics-based, multiscale model for impact ice adhesion stress.

Current adhesive stress model in LEWICE - Wright (2002)

- Based on experimental data of Scavuzzo and Chu (1987)
- Large reported error (±100%)
- Based on limited parameter set (purely a function of temperature)

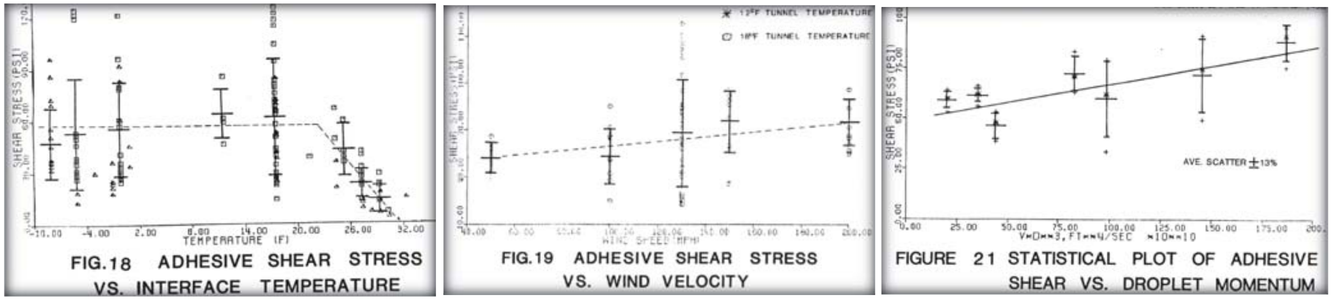


FIG.18 ADHESIVE SHEAR STRESS VS. INTERFACE TEMPERATURE

FIG.19 ADHESIVE SHEAR STRESS VS. WIND VELOCITY

FIGURE 21 STATISTICAL PLOT OF ADHESIVE SHEAR VS. DROPLET MOMENTUM

Scavuzzo and Chu (1987)

$$F_{AD} = \begin{cases} 14583(T_{MP} - T_S) & 267.7K \leq T_S \leq T_{MP} \\ 34475(1.5(267.3 - T_S) + 4) & 258.15K \leq T_S \leq 267.7K \\ 34475(3.79(258.15 - T_S) + 12.5) & T_S \leq 258.15K \end{cases}$$

MISSISSIPPI STATE UNIVERSITY

AIRBUS

Background Summary

It is difficult to draw firm conclusions from previous studies due to inconsistencies in the data

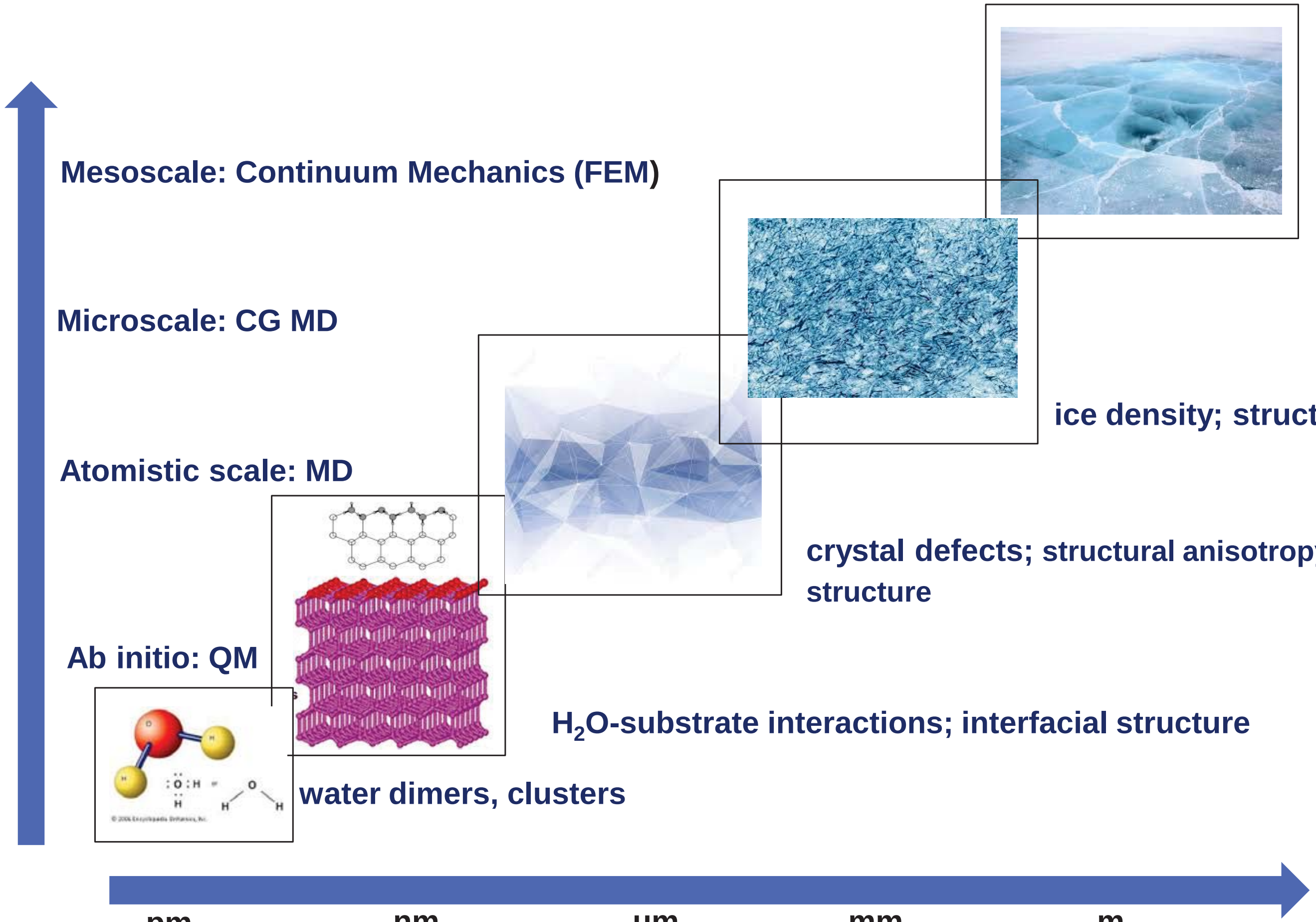
These inconsistencies demonstrate:

1. the challenges associated with accurate measurement of ice adhesion
2. the relatively poor understanding of the effects of traditional surface characterization parameters, e.g., contact angle, on ice adhesion.

Hypothesis: Attempting to identify correlations with other parameters might be an attractive alternative.

Multiscale Modeling of Ice Adhesion

Challenges of Predicting Impact Ice Adhesion



Mesoscale: Continuum Mechanics (FEM)

Microscale: CG MD

Atomistic scale: MD

Ab initio: QM

ice density; structure porosity

crystal defects; structural anisotropy; substrate structure

H_2O-substrate interactions; interfacial structure

water dimers, clusters

Multiscale Strategy for Predicting Impact Ice Adhesion

Currently, it is not feasible to predict the adhesive stress for an aircraft icing scenario based purely on first principles. Some type of modeling is necessary.

Observation: The adhesive stress is a function of parameters at multiple spatial and temporal scales

$$F_{AD} = f\left(\varphi_{m_1}, \dots, \varphi_{m_{N_m}}, \varphi_{\mu_1}, \dots, \varphi_{\mu_{N_\mu}}, \varphi_{n_1}, \dots, \varphi_{n_{N_n}},\right)$$

`

$\varphi_{m_n}(n = 1, N_m)$ represent the relevant macroscale parameters
$\varphi_{\mu_n}(n = 1, N_\mu)$, represent the relevant mesoscale parameters
$\varphi_{n_n}(n = 1, N_n)$ represent the relevant nanoscale parameters

This effort seeks to combine different methodologies (experimental and numerical) at multiple relevant scales to estimate this functional dependency.

When coupled with an ice accretion prediction code, such as LEWICE, such a relationship provides a pathway to á priori evaluation of the effectiveness of a surface designed for icing mitigation.

MISSISSIPPI STATE UNIVERSITY

AIRBUS

Objectives

1. Perform experiments to characterize impact ice adhesion on variety of surfaces over a range of icing conditions
2. Develop a purely empirical model to predict ice adhesion based on the experimental database
3. Develop a hybrid model to predict ice adhesion that incorporates both experimental measurements and numerical predictions
4. Lay the groundwork for a purely predictive multi-scale model for impact ice adhesion

Outline of Presentation

1. Overview
2. **Experimental Method**
3. Ice Adhesion Stress: Empirical Model
4. Ice Adhesion Stress: Predictive Models
5. Summary

EU Horizon 2020 project “Phobic2Ice“

PHOBIC2ICE
www.phobic2ice.com

Super-IcePhobic Surfaces to Prevent Ice Formation on Aircraft

Develop a new generation of icephobic surfaces to be used in aeronautic applications

Expected Results

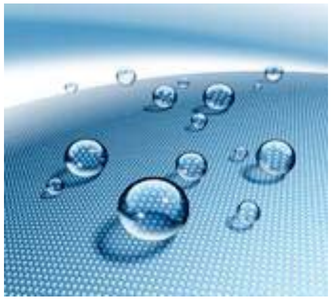
- List of use cases, substrate materials, definition of technical requirements and specifications
- Modelling and simulation of ice accretion
- Coating development
- Characterization and testing, development of ice detection sensors
- Coating application on prototype component & full-scale testing

Why it matters

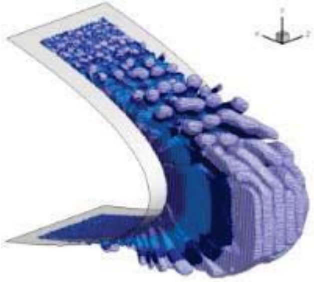
- Energy efficient anti-/de-icing systems
- Icing-detection sensors & early warning systems
- Ice accretion models depending on material properties

SoA & Gaps – Used & developed technologies

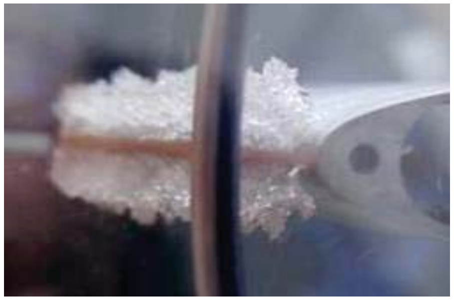
- Numerical Ice accretion tools need constant amelioration. No satisfactory tool exists combining aerodynamics, thermodynamics, and material properties
- No fabrication technology for obtaining a durable, erosion resilient icephobic coating is currently existing



Coating Development



Icing Simulations



Wind Tunnel Testing

MISSISSIPPI STATE UNIVERSITY

AIRBUS

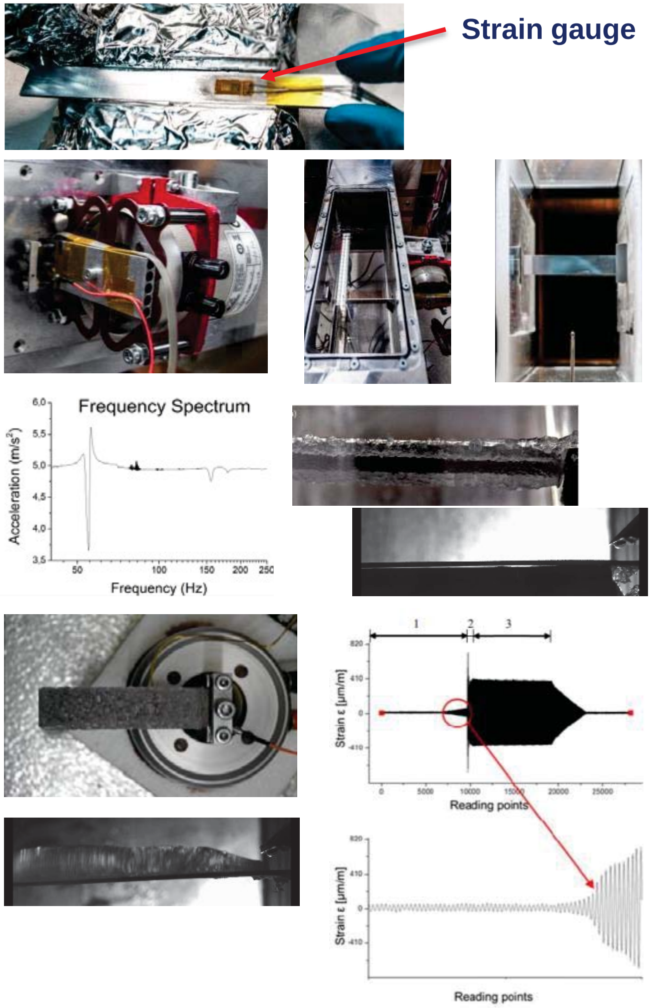
Measurement of Impact Ice Adhesion

Test Method & Protocol

Excitation of ice-metal composite beam with a sinusoidal stimulus by an electromagnetic (EM) shaker

- Bond strain gauge to back of cantilever
- Fix bare or coated cantilever to head of EM shaker, cantilever has one face exposed to airstream in test section
- Determine resonance frequency of bare cantilever by performing a resonance sweep with EM shaker
- Turn on airstream, cooling, and icing cloud and let ice accrete on exposed face of cantilever at set icing condition
- Turn off fan, cooling, and icing cloud for avoiding interferences with measurement
- Increase vibration amplitude of cantilever until ice layer debonds from the surface
- Read strain gauge measurement to determine interfacial shear stress at time of debonding

Advantage: *in situ* ice adhesion measurement



MISSISSIPPI STATE
UNIVERSITY™

Measurement of Impact Ice Adhesion

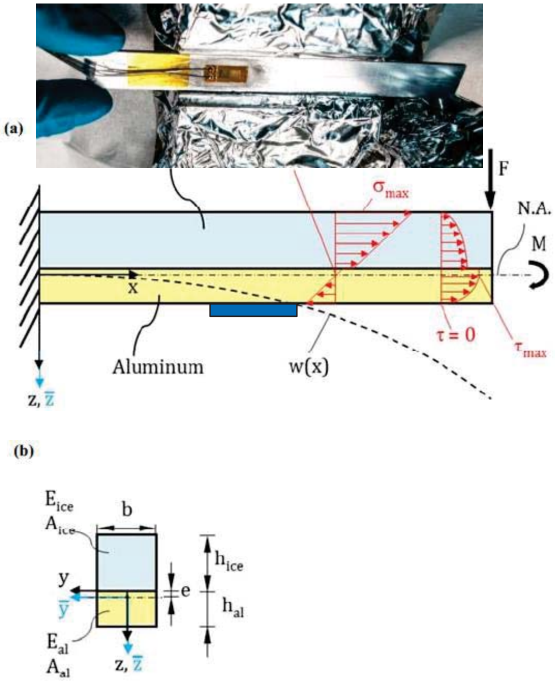
Model employed

Maximum adhesion shear strength corresponds to the reading from the strain gauge signal at the end of stage 1; it represents the maximum shear stress at interface ice/substrate just before ice debonding

Interfacial shear stress of cantilever, τ_{int}, is calculated by

$$\tau_{int} = \frac{\varepsilon_{EF-al} E_{ice} (h_{ice}^2 + 2h_{ice}|e|)}{2(x-l)(h_{cl}-|e|)}$$

- ε_{EF-al}: strain measured by the strain gauge
- x: distance between center of strain gauge and fixed end of cantilever
- l: total length of composite beam
- h_{ice}, h_{al}: thickness of ice layer and of cantilever
- E_{ice}: Young's modulus for ice
- e: eccentricity (function of E_{ice} and E_{al})



Preliminary Results

Samples

Sample No.	Material	Surface Treatment	Water CA	Water RoA	Surface Roughness, R_a / R_z (μm)
3	Al 2024	Polished + TSA[1]	60°	>90°	0.01 / 0.16
1	Al 2024	Polished + TSA + Episurf[2]	122°	>90°	0.02 / 0.16
0	Ti6Al4V	Anodized[3] + Episurf	155°	20°	0.58 / 4.40

Do the samples exhibit the same trend in ice adhesion over a range of freezing fractions?

[1] Tartaric Sulphuric Acid Anodizing

[2] Commercially available perfluoropolyether phosphonate compound in a HFO solvent

[3] TiO_2-Nanotube Layer

Preliminary Results

Samples

Sample No.	Material	Surface Treatment	Water CA	Water RoA	Surface Roughness, R_a / R_z (μm)
3	Al 2024	Polished + TSA[1]	60°	>90°	0.01 / 0.16
1	Al 2024	Polished + TSA + Episurf[2]	122°	>90°	0.02 / 0.16
0	Ti6Al4V	Anodized[3] + Episurf	155°	20°	0.58 / 4.40

Do the samples exhibit the same trend for ice adhesion over a range of freezing fractions?

[1] Tartaric Sulphuric Acid Anodizing

[2] Commercially available perfluoropolyether phosphonate compound in a HFO solvent

[3] TiO_2-Nanotube Layer

MISSISSIPPI STATE UNIVERSITY

AIRBUS

Preliminary Results

Test Matrix

Icing conditions with supercooled droplets	Total Air Temperature (TAT)	Airspeed	Liquid Water Content (LWC)	Mean effective droplet diameter (MVD)	Approx. Freezing Fraction
Ice type	(°C)	(m/s)	(g/m^3)	(µm)	
Rime	-20	50	0.3	20	1.00
Mixed	-20	50	0.8	20	0.55
Mixed	-5	50	0.3	20	0.7
Glaze	-5	80	1.0	20	0.2

The Icing time was sufficient to obtain the desired ice thickness.

MISSISSIPPI STATE
UNIVERSITY™

AIRBUS

Preliminary Results

Test Matrix

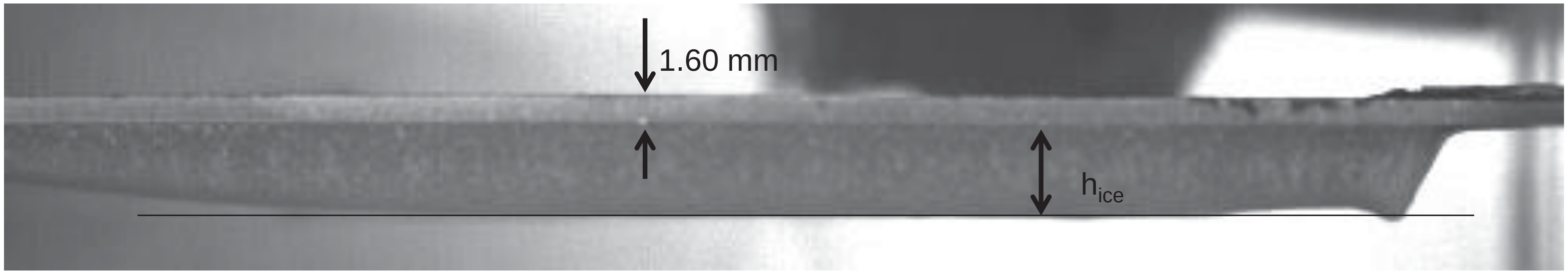
Icing conditions with supercooled droplets	Total Air Temperature (TAT)	Airspeed	Liquid Water Content (LWC)	Mean effective droplet diameter (MVD)	Approx. Freezing Fraction
Ice type	(°C)	(m/s)	(g/m^3)	(µm)	
Rime	-20	50	0.3	20	1.00
Mixed	-20	50	0.8	20	0.55
Mixed	-5	50	0.3	20	0.7
Glaze	-5	80	1.0	20	0.2

The Icing time was sufficient to obtain the desired ice thickness.

Preliminary Results

Test Matrix

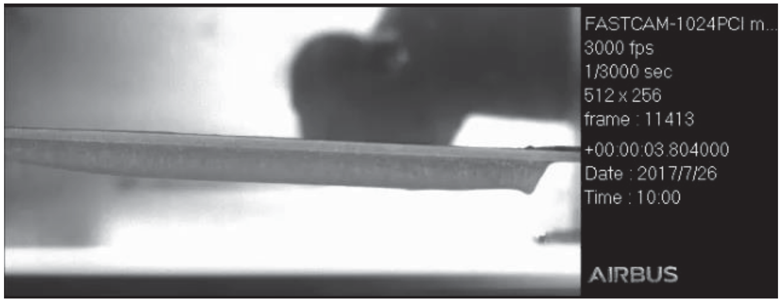
Icing conditions with supercooled droplets	Total Air Temperature (TAT)	Airspeed	Liquid Water Content (LWC)	Mean effective droplet diameter (MVD)	Approx. Freezing Fraction
Ice type	(°C)	(m/s)	(g/m³)	(µm)	
Mixed	-20	50	0.8	20	0.55
Mixed	-5	50	0.3	20	0.7
Glaze	-5	80	1.0	20	0.2



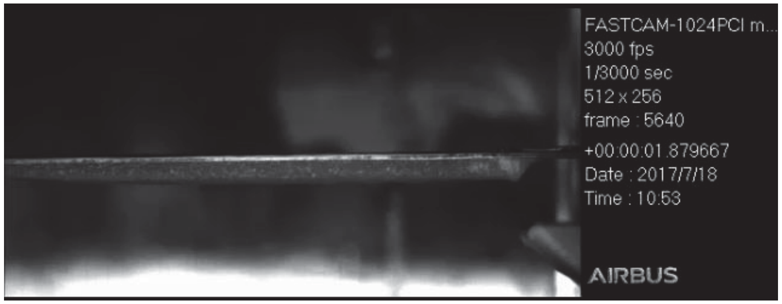
Preliminary Results

Test Matrix

Icing conditions with supercooled droplets	Total Air Temperature (TAT)	Airspeed	Liquid Water Content (LWC)	Mean effective droplet diameter (MVD)	Approx. Freezing Fraction
Ice type	(°C)	(m/s)	(g/m³)	(µm)	
Mixed	-20	50	0.8	20	0.55
Mixed	-5	50	0.3	20	0.7
Glaze	-5	80	1.0	20	0.2



Reference (Sample 3)



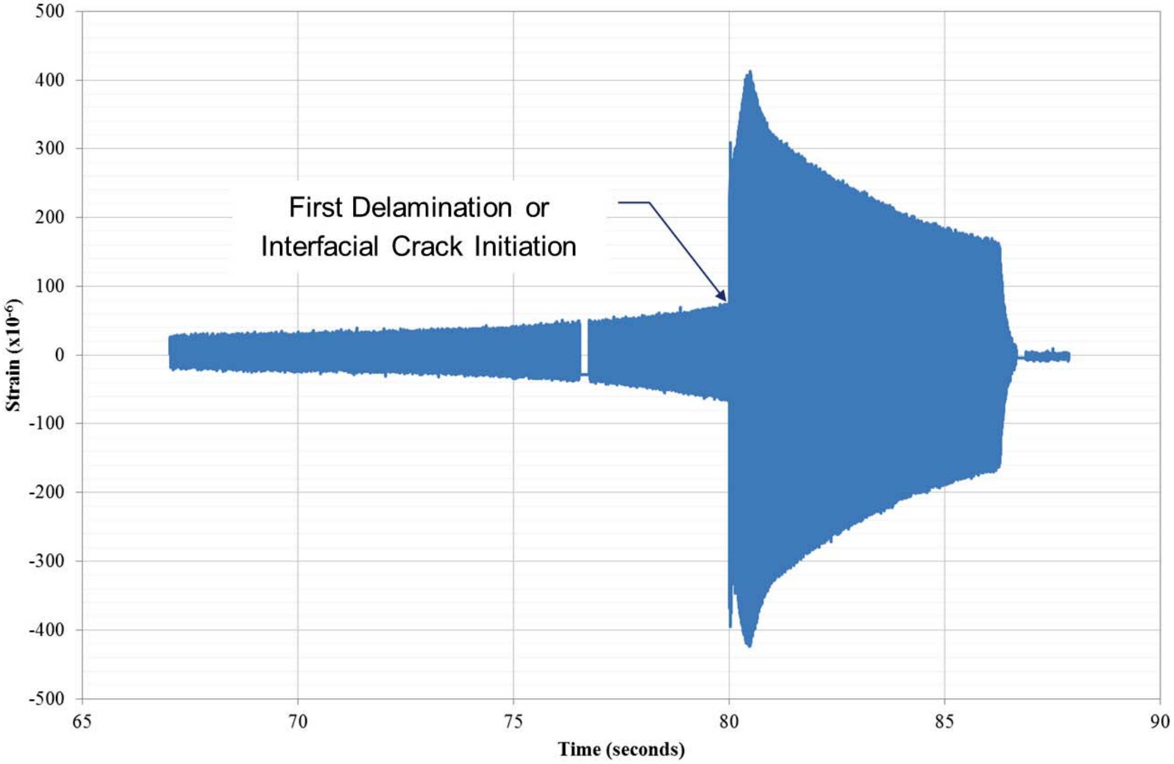
Superhydrophobic (Sample 0)

MISSISSIPPI STATE UNIVERSITY

AIRBUS

Preliminary Results

Strain gauge data analysis – identification of crack initiation

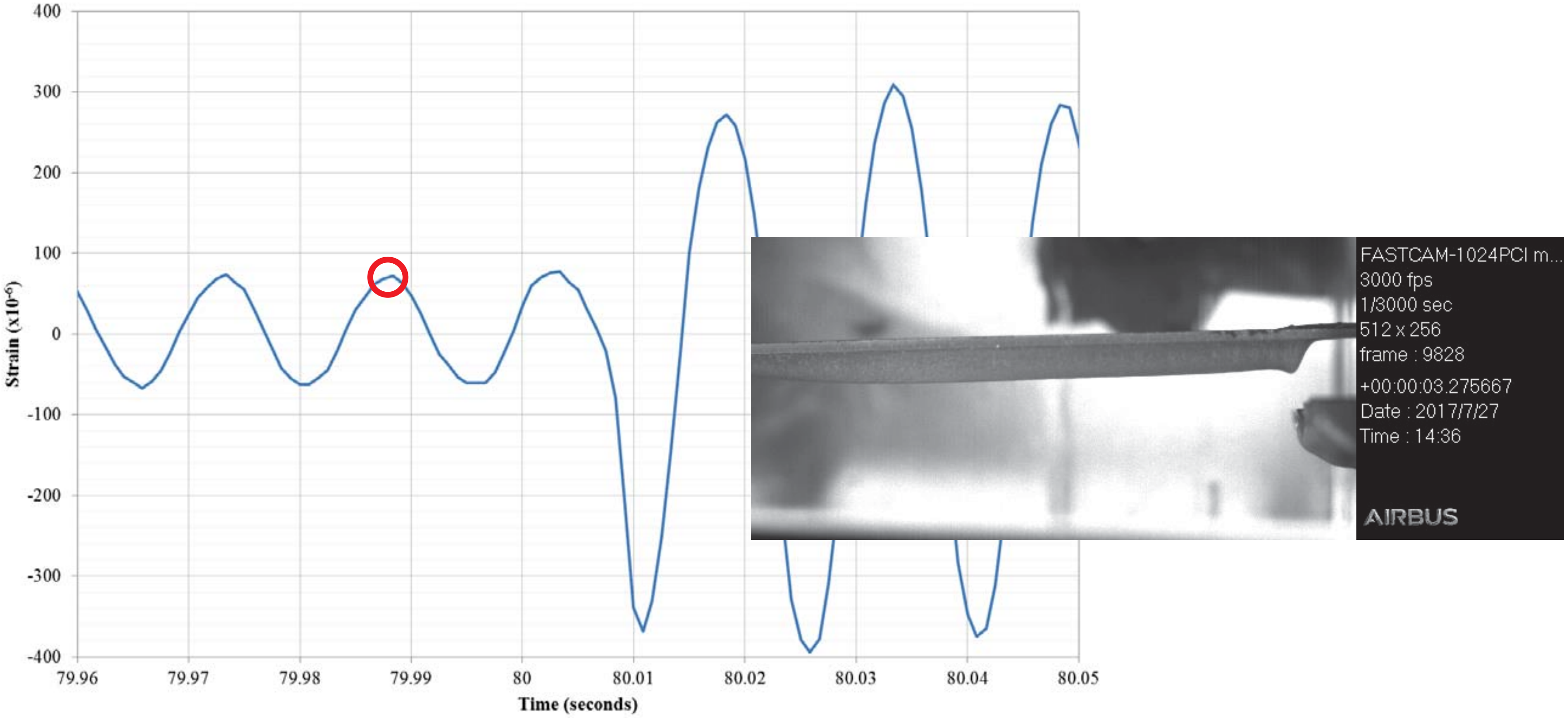


MISSISSIPPI STATE UNIVERSITY

AIRBUS

Preliminary Results

Strain gauge data analysis with HSC video

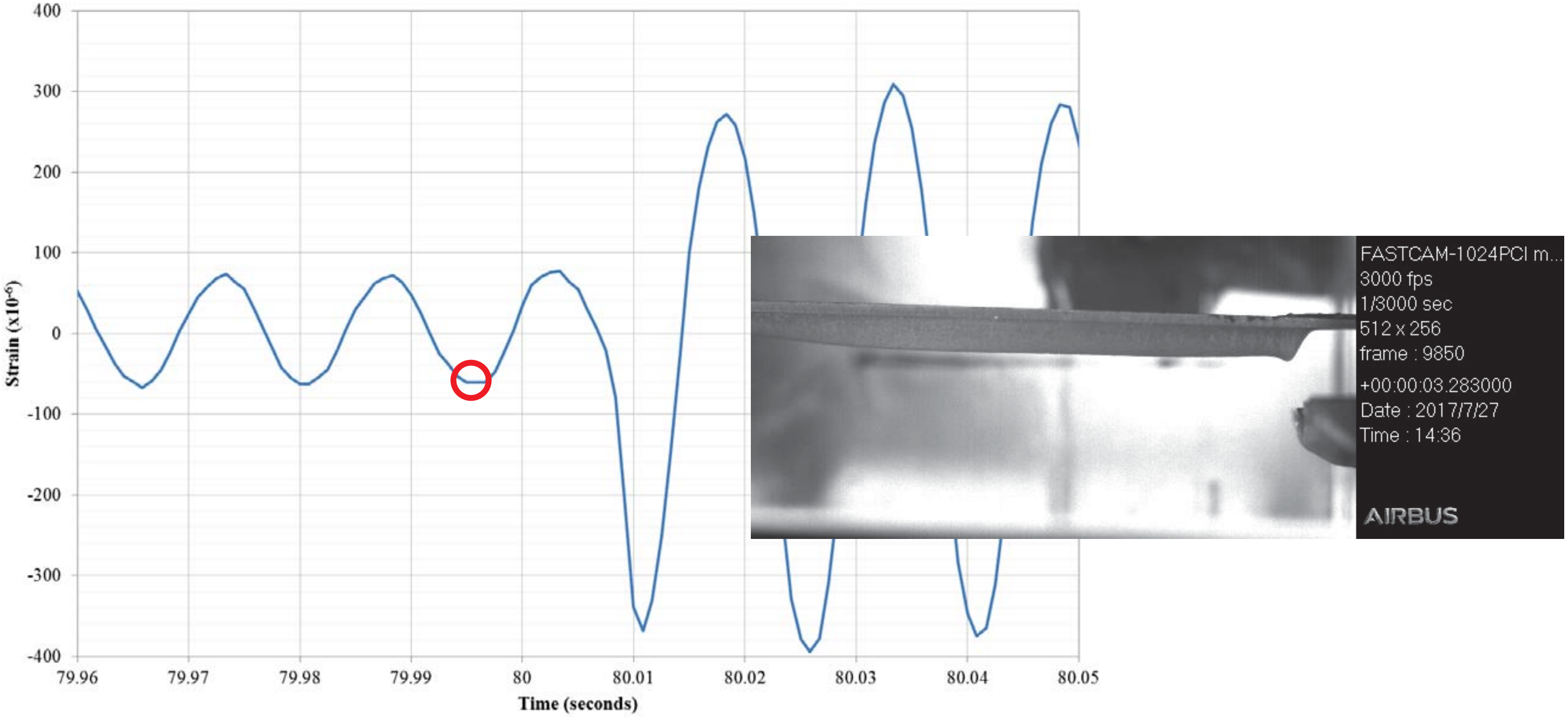


MISSISSIPPI STATE UNIVERSITY™

AIRBUS

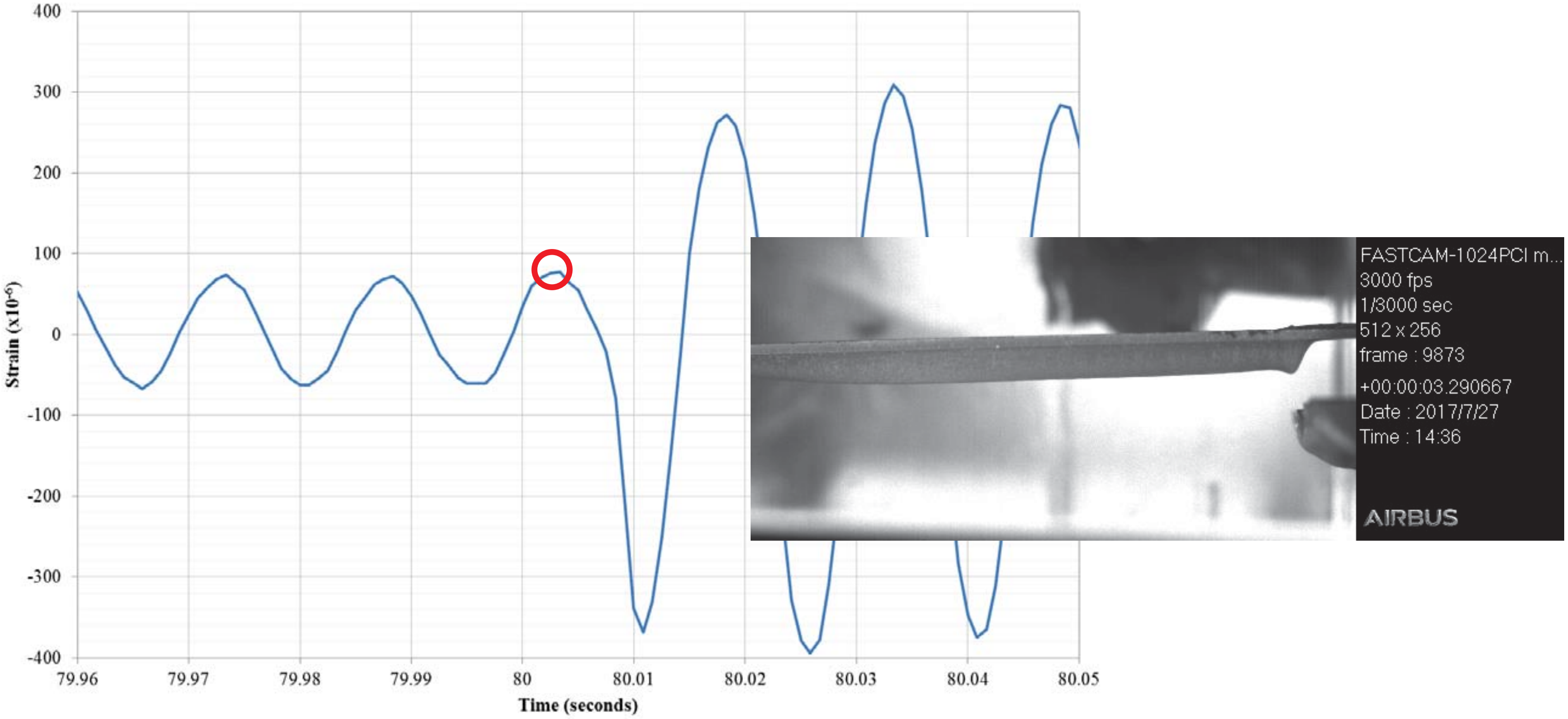
Preliminary Results

Strain gauge data analysis with HSC video



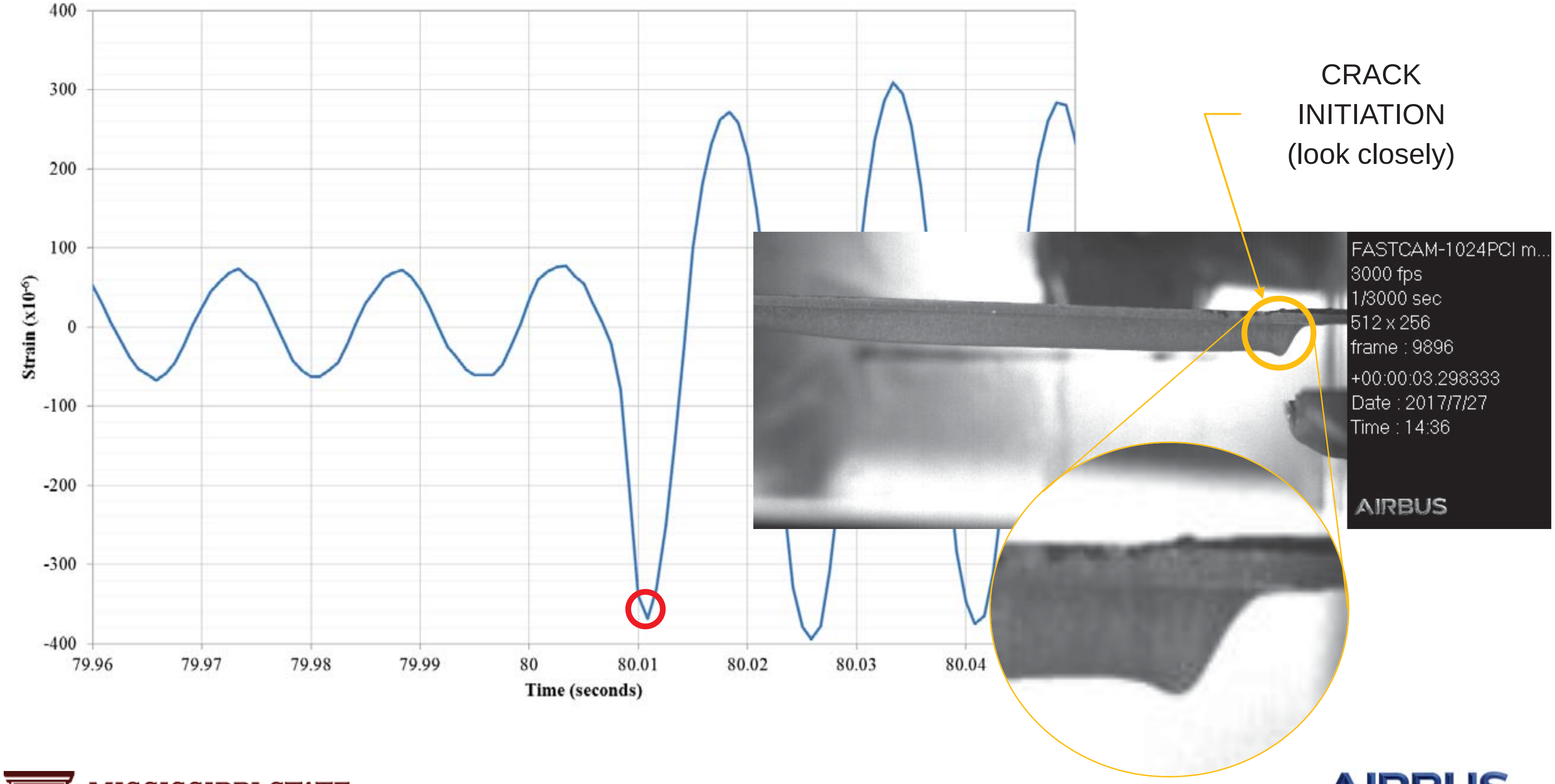
Preliminary Results

Strain gauge data analysis with HSC video



Preliminary Results

Strain gauge data analysis with HSC video



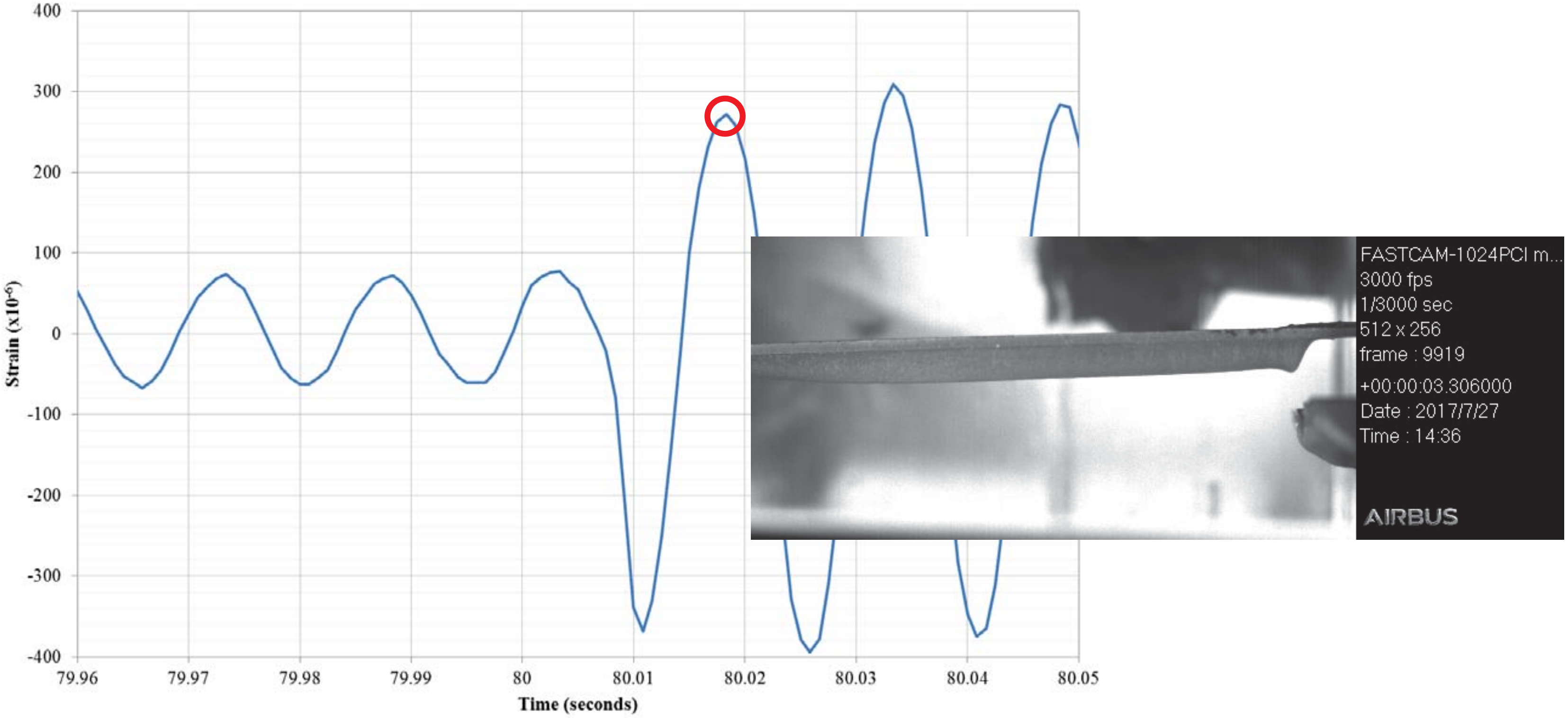
CRACK
INITIATION
(look closely)

MISSISSIPPI STATE
UNIVERSITY

AIRBUS

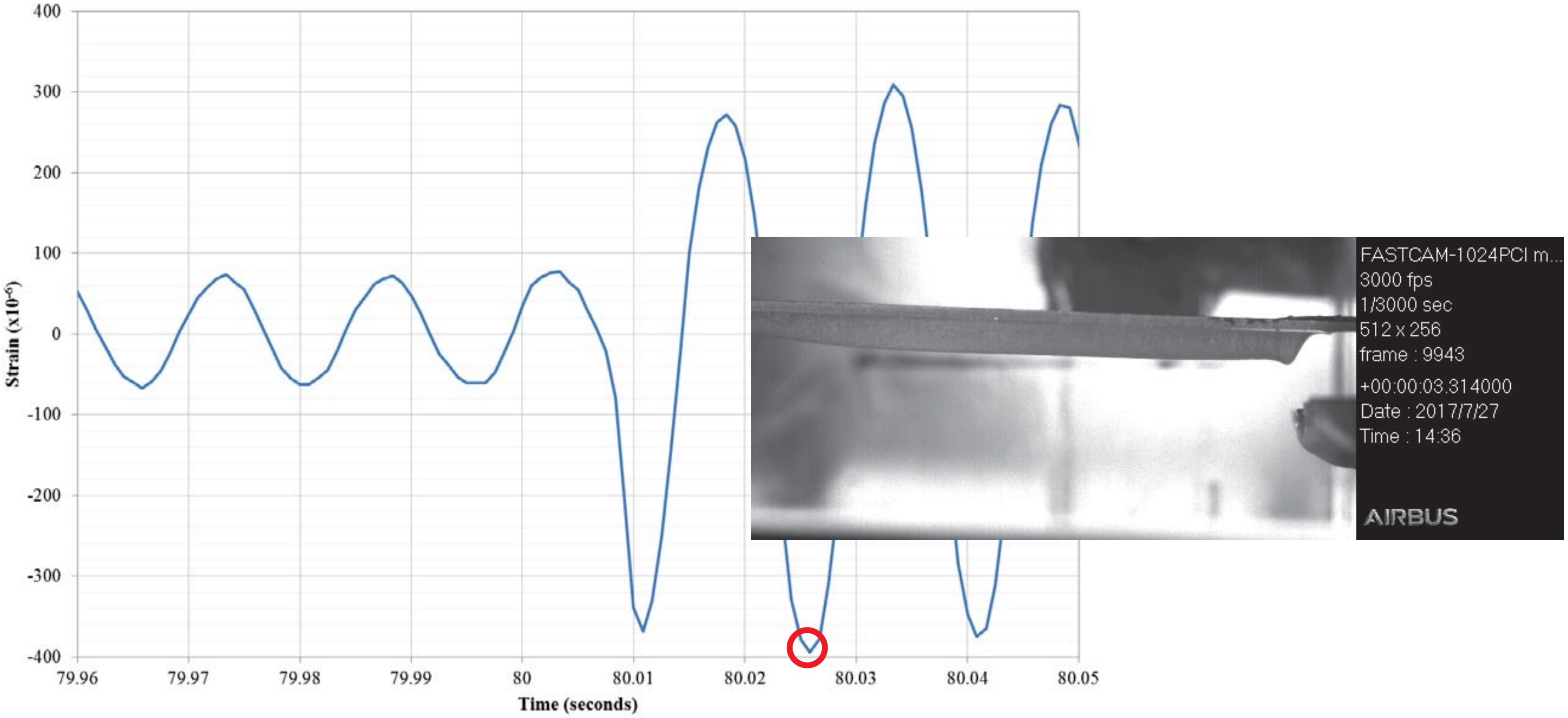
Preliminary Results

Strain gauge data analysis with HSC video



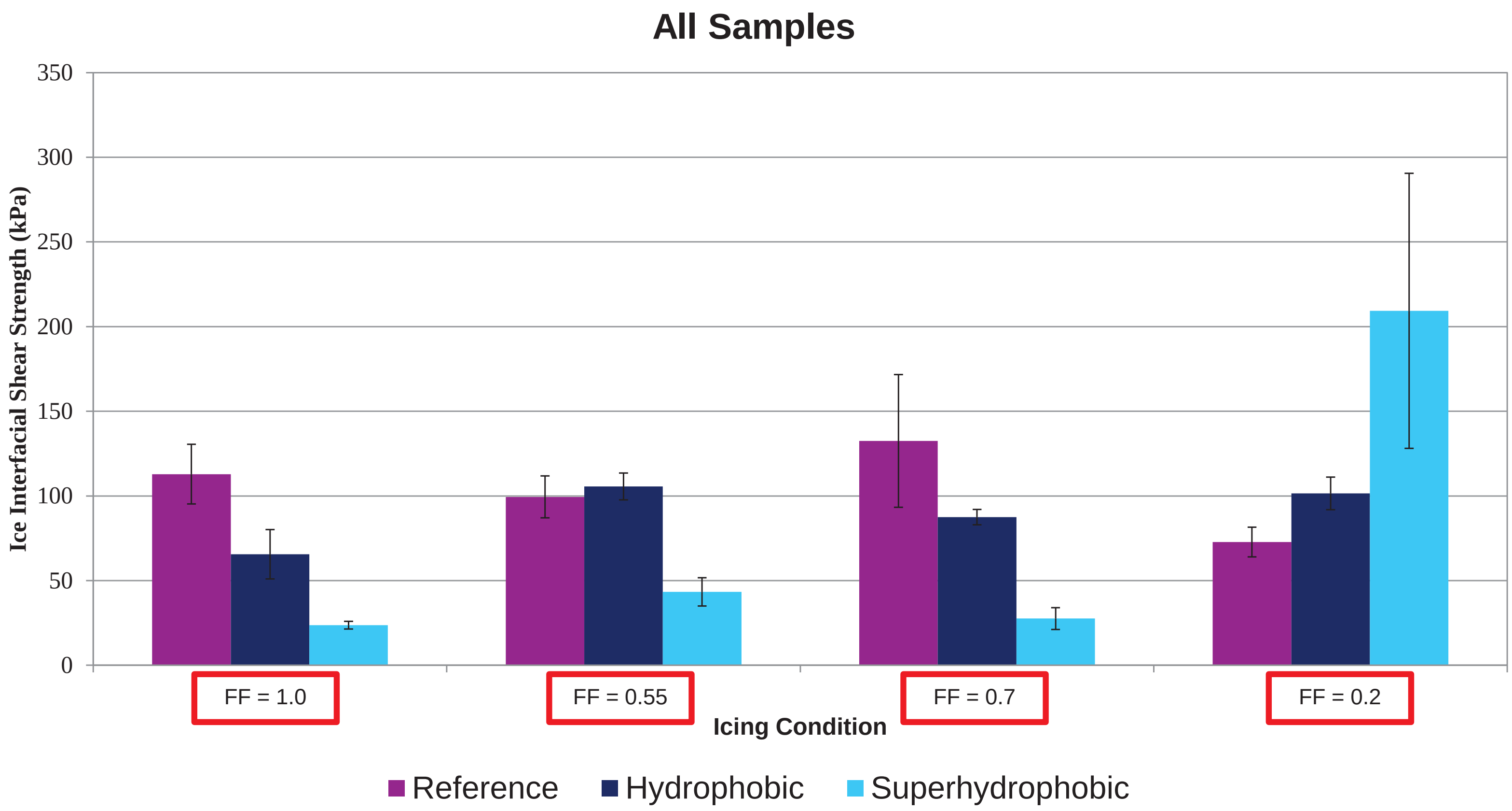
Preliminary Results

Strain gauge data analysis with HSC video



Multiscale Modeling of Ice Adhesion

Preliminary Results



MISSISSIPPI STATE UNIVERSITY

AIRBUS

Outline of Presentation

1. Overview
2. Experimental Method
3. **Ice Adhesion Stress: Empirical Model**
4. Ice Adhesion Stress: Predictive Models
5. Summary

MISSISSIPPI STATE UNIVERSITY™

AIRBUS

Empirical Modeling

Strategy: Surface fitting of experimentally-determined data

- Develop a parametric representation of the adhesive as a function of the N_m macroscale parameters

$$F_{AD} = f\left(\varphi_{m_1}, \dots, \varphi_{m_{N_m}}\right)$$

  - $\varphi_{m_n}(n = 1, N_m)$ represent the relevant macroscale parameters
  - F_{AD} is an $(N_m + 1)$-dimensional hypersurface.

- Determine significant parameters through a study of the correlation of the experimentally-measured adhesive stress to measured macroscale parameters
- Functional form depends on experimental data
  - Smooth and continuous curves (at least C_1-continuous)
  - Non-monotone (implies non-linear function)

Outline of Presentation

MISSISSIPPI STATE UNIVERSITY™

AIRBUS

Predictive Modeling

Central question

How does the adhesive shear stress measured in experiments (a macroscopic property) relate to the properties of ice-substrate interface @ *multiple length scales* (such as chemical composition, structural features, wetting profiles, etc.).

Perspective

We cast this problem into category of research on heterogeneous materials, where the behavior at the solid-solid interface (ice-substrate interface) is a key factor in determining overall performance.

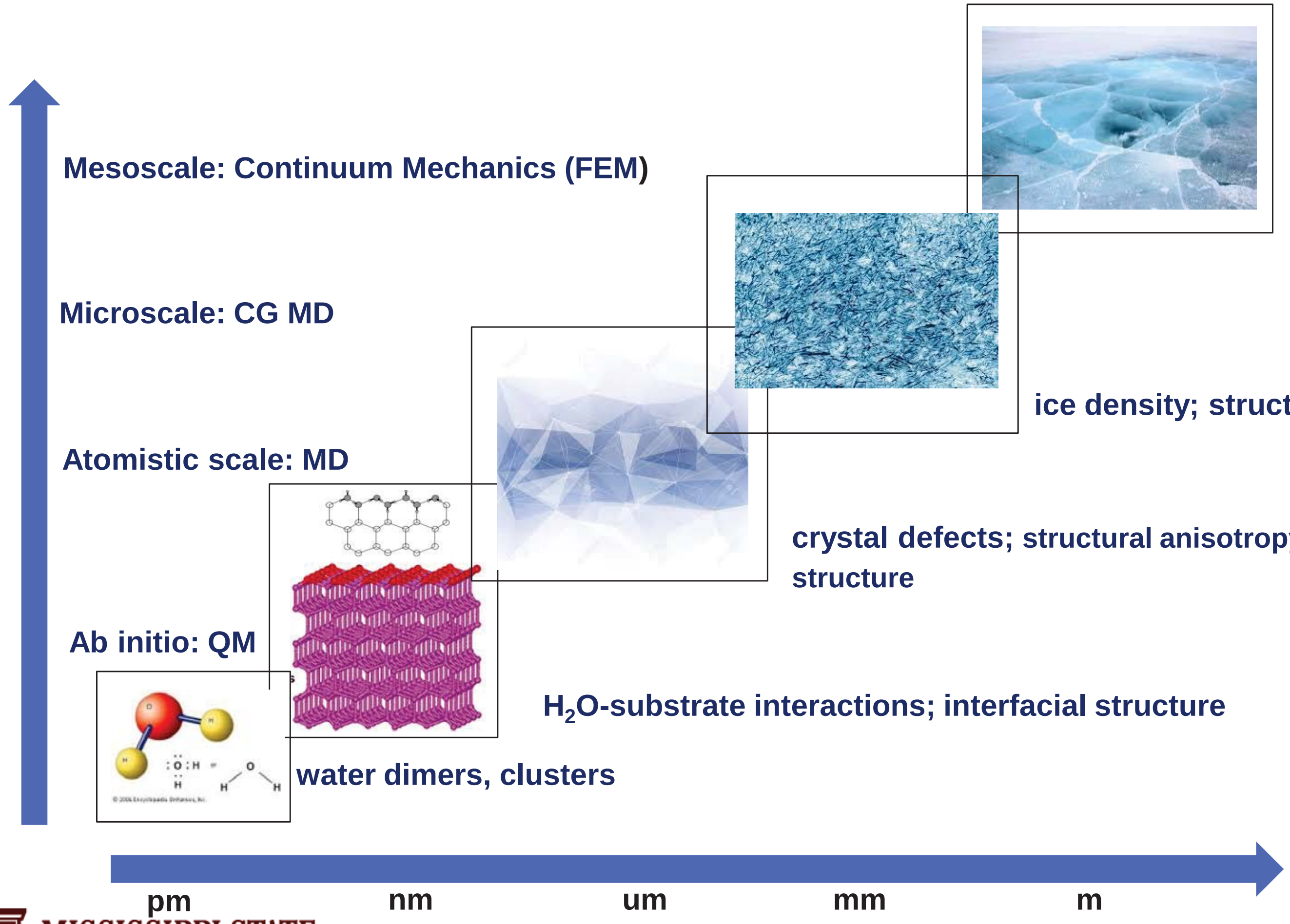
Scope

The investigation of the interface involves its chemical stability, physical compatibility, microstructures, intra/inter-phases, mechanical failure, …

Goals

1. Elucidate the roles played by each of the above mentioned phenomena in determining mechanical properties of the ice-substrate interface.
2. Make a first attempt at developing a unified approach (a surrogate model) for incorporating multiscale factors.
3. Provide important considerations for the next generation model.

The Multiscale Nature of Ice Fracture



Mesoscale: Continuum Mechanics (FEM)

Microscale: CG MD

Atomistic scale: MD

Ab initio: QM

ice density; structure porosity

crystal defects; structural anisotropy; substrate structure

H_2O-substrate interactions; interfacial structure

water dimers, clusters

MISSISSIPPI STATE UNIVERSITY

AIRBUS

Hybrid Multi-scale Model

Hypothesis: Measurable macroscopic quantities, i.e., the ice adhesion stress can be correlated with computed nanoscale and mesoscale parameters, i.e., **surrogate parameters**

Macroscale quantities – easily measured but not easily predicted

Atomistic scale and microscale properties – readily predicted but not easily measured

- The atomistic and microscale MD simulations reveal characteristics that provide an alternative to phenomenological parameters such as contact angle
  - Atomistic MD simulations characterize interfacial molecular bonding, interfacial crystal structure, mechanics of interfacial debonding under shear.
  - Microscale MD simulations characterize crystal interfacial defects, grain boundaries, and their roles in fracture mechanics.

Hybrid Multi-scale Model

Proposed functional variation

$$F_{AD} = f\left(\varphi_{m_1}, \dots, \varphi_{m_{N_m}}, \varphi_{\mu_1}, \dots, \varphi_{\mu_{N_\mu}}, \varphi_{n_1}, \dots, \varphi_{n_{N_n}},\right)$$

$\varphi_{m_n}(n = 1, N_m)$ represent the relevant macroscale parameters

$\varphi_{\mu_n}(n = 1, N_\mu)$, represent the relevant mesoscale parameters

$\varphi_{n_n}(n = 1, N_n)$ represent the relevant nanoscale parameters

Hybrid Strategy

- Measure macroscale parameters.
- Predict mesoscale and nanoscale parameters.
- Systematically determine significant parameters through a correlation study relating the experimentally-measured adhesive stress to measured macroscale parameters and computed mesoscale and nanoscale parameters.
- Use a fitting strategy similar to the one employed for the purely empirical model to estimate the functional relationship.

MISSISSIPPI STATE UNIVERSITY

AIRBUS

Predictive Modeling (1μm-100μm)

Motivation

1. In the previous methods, correlation studies are employed to bridge the gap between scales, i.e., relate parameters predicted from simulations with experimental measurements
2. Our simulations provide a deeper understanding of fracture mechanics at the lower length scales. With these insights, a rationally-designed “purely” predictive model can be formulated that better incorporates lower length scale details, in place of the correlation studies.

Proposed Strategy

1. At the length scale from 1μm-100μm, ice density as a result of structure porosity needs to be considered. However, predicting the pore size and distribution is beyond the capability of current microscale simulations. However, this information can be available from experimental measurements (e.g. in research on catalysis, gas adsorption models can be used for estimating such information).
2. Provided with the micronscale structure, a finite element method can be employed to construct an ice-substrate model, with the spatially varying elastic modulus delivered by **microscale simulations**.

Microscale (MD) Simulations (10nm-1000nm)

Objective

Investigate nanoscale structural features (e.g. due to crystal defects, surface roughness, etc), and their effects on fracture mechanics. The “chemical ingredients” are incorporated through coarse grain parameters guided by **atomistic simulations**.

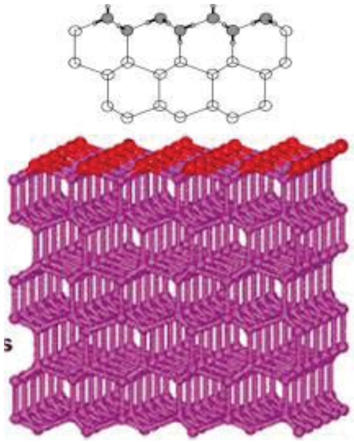
Scope of study

1. Characterize structure and structural defects at interfaces, such as crystal grain size and boundaries, as the result of *nanoscale* surface roughness.
2. Investigate effects of droplet impingement speed on the wetting behavior of super cooled droplets on a surface with *nanoscale* roughness, and the subsequent crystallization structure (Wenzel vs. Cassie).
3. Investigate fracture mechanics of ice-substrate interface @ nanoscale, which involves failure of grain boundary networks and crack propagation.

Methods of study

1. Equilibrium molecular dynamics simulations employing coarse grained (CG) models for water and substrates. New CG models may be needed in order to extend length scales of simulations.
2. Non-equilibrium molecular dynamics simulations based on CG models under shear/deformation.

Atomistic-scale (MD) Simulations (0.1nm-10nm)



Objective

Characterize the structure and failure mechanics of ice-substrate interface at the atomistic scale, with the *effects of* ***molecular details fully captured***.

Scope

1. Characterize ice-substrate interfacial structure and its dependence on molecular ordering and chemical composition.
2. Investigate effects of droplet impingement speed on ice crystallization at interface (e.g. shock-induced crystallization, fluctuations in molecular distribution near interface).
3. Quantify respective contributions to the “interfacial bonding” from molecular interactions such as H-bond, electrostatics and van der Walls.
4. Investigate the mechanics of “interfacial bond breaking” at the heterogeneous ice-substrate interface under shear and active deformation

Methods

1. Equilibrium MD simulations using all-atom models for water (TIP3P, TIP4P, SPC/E) and substrate (Al, steel, polymers).
2. All-atom, non-equilibrium molecular dynamics simulations under shear/ active deformation.

MISSISSIPPI STATE UNIVERSITY

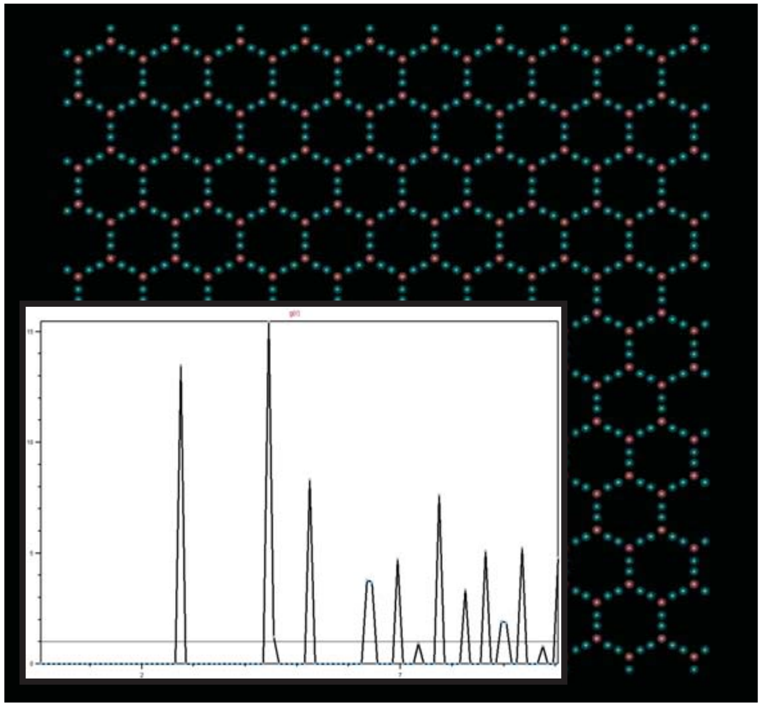
AIRBUS

Preliminary Results: Force Field Calibration Liquid State

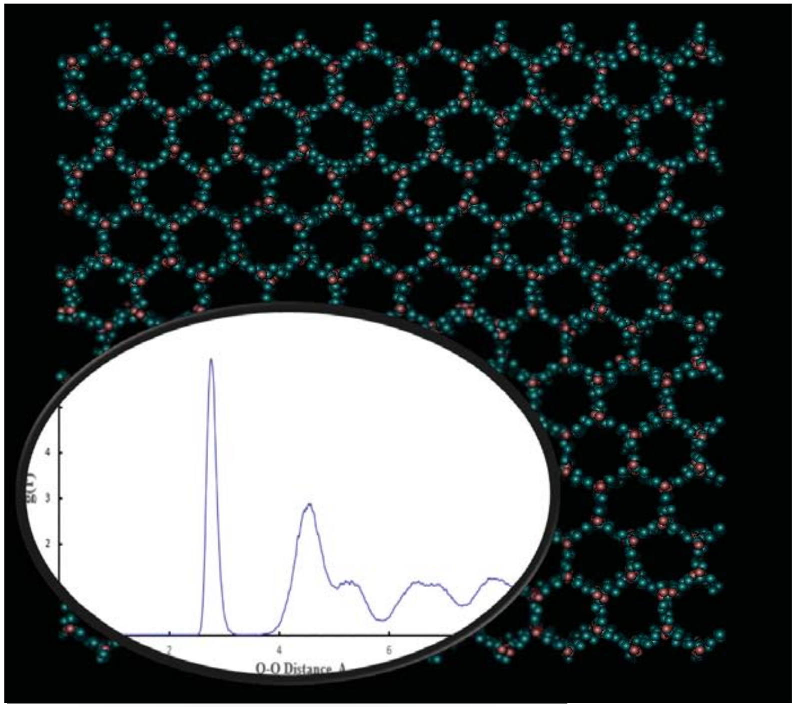
TIP4P/ice water model simulation compared well with experimental results.

TIP4P/ice Water Model- Simulation result- 300K * A. K. Soper, Chem. Phys. 258, 121 (2000)
TIP4P/ice Water Model- Published result- 298KAbascal et al. J. Chem. Phys. 122, 234511 (2005)

Preliminary Results: Equilibrated *ih* Ice Structure



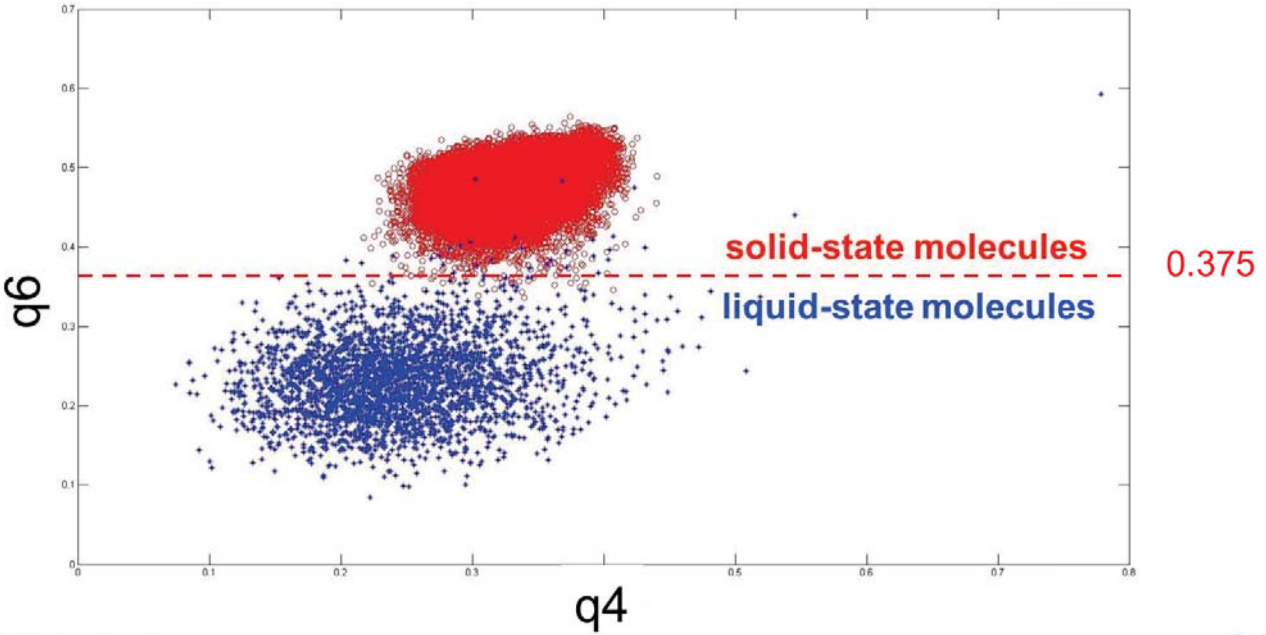
Perfect Crystal Structure at 0K



NPT Simulation at 200K and P = 1.0 atm

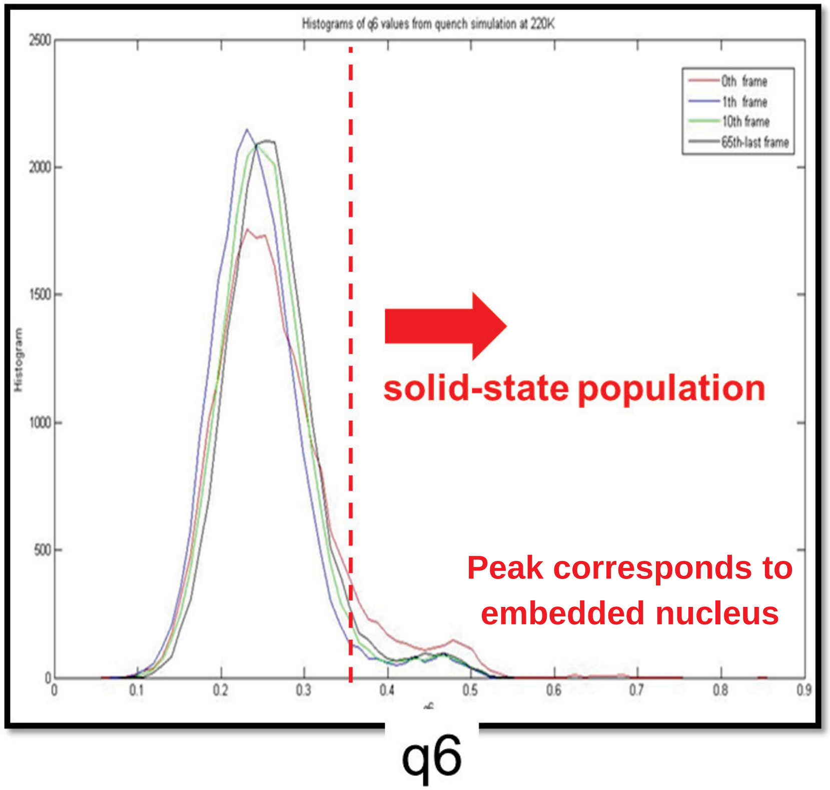
Preliminary Results: Ice Nucleation by ”Seeding”

- **Procedure**
  - Embed a spherically-shaped ice nucleus in super-cooled liquid water;
  - Measure change in size of solid-state (ice) cluster with time. The size of solid-state cluster can be characterized fully by the *q6* order parameter.



Preliminary Results: Ice Nucleation by ”Seeding”

Solid-state Population Change with Time Measured by q6 (work in progress)



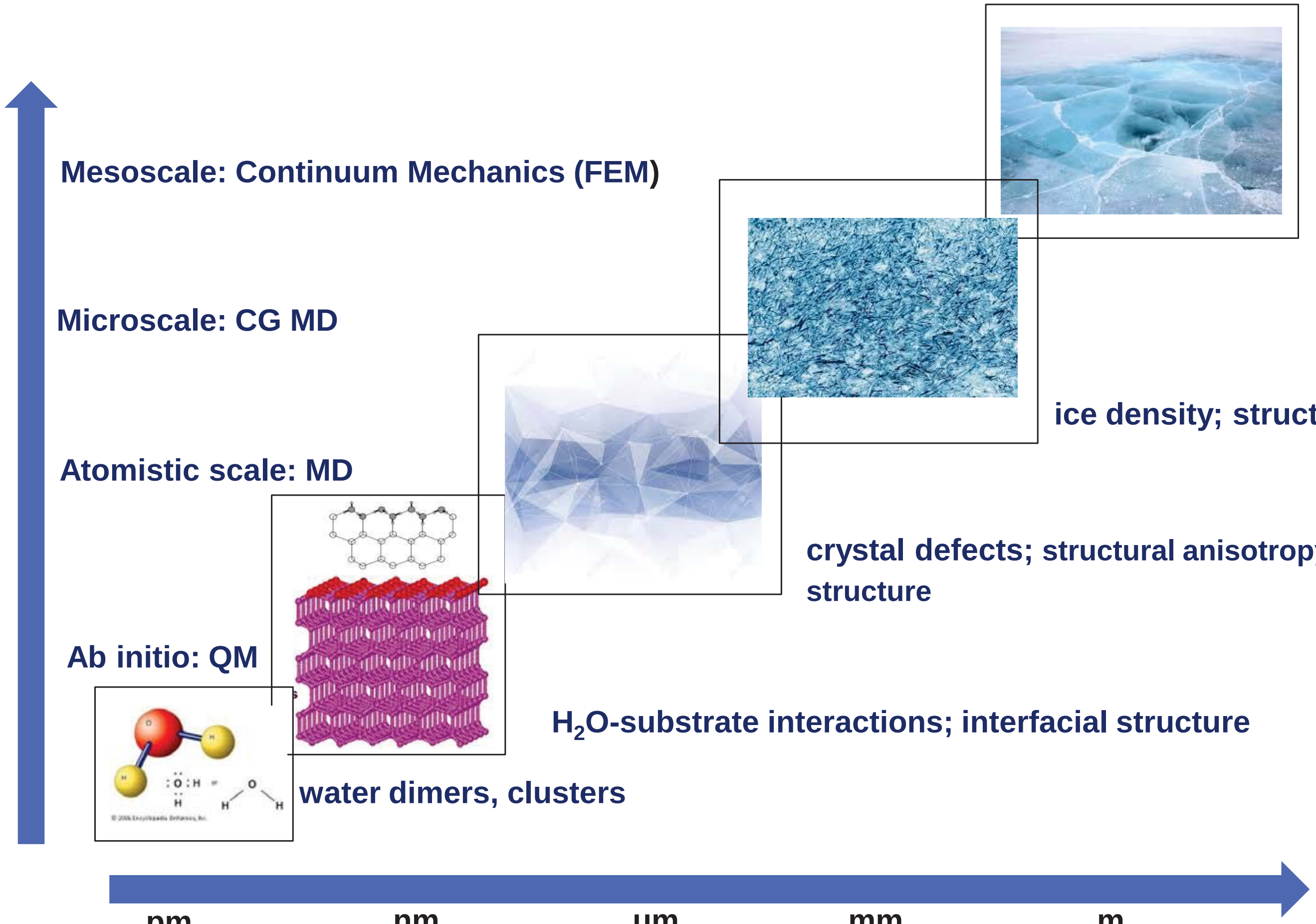
solid-state population

Peak corresponds to embedded nucleus

Outline of Presentation

1. Overview
2. Experimental Method
3. Ice Adhesion Stress: Empirical Model
4. Ice Adhesion Stress: Predictive Models
5. **Summary**

Summary



Mesoscale: Continuum Mechanics (FEM)

Microscale: CG MD

Atomistic scale: MD

Ab initio: QM

ice density; structure porosity

crystal defects; structural anisotropy; substrate structure

H_2O-substrate interactions; interfacial structure

water dimers, clusters

pm **nm** **um** **mm** **m**

Ice Adhesion Characterization of Icephobic Materials for Aircraft Icing Mitigation

Ashraf Bastawros, Wei Hong, Hui Hu
Department of Aerospace Engineering
Iowa State University

Eric E. Kreeger, TM

August 10, 2017

This material is based on work supported by the NASA Grant and Cooperative Agreement # NNX16AN21A, and performed at Iowa State University

Ice Adhesion Characterization of Icephobic Materials for Aircraft Icing Mitigation

Post Doctoral Research Associate:

Rye Waldman

Graduate Students:

Bishoy Dawood, Christopher Giuffre, Prashanth S R Beeram

Undergraduate Students:

Ryan Cazin, Aaron Still, David Hoskins

IOWA STATE UNIVERSITY
OF SCIENCE AND TECHNOLOGY

This material is based on work supported by the NASA Grant and Cooperative Agreement # NNX16AN21A, and performed at Iowa State University

Objective

Experimental Mechanics
Ashraf Bastawros

Multi-Physics Modeling
Wei Hong

Improve Performance of Ice Protection System

- In situ characterization of adhesion and cohesion of impact ice over various icephobic materials.
- Establish fundamental correlations between thermo/hydro/aero-dynamic effects and ice accretion on airfoil surfaces

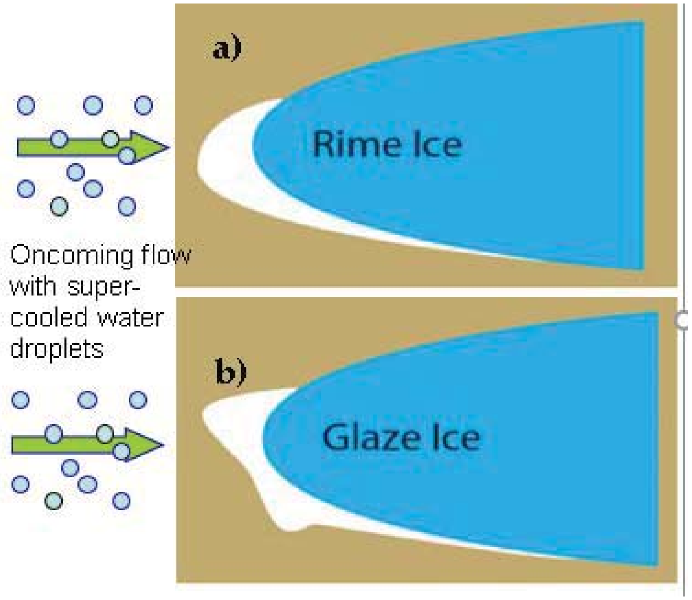
Experimental Aerodynamics/ Icing Tunnel
Hui Hu

2. Road Block

Impact Ice: Physical and Mechanical

properties change with:

- Flow condition (impact velocity, liquid water content, LWC)
- Environment (temp)
- Surface (topology, coating)
- Contaminates (polar vs. non-polar surfaces)



➢ Needs to characterize ice adhesion under these conditions

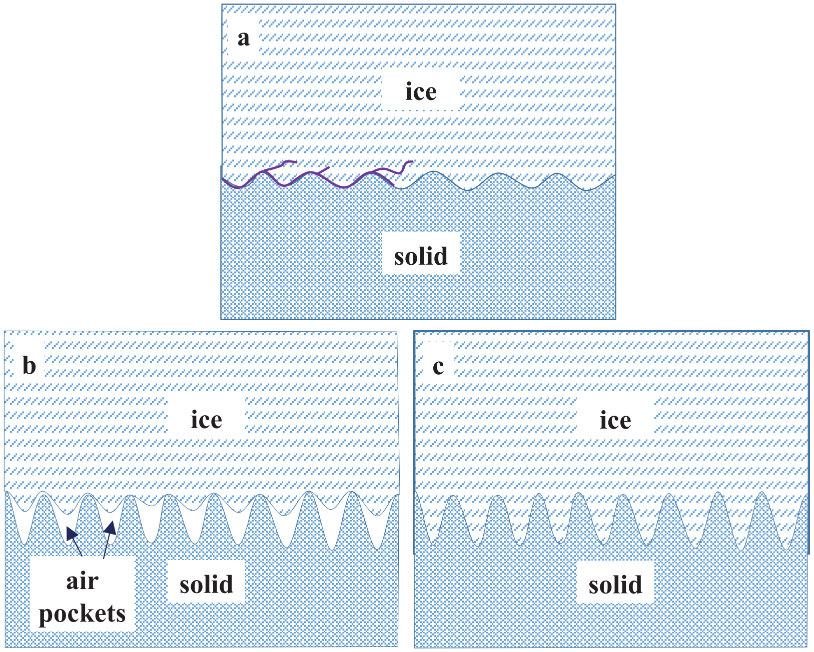
3. Goals

(1) *Development of in situ adhesive and cohesive measurement system for impact ice:* Characterization of mechanical and physical properties of impact ice under different icing condition for calibration of modeling framework.

(2) *Development of multiscale physics-based modeling of ice adhesion:* Address role of microscale surface roughness, environmental and flow conditions; and provide prediction of nucleation and growth of multiple cracks.

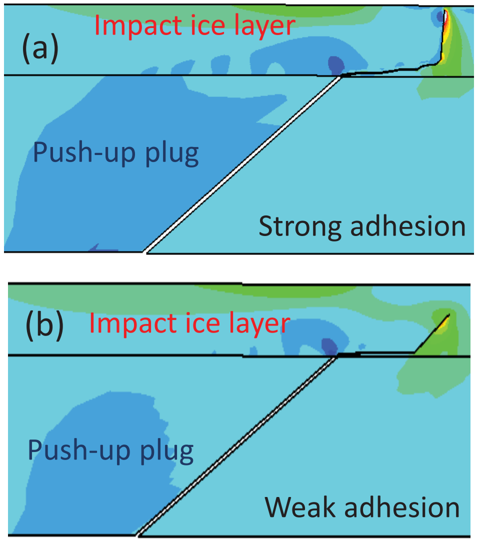
(3) *Quantify the performance of IPS over ice accretion surfaces under different conditions:* Provide phenomenological understanding, and laboratory measurements for ice accreting surfaces of the airfoil/wing models under different icing conditions similar to NASA N+2/N+3 vehicles encounter along their flight envelopes.

4. Physics-Based Multi-Scale Ice Adhesion Model



(i) Microscopic Scale

- Local surface roughness
- Phase Field Model of fracture



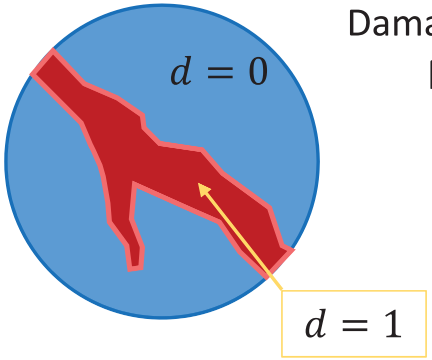
(ii) Macroscopic Scale

- Crack trajectory
- Extended Finite-element methods

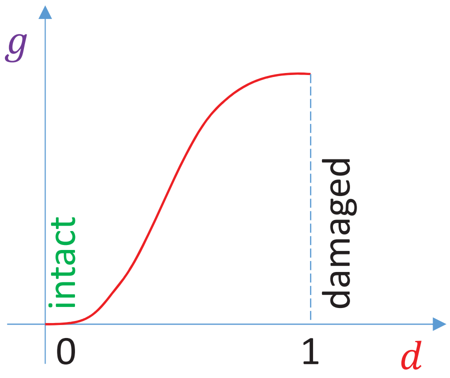
4.1 Phase-field model of fracture (KKL)

One difficulty in computational fracture mechanics:
tracking the crack(s)!

The phase-field approach:
A continuous phase field $d(\mathbf{x}, t)$



Damage degrades structural integrity:
Modulus $E = E_0[1 - g(d)]$



- Karma, Kessler, Levine, PRL (2001)
- Hakim, Karma, JMPS (2009)

Thermodynamics of damage

Intrinsic length scale

(process-zone size) $r_0 \sim \sqrt{\frac{W_0}{a}}$

Total potential energy

$$\Pi = \int \left\{ \underbrace{[1 - g(d)]W_s(\boldsymbol{\epsilon})}_{\text{Strain energy}} + \underbrace{\underbrace{g(d)W_0}_{\text{Damage energy}} + \underbrace{\frac{a}{2}|\nabla d|^2}_{\text{Gradient energy}}}_{\text{Fracture energy}} \right\} dV$$

Kinetics of damage evolution

$$\frac{\partial d}{\partial t} = -m \frac{\delta \Pi}{\delta d}$$

- Karma, Kessler, Levine, PRL (2001)
- Hakim, Karma, JMPS (2009)

Phase-field model of fracture

Pros

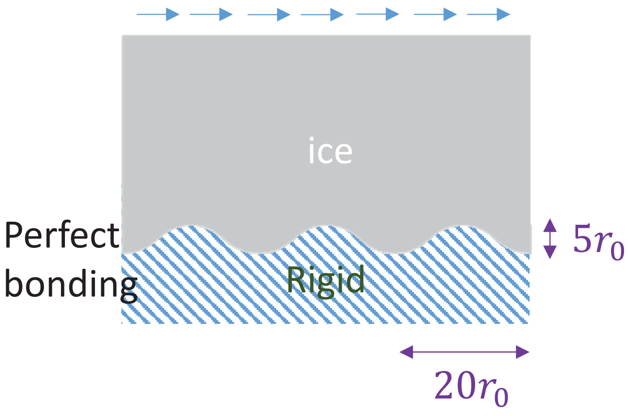
- Fracture-mechanics / thermodynamics based
- Versatile & robust, no need for pre-determined crack path
- Can handle large deformation & plasticity
- Can include surface chemistry*

Cons

- Size limited by computational power
- Phenomenological model (non first principle)
- Needs parameters from experiments / lower level models

Preliminary calculation

Dependence of apparent adhesion strength on surface roughness

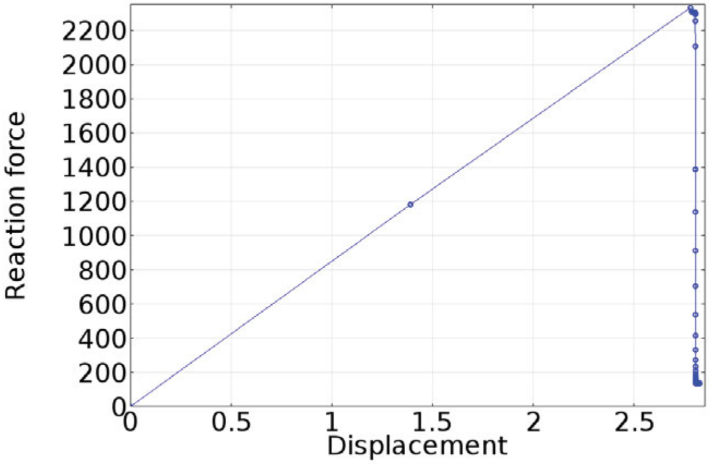


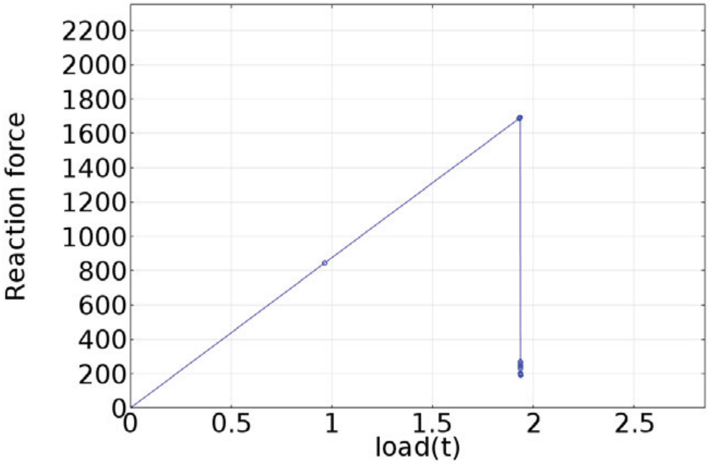
Process zone size of ice (estimate)

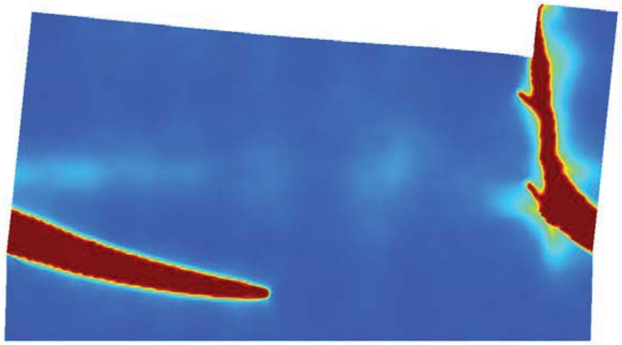
$$r_0 \sim \frac{1}{2\pi}\left(\frac{K_{\mathrm{Ic}}}{\sigma_Y}\right)^2 \sim \frac{1}{2\pi}\left(\frac{100\mathrm{kPa\ m}^{\frac{1}{2}}}{10\mathrm{MPa}}\right)^2 \sim 10\mu\mathrm{m}$$

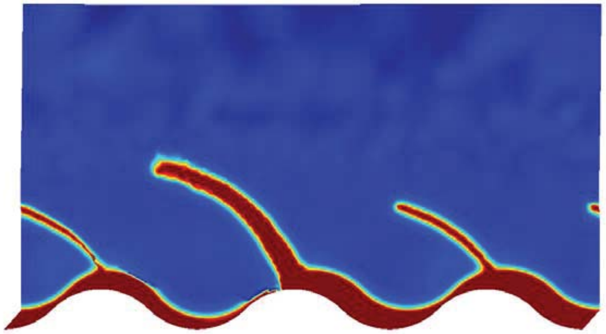
Liu & Miller, J. Glaciology (1979)

Preliminary results



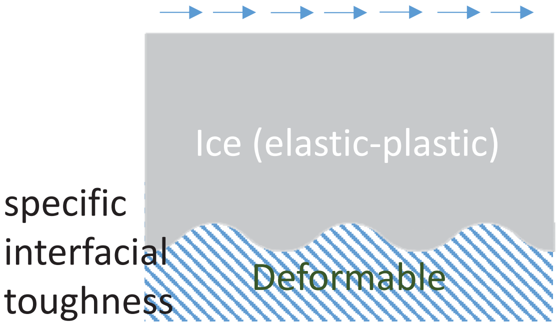






For brittle material (ice), rougher surface has lower apparent adhesion

Better models



- Plastic deformation in ice (during compression)
- Competition between adhesive (interfacial) & cohesive (bulk) cracks
- Deformable airfoil (effect of stiffness on adhesion)
- Other modes of loading
- Effect of impurities (e.g. particles and air bubbles) & microstructure
- Effect of surface chemistry

4.2 Cohesive-zone model

Pros

- Easy to implement
- Available in various commercial FEM packages (Abaqus, Ansys, etc.)

Cons

- Need to prescribe crack path
- Dubious traction-separation law
- Finer mesh near the crack

- Barenblatt, Adv. Appl. Mech. (1962)
- Dugdale, JMPS. (1960)

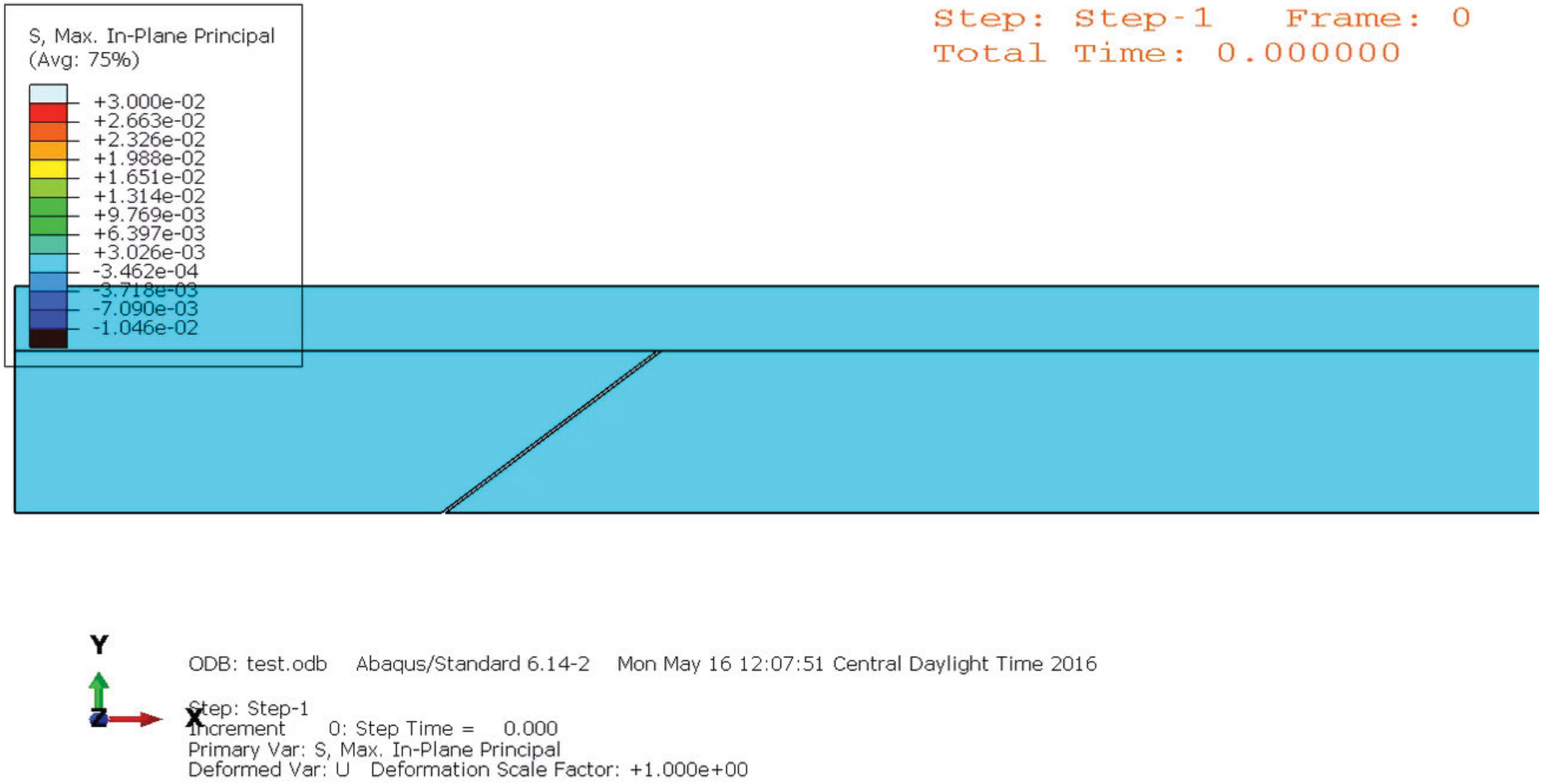
4.3 XFEM

- No need for pre-determined crack path
- Available in Abaqus / Ansys
- Can handle mode mixity

- Crack in element (tracked by level-set)
- Discontinuity across crack
- Enriched by local singular stress field (LEFM) or cohesive behavior

Belytschko & Black, I. J. Num. Meth. Eng. (1999)

Example of cohesive elem + XFEM

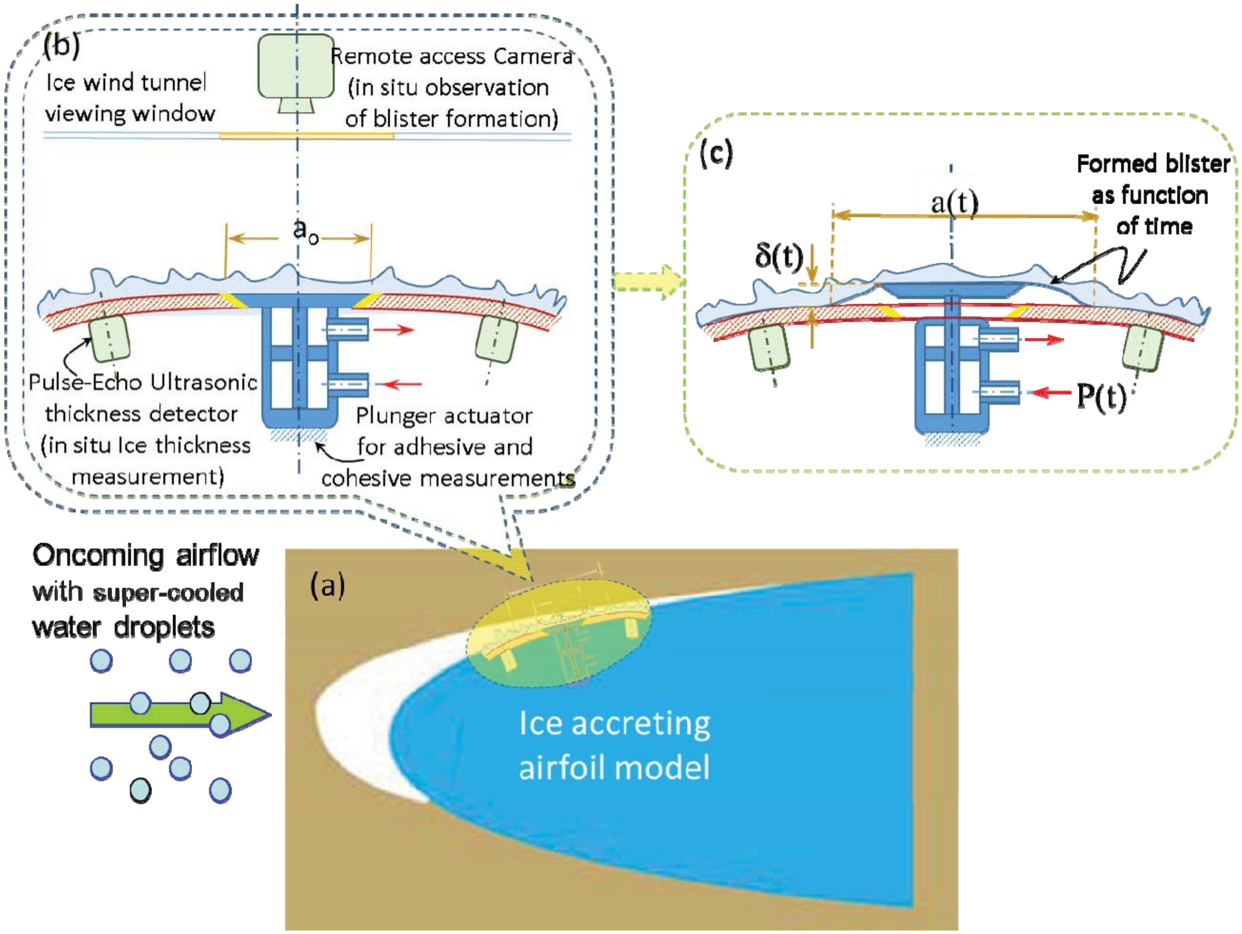


Step: Step-1 Frame: 0
Total Time: 0.000000

ODB: test.odb Abaqus/Standard 6.14-2 Mon May 16 12:07:51 Central Daylight Time 2016

Step: Step-1
Increment 0: Step Time = 0.000
Primary Var: S, Max. In-Plane Principal
Deformed Var: U Deformation Scale Factor: +1.000e+00

5. Design of In Situ Ice Adhesion/ Cohesion Measurement System



Background:

Strength vs. Fracture Mechanics View

Brittle Materials,

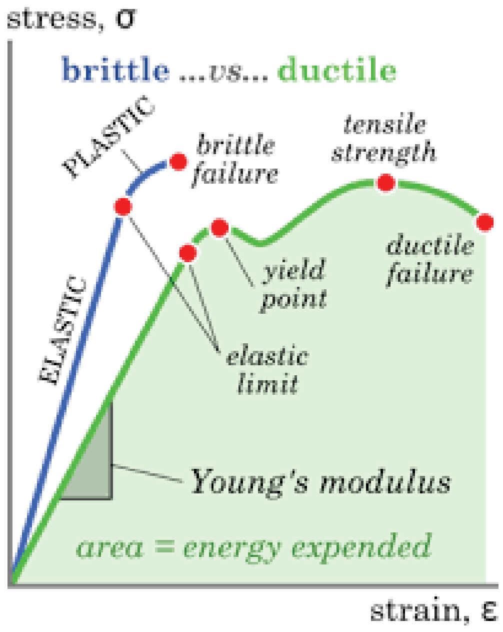
Energy = Elastic + Surface

$$\Rightarrow \quad \sigma_c\sqrt{a} = \sqrt{\frac{2\gamma E}{\pi}} \longrightarrow \gamma \sim 1J/m^2$$

Ductile Materials,

$$\Gamma \sim 100kJ/m^2$$

$$\sigma_c\sqrt{a} \approx \text{Constant} \gg \sqrt{\frac{2\gamma E}{\pi}}$$



Irwin and Orowan; apply it in an effective way,

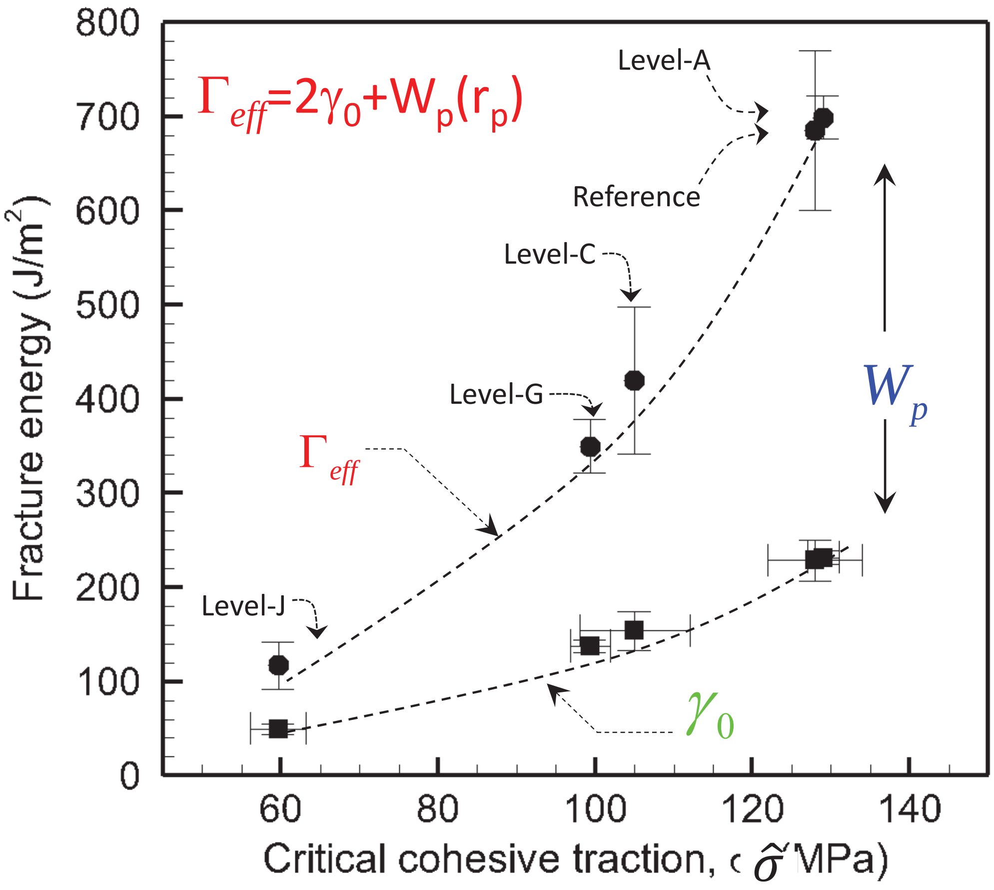
$$\sigma_c\sqrt{a} = \sqrt{\frac{\Gamma E}{\pi}};$$

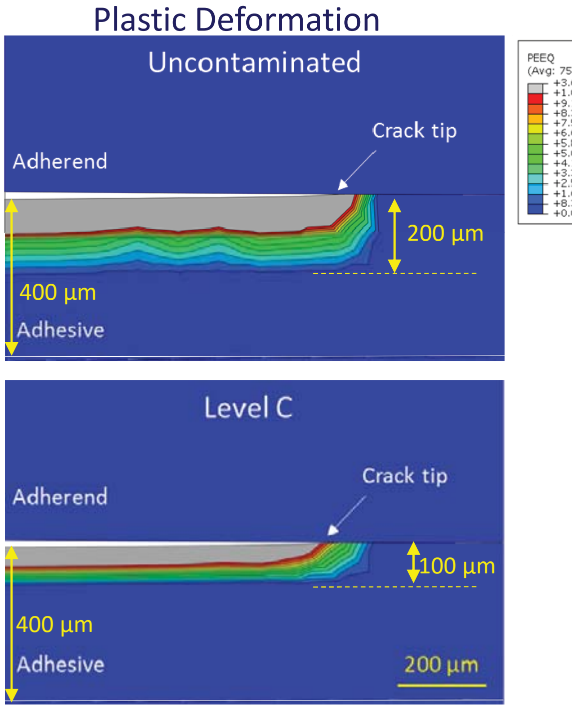
$\Gamma = 2\gamma + W_P$: Total Fracture Energy

σ_c : Adhesion Strengh

a : Crack Length (roughness)

Intrinsic vs. Effective Adhesion

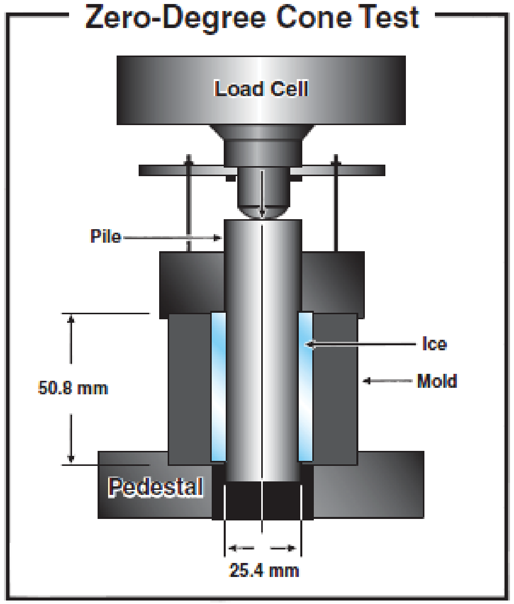


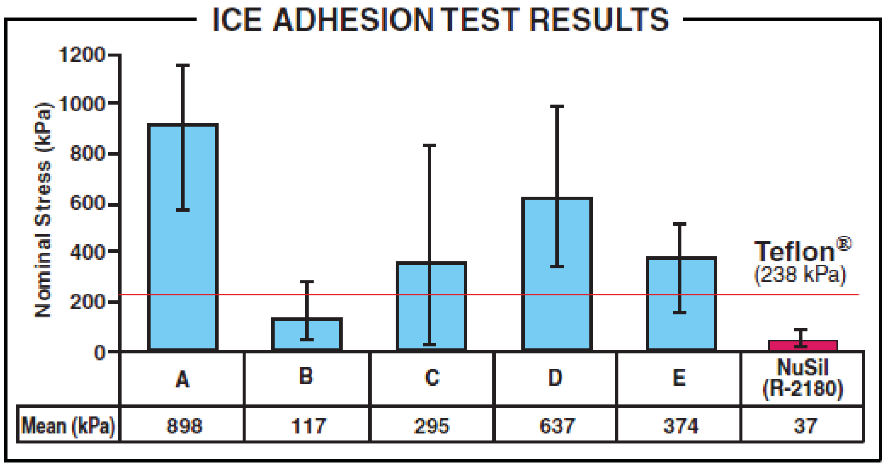


(*Example for polymer adhesion*)

Total Fracture Energy = Intrinsic Cohesive Energy + Plastic Dissipation Energy

Strength Testing





	A	B	C	D	E	NuSil (R-2180)
Mean (kPa)	898	117	295	637	374	37

R-2180, demonstrates the least amount of nominal stress compared to the other coatings tested. The other coatings with low mean stress values show high variability that is associated with the observation that the ice was in various states of solidification due to solutes leaching from the coatings into the water that lower the freezing point of the surrounding water.

Adhesion Strength Testing of Ice

One macroscopic parameter, nominal shear resistance

(Zero-degree cone test-CRREL, US Army Corps of Engineers)

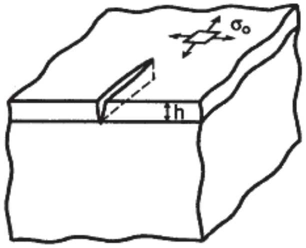
Background: Fracture Testing

Fracture Characterization:

(a) Channel crack within a thin ice films (cohesion)

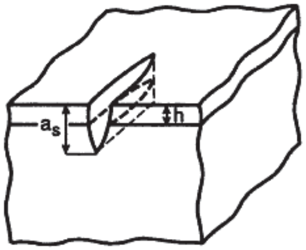
(b) Channel crack penetrating into the substrate

(c) Channel crack with interface debonding (adhesion)

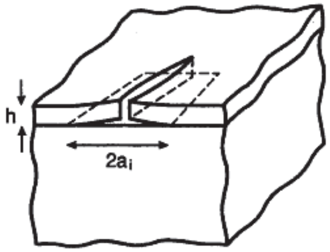


a) Film Cracking

✓



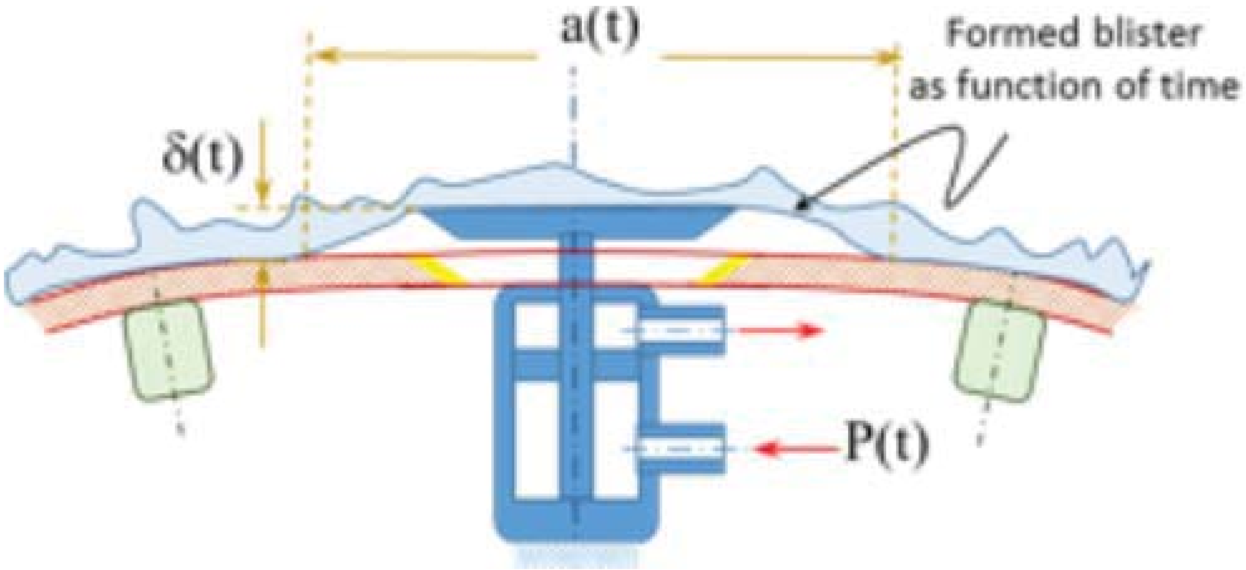
b) Substrate Penetration



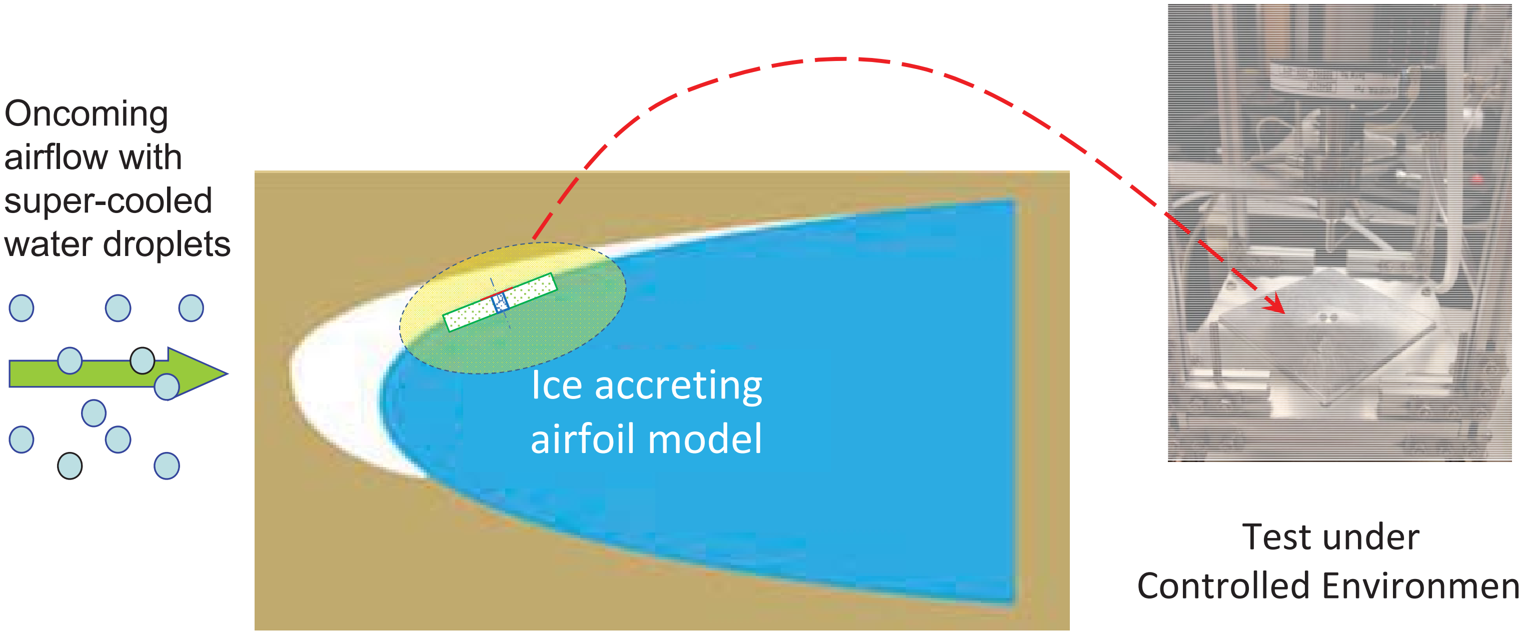
c) Interface Debonding

✓

5.1 Experimental Setup



In situ Characterization



Oncoming airflow with super-cooled water droplets

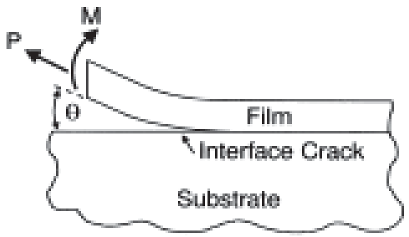
Ice accreting airfoil model

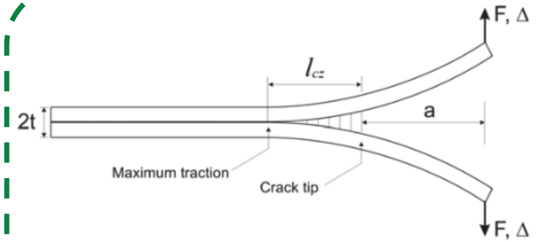
Test under Controlled Environment

Grow Impact Ice in the Icing Tunnel (Different flow and WC parameters)

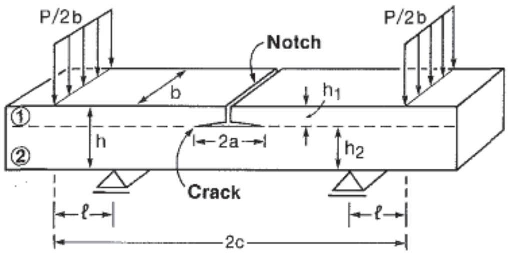
In-Tunnel Growth, Off-Tunnel Testing

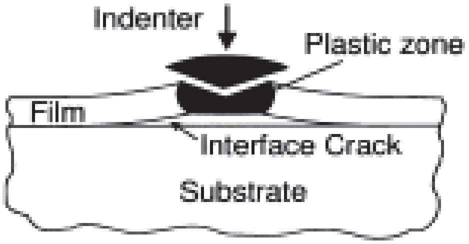
5.2 Well Characterized Fracture Experiments

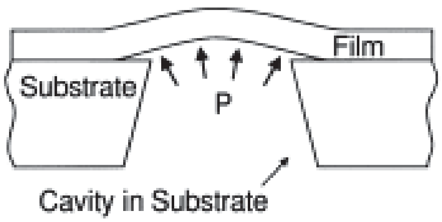




DCB for Calibration



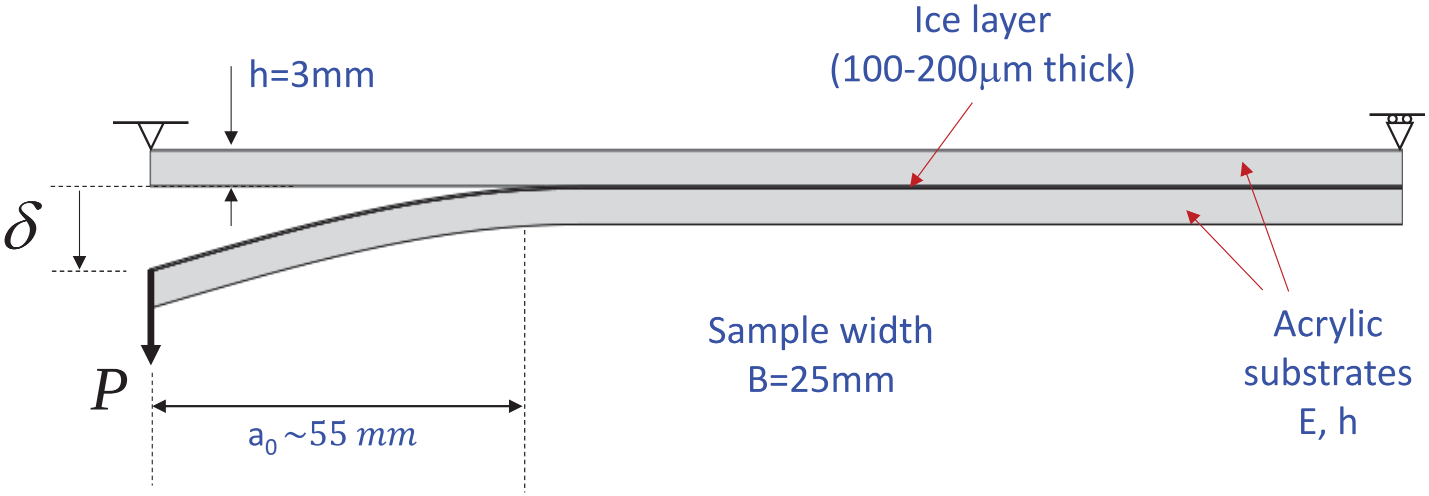




Blister test for in situ

- Estimates of impact ice properties by well characterized experiment
- Preliminary design for off-tunnel testing
- Design and implementation for in situ testing

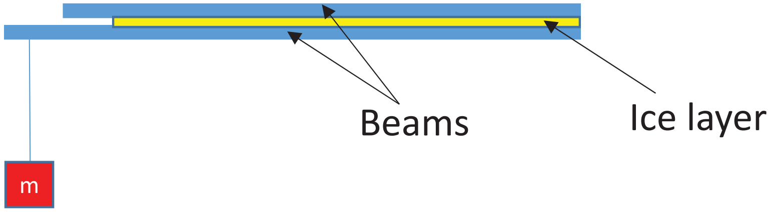
5.3 Preliminary Fracture Experiments

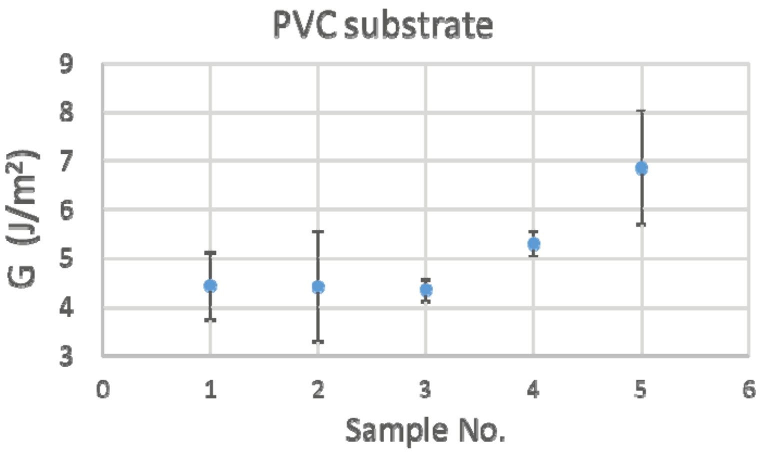


Fracture driving force $$G = \frac{6P^2a^2}{EB^2h^3}$$

Total Fracture energy $$G_0 = G\big|_{a=a_c}$$

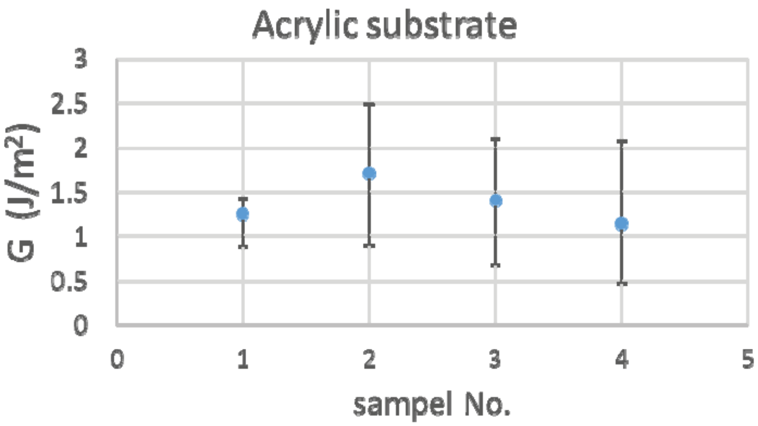
Well Characterized Fracture Experiments





G_{ave}=5.07 J/m^2

Ra	0.201 um
Rmax	2.479 um

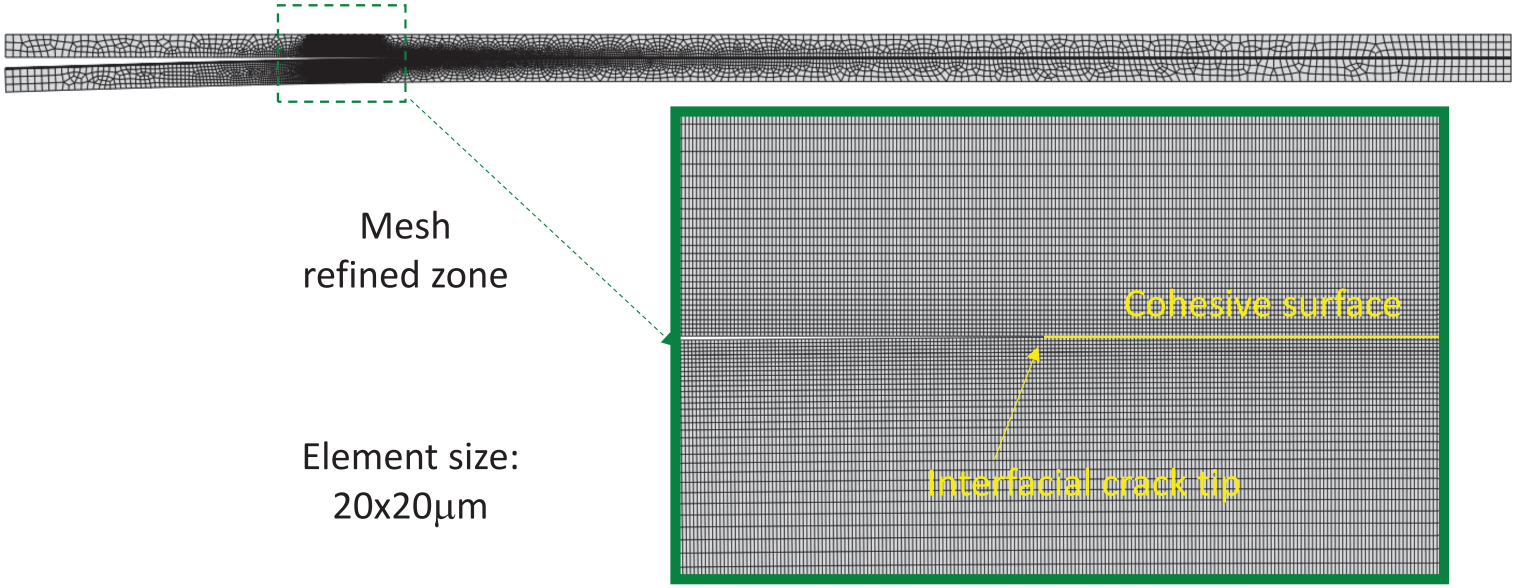


G_{ave}= 1.37 J/m^2

Ra	0.008 um
Rmax	0.08 um

5.4 Model Calibration, Cohesive Surfaces

- FEM package ABAQUS® V-6.14-2.
- 2-D 4-node quadrilateral linear plane strain elements (CPE4R).
- Single layer of Cohesive surface (Bilinear CZM).
- Refined element size of 20x20μm

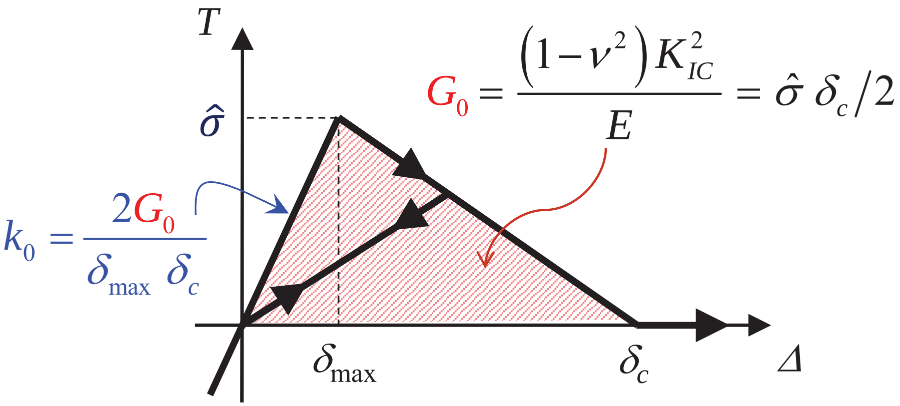


Mesh refined zone

Element size: 20x20μm

Calibration of Interfacial Cohesive Strength, $\hat{\sigma}$

Bilinear traction- separation curve



$\hat{\sigma} \sim 100kPa$

$G_o \sim 4 \quad J/m^2$

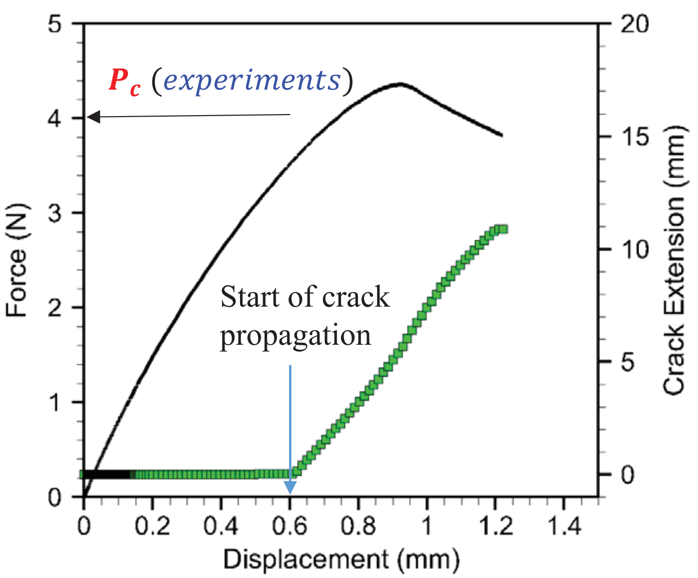
Calibration Scheme

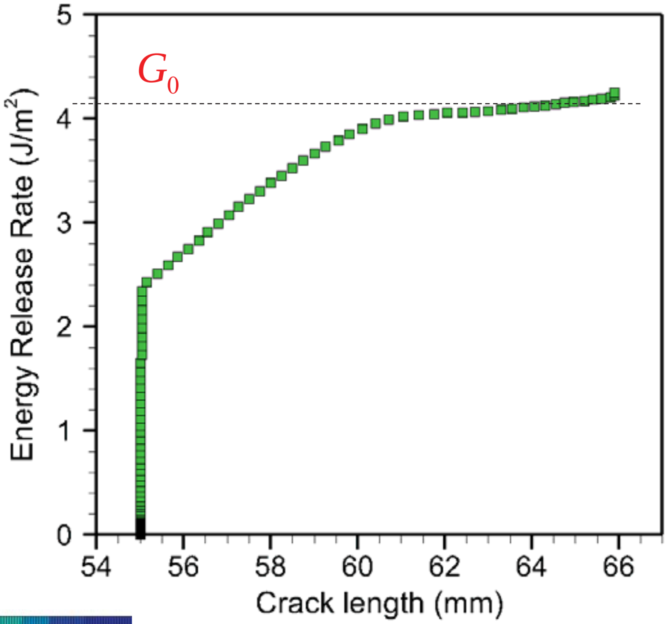
$G_0|_{FEM} \approx G_0|_{Experiment}$

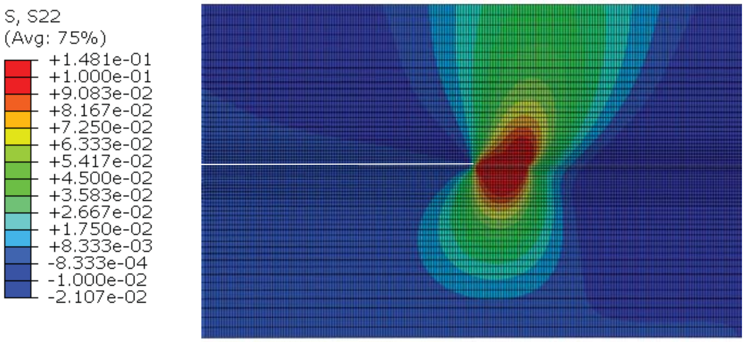
Matching Experimentally measured P_c by tuning $\hat{\sigma}$

Interfacial fracture parameters

Estimate of cohesive fracture parameters



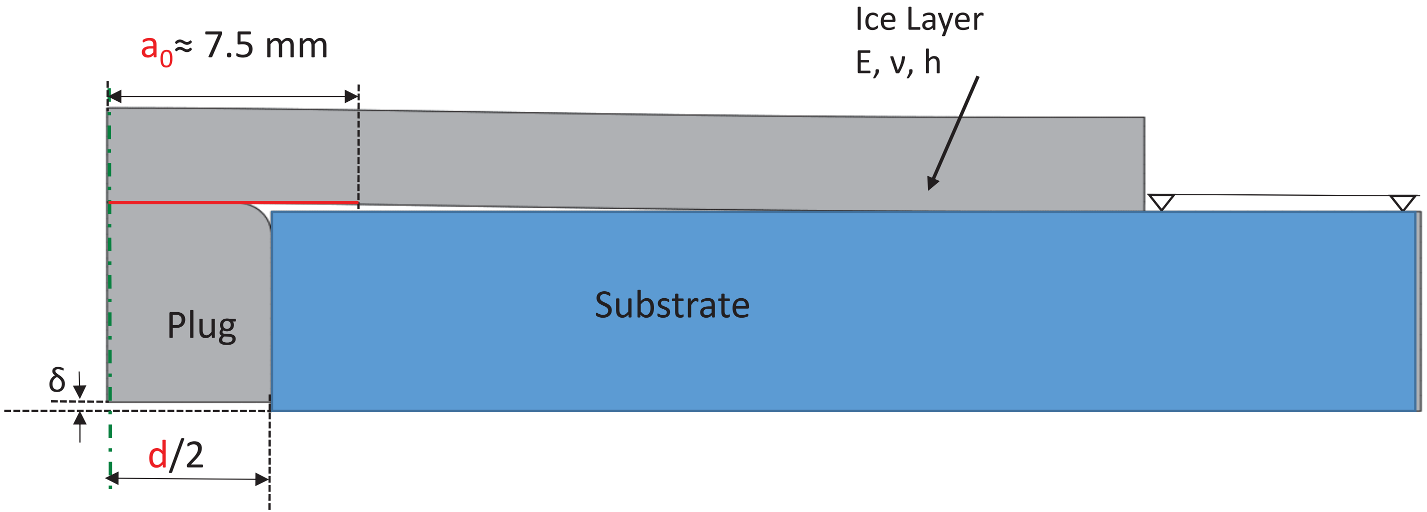




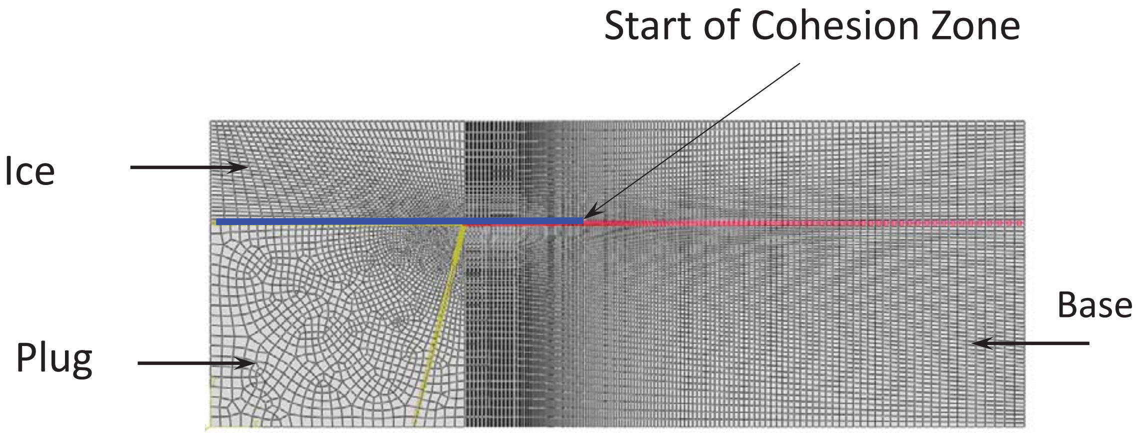
$$G_0 = 4 \ J/m^2$$
$$\hat{\sigma} = 0.1 MPa$$

Opening stress contours

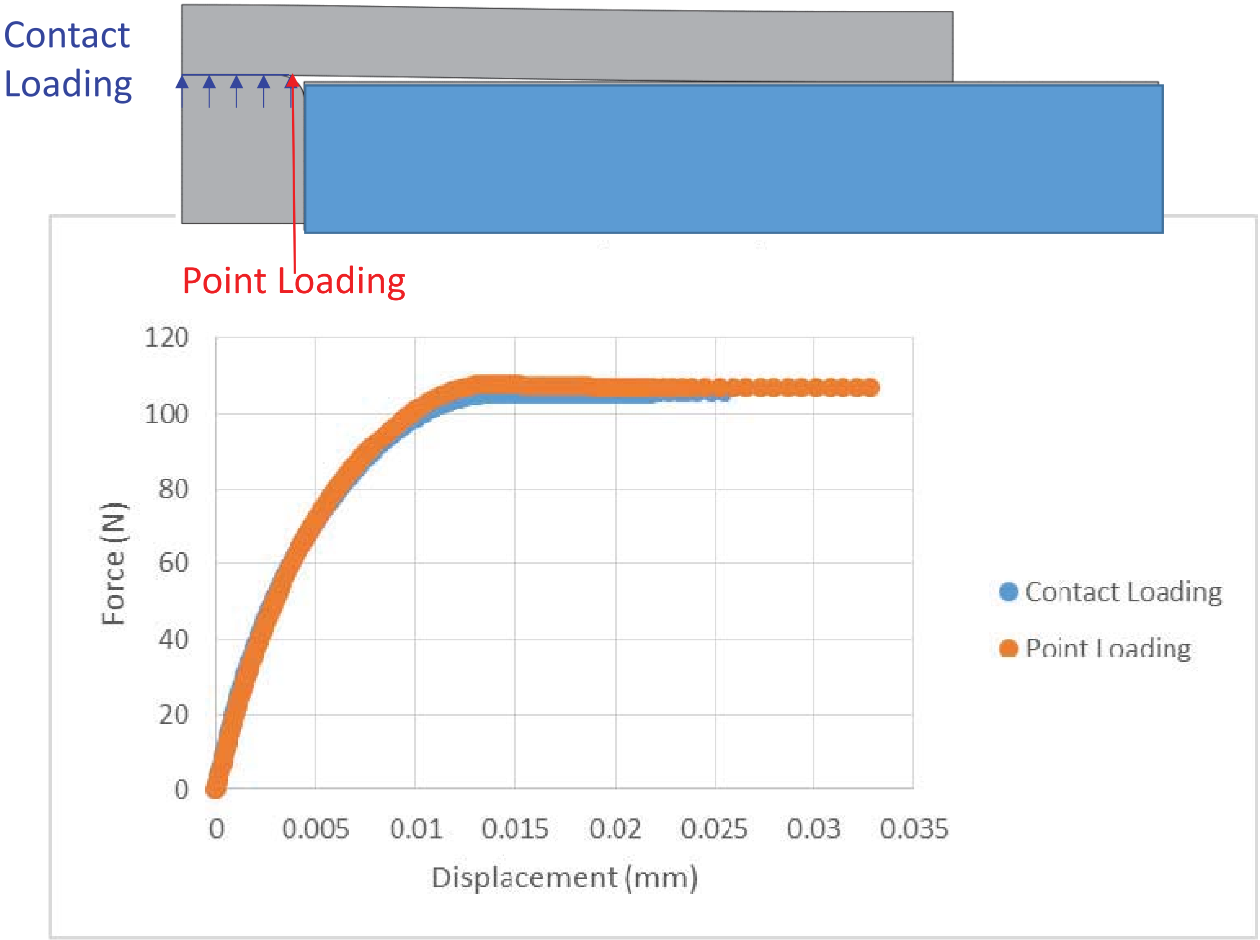
5.4 Parametric Study for the design of experimental apparatus (Modified Blister Test)



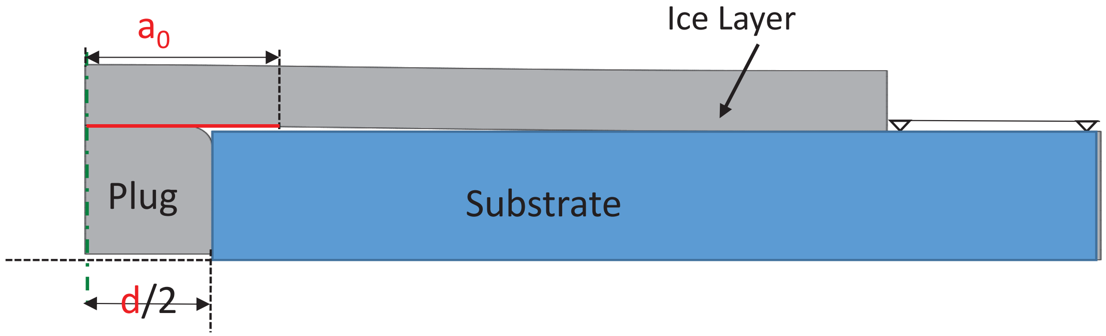
Material	E (GPA)	ν
Ice	9.8	0.3
Aluminum	70	0.33



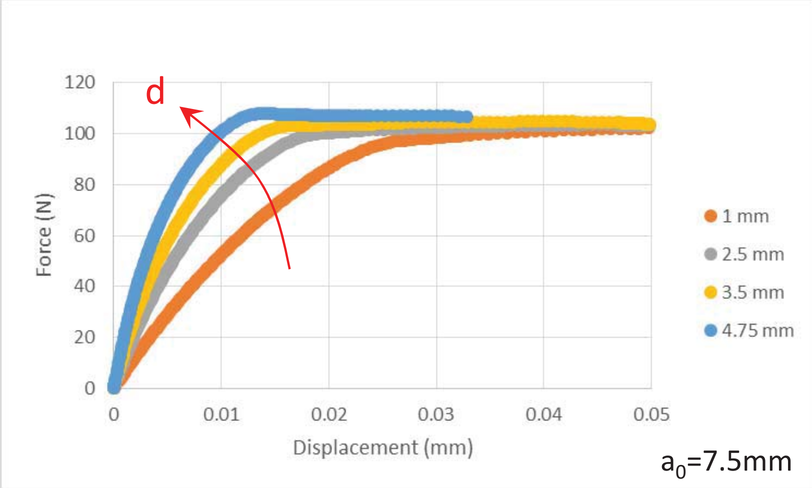
(a) Parametric Study (Loading configuration)



(b) Parametric Study (Crack aspect ratio)

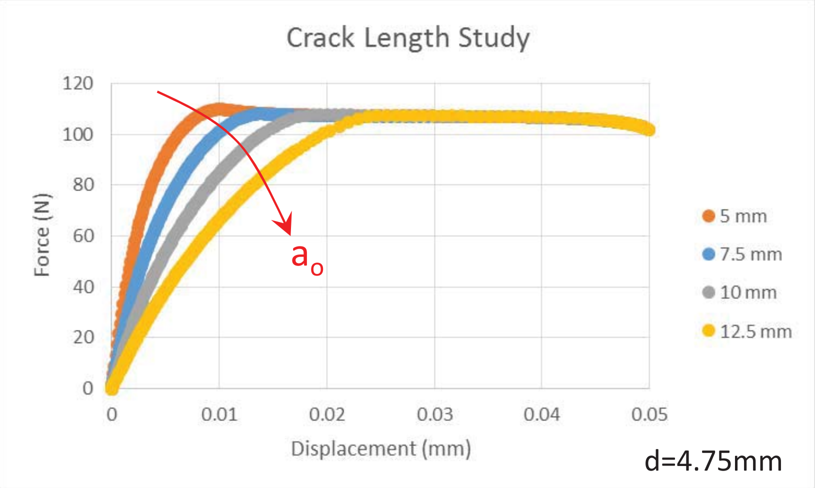


Role of plug diameter, d



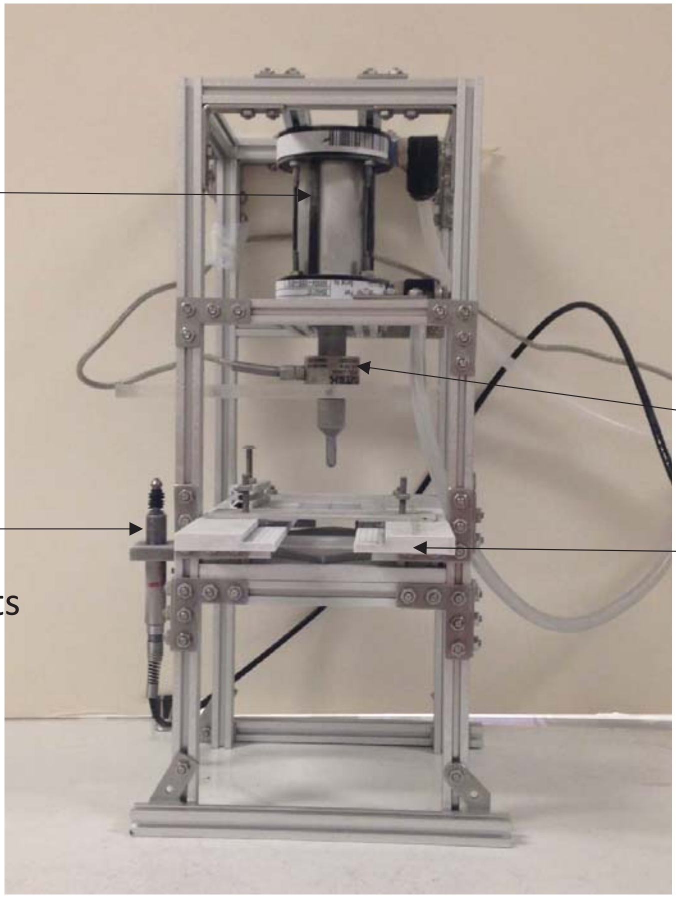
a_0=7.5mm

Role of crack length, a_o



d=4.75mm

5.5 Implementation of Experimental Apparatus

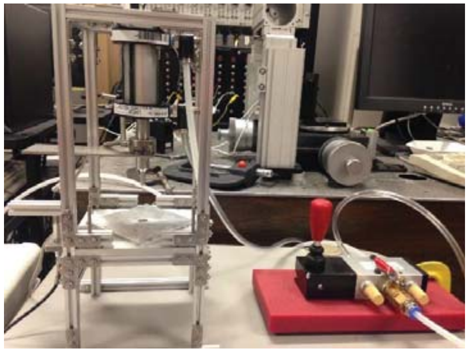


Pneumatic Cylinder

Load Cell

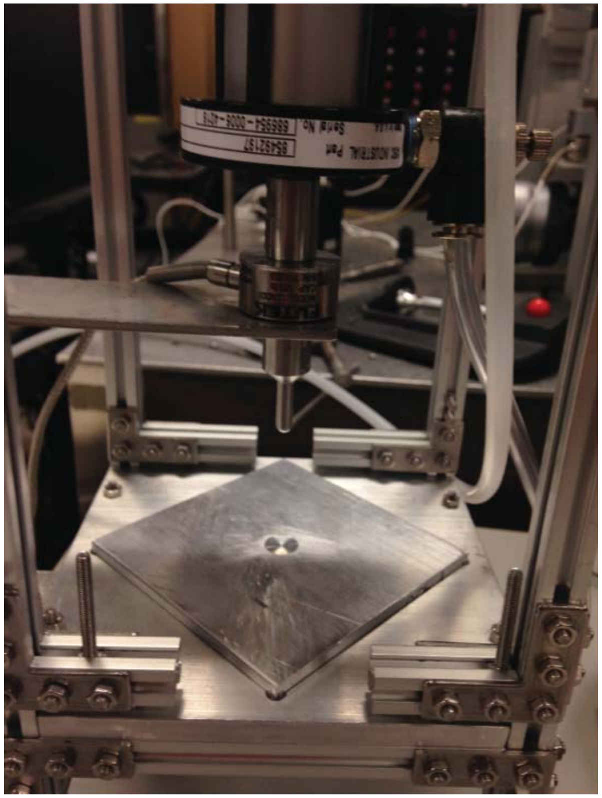
LVTD for Displacement Measurements

SCB Fixture

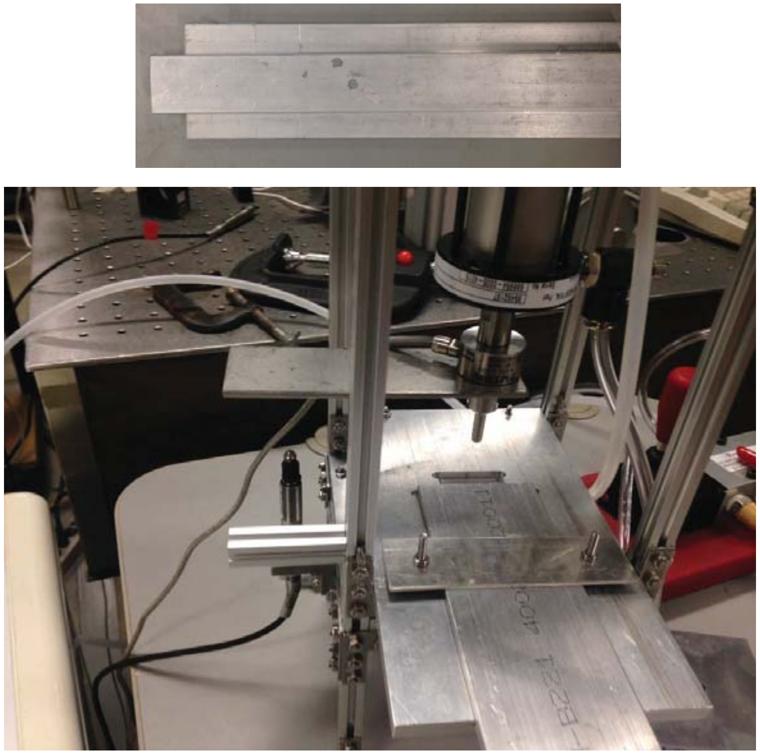


Pneumatic loading system
Dry Nitrogen Gas Operated

(a) Dual Loading Configuration (Blister and SCB)



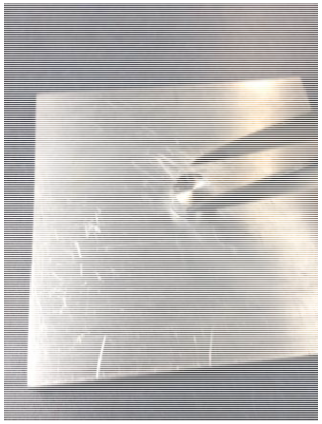
Blister test

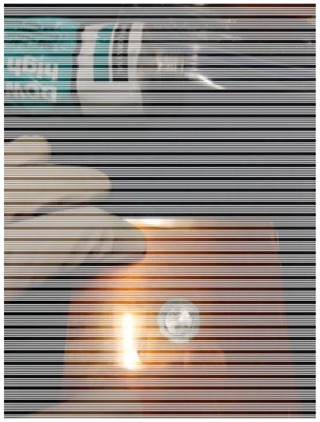


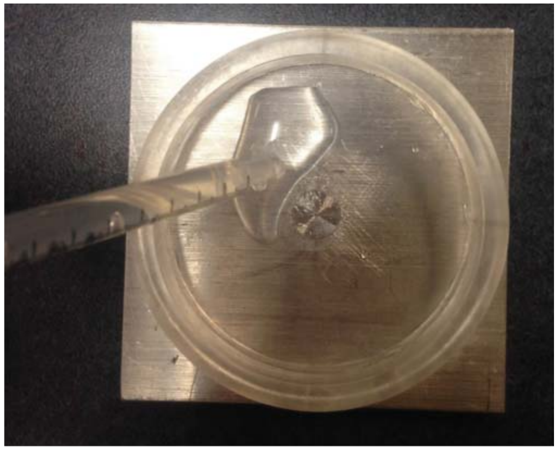
Single Cantilever beam

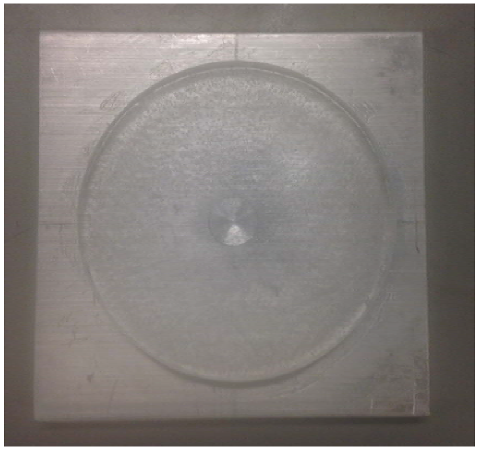
(b) Sample Preparation


(a) Methanol cleaning


(b) Greasing Plug

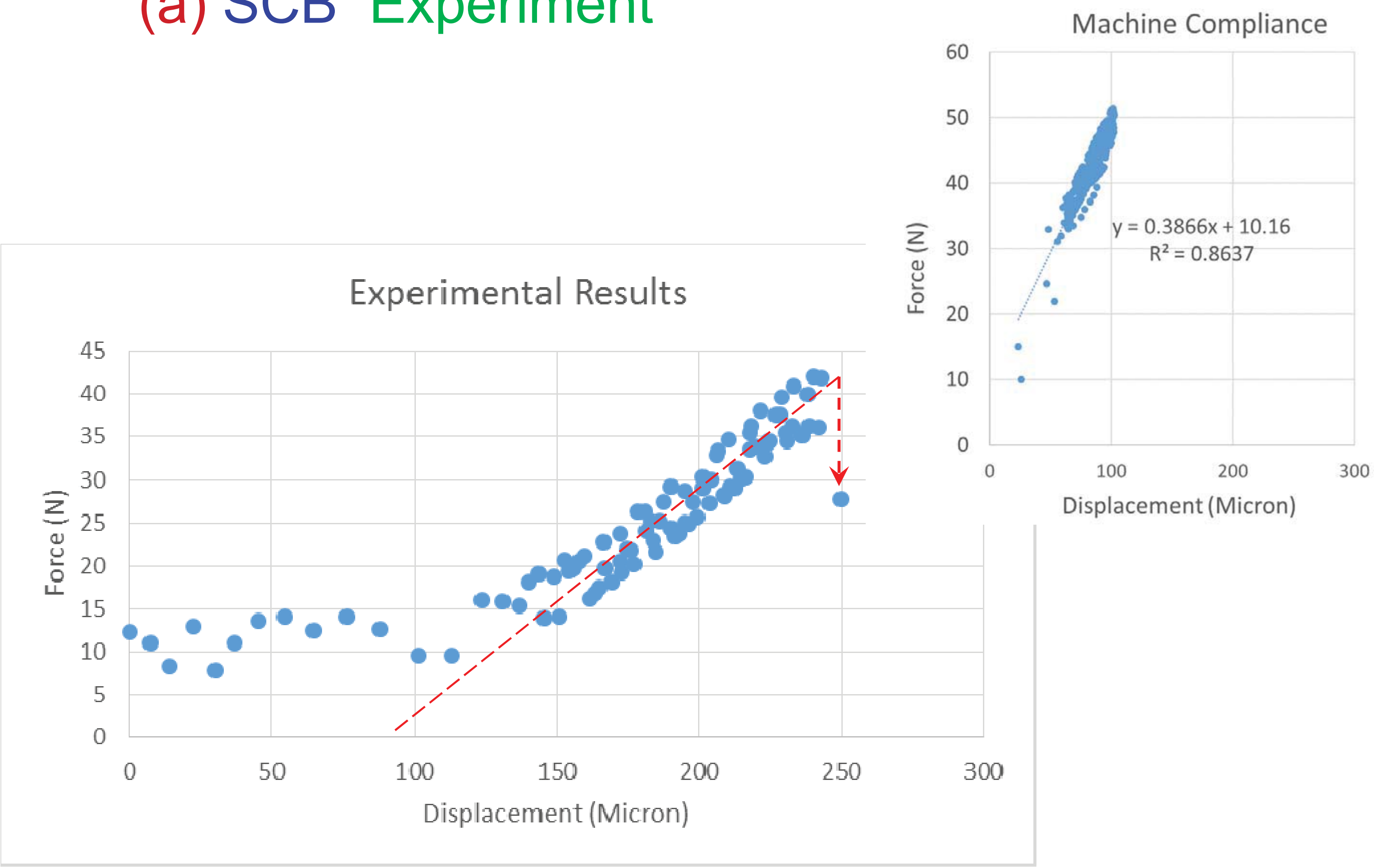

(c) Defining crack


(d) Controlled water volume


(e) Final sample

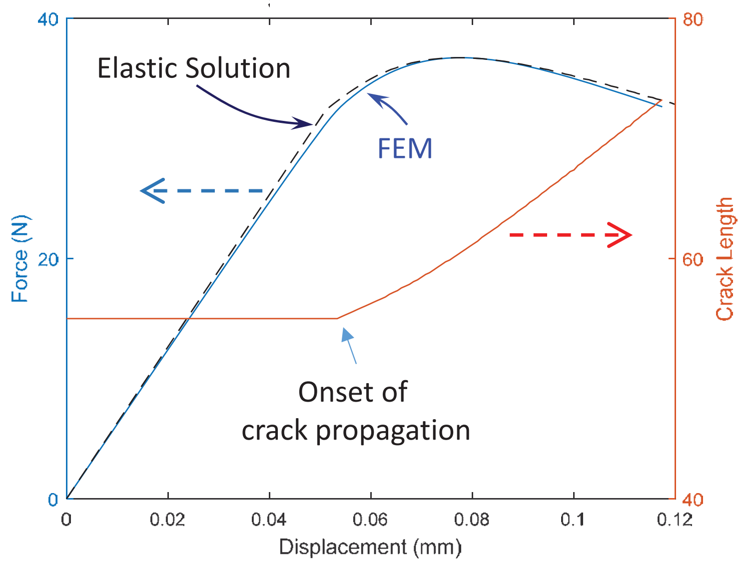
5.3.5 Preliminary Experimental & Model Results

(a) SCB Experiment

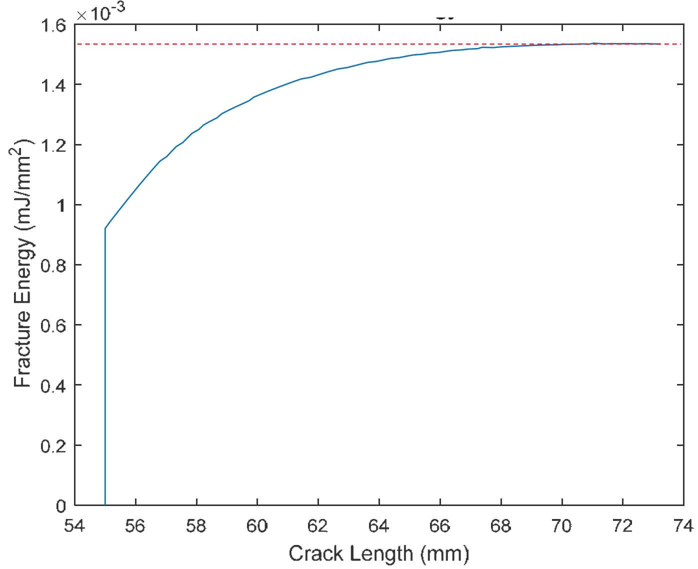


(b) SCB Modeling

Force-Displacement Curves



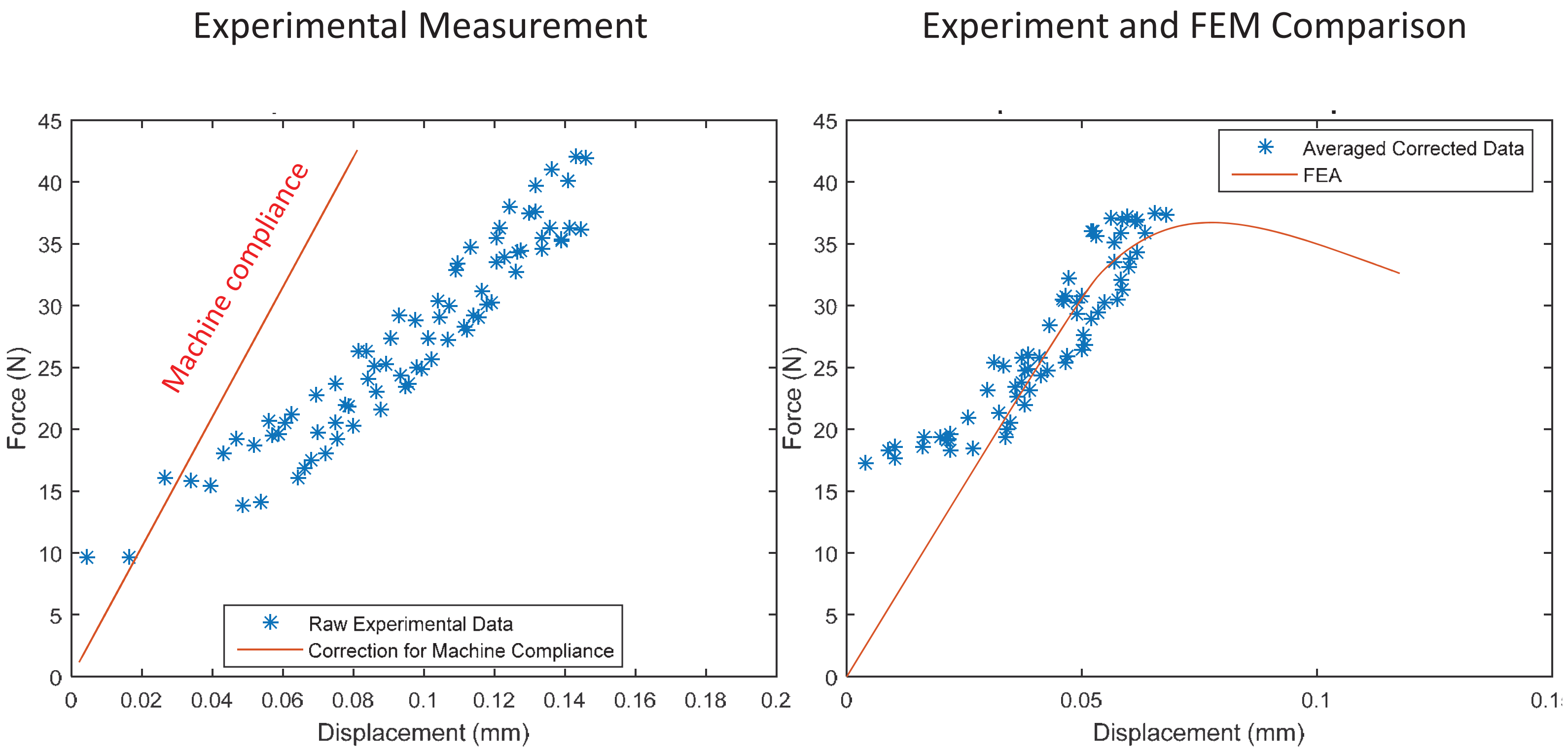
Fracture Energy

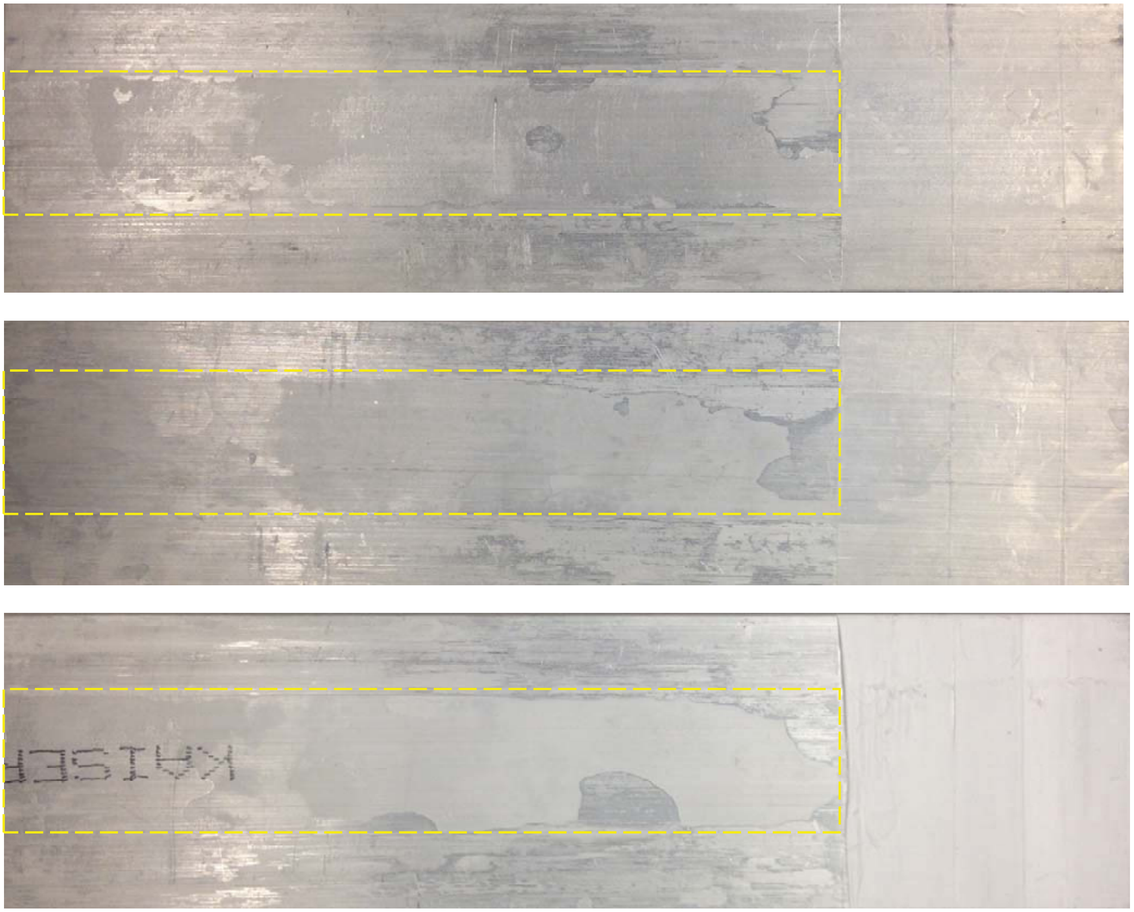


FEM results vs. Elastic Beam Theory $\delta = f(P, a) = \dfrac{Pa^3}{3EI}$

Cohesive parameters: $G = 1.5\ J/m^2\ ,\ \hat{\sigma} \simeq 1.0\ MPa$

(c) SCB Comparison

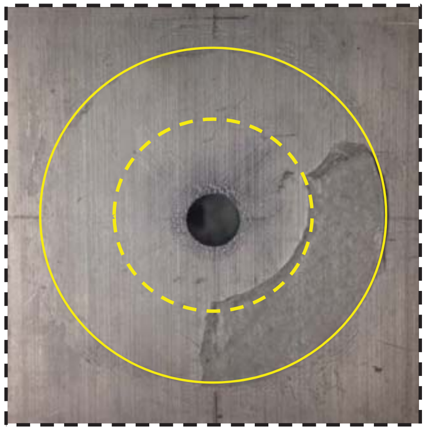




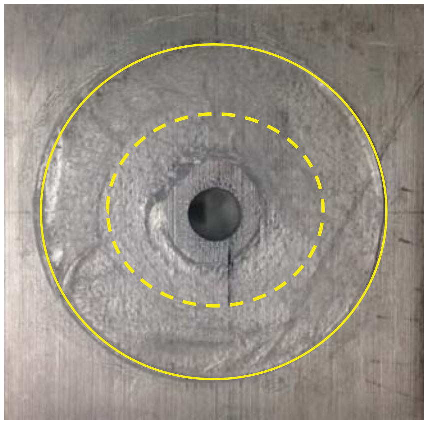
Failure is primarily adhesive

(d) Blister Test Experiment

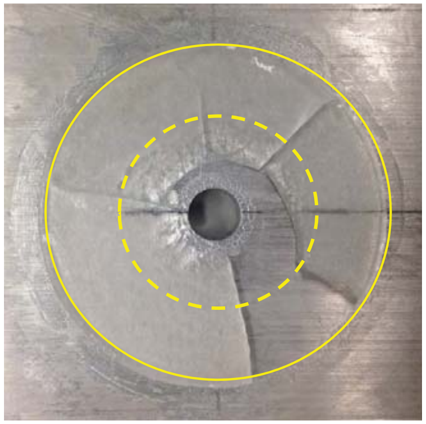
Test Group (06-23-2017)
Plate: As received 6061 AL
Ice thickness: 3 mm
Temp: -17.5 °C



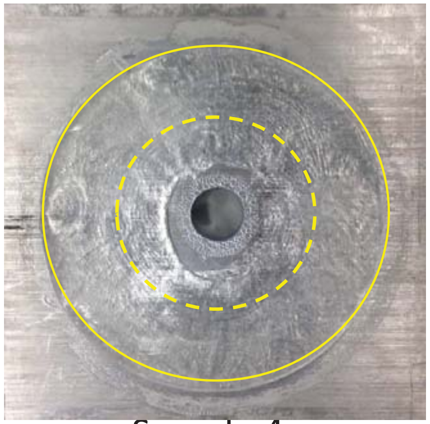
Sample 1



Sample 2

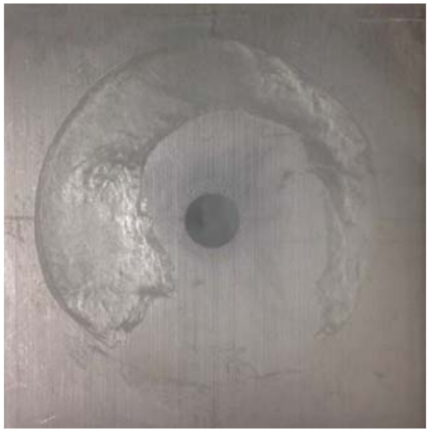


Sample 3



Sample 4

- Test Group (06-28-2017)
- Plate: As received 6061 AL
- Ice thickness: 4 mm
- Temp: -17.5 ° C



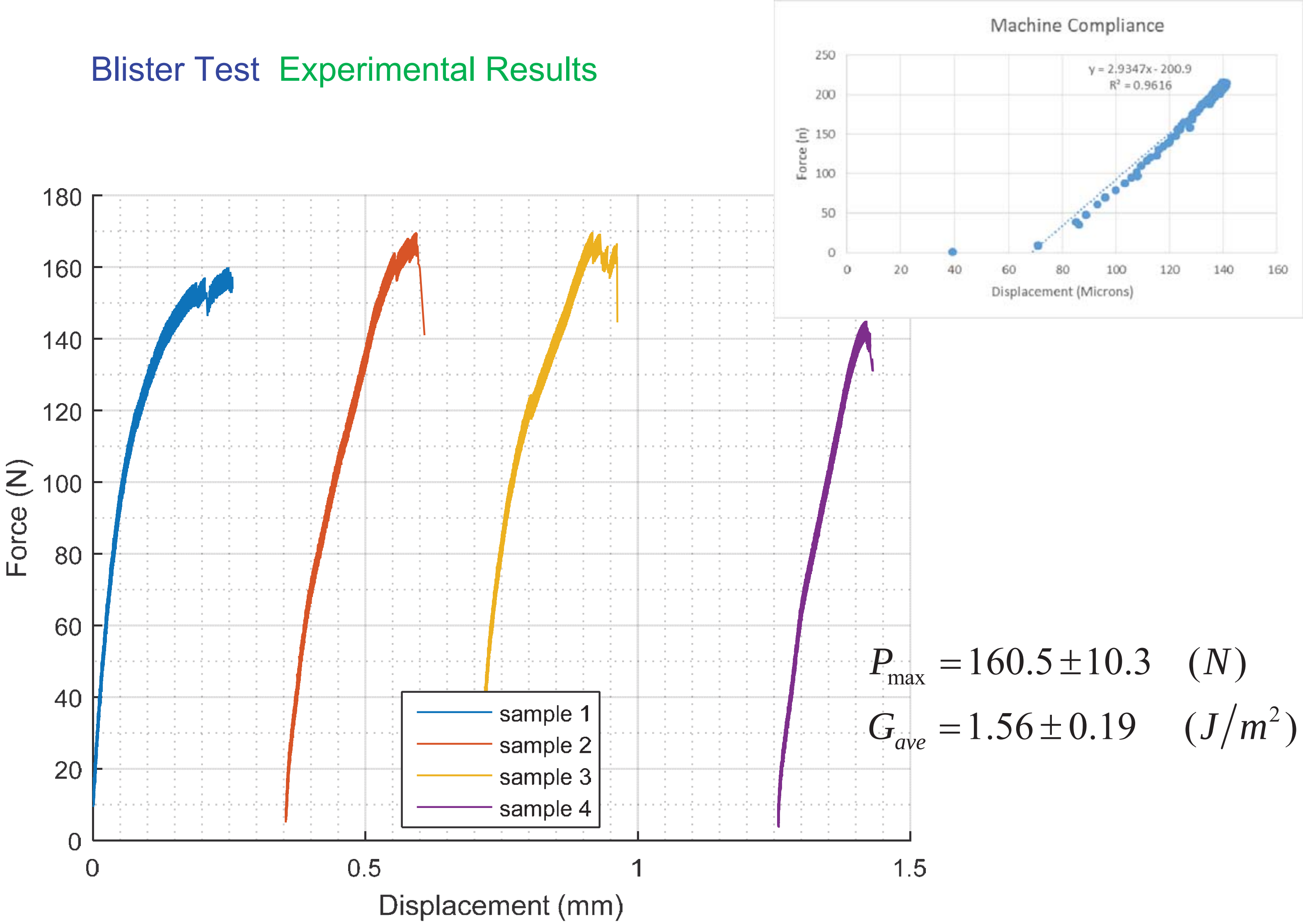
Sample 1

Sample 2

Sample 3

Sample 4

Blister Test Experimental Results

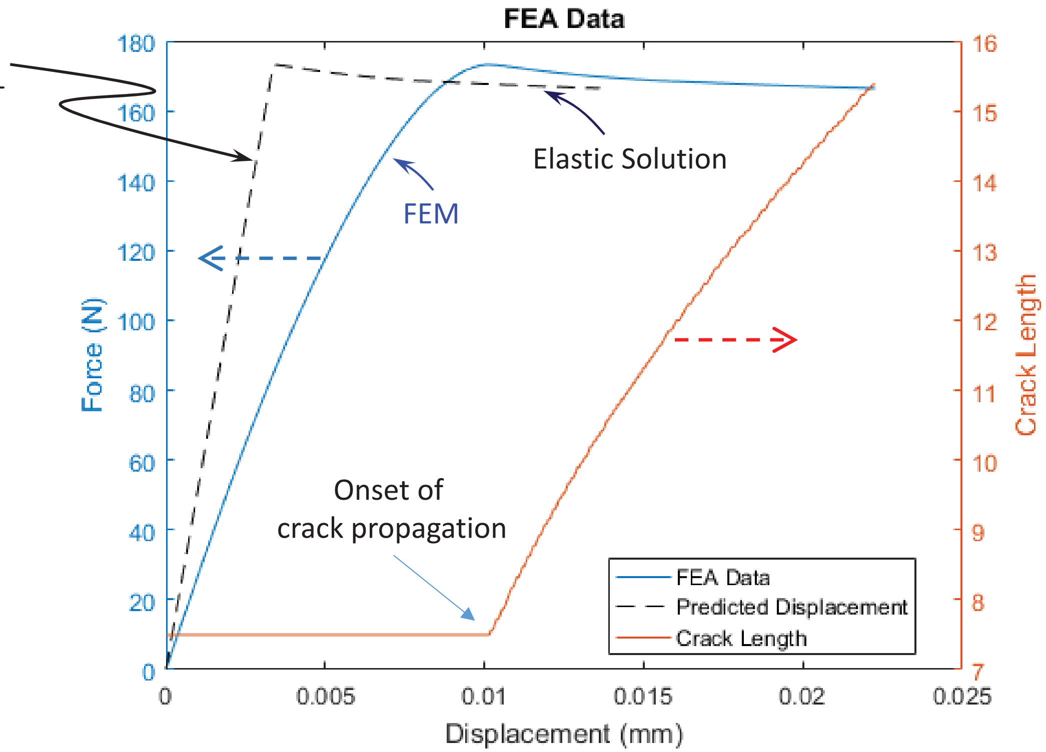


$$P_{\max} = 160.5 \pm 10.3 \quad (N)$$

$$G_{ave} = 1.56 \pm 0.19 \quad (J/m^2)$$

(e) Blister Test Modeling

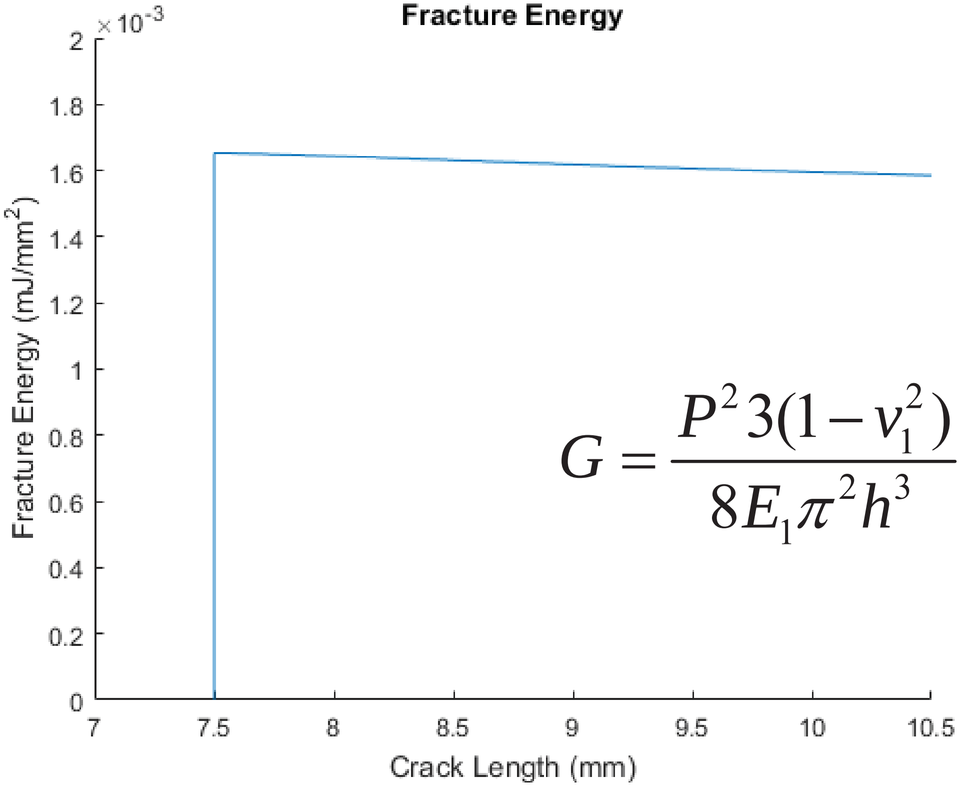
$$\delta = \frac{3(1-v_1^2)PR^2}{4\pi E_1 h^3}$$



Cohesive parameters:

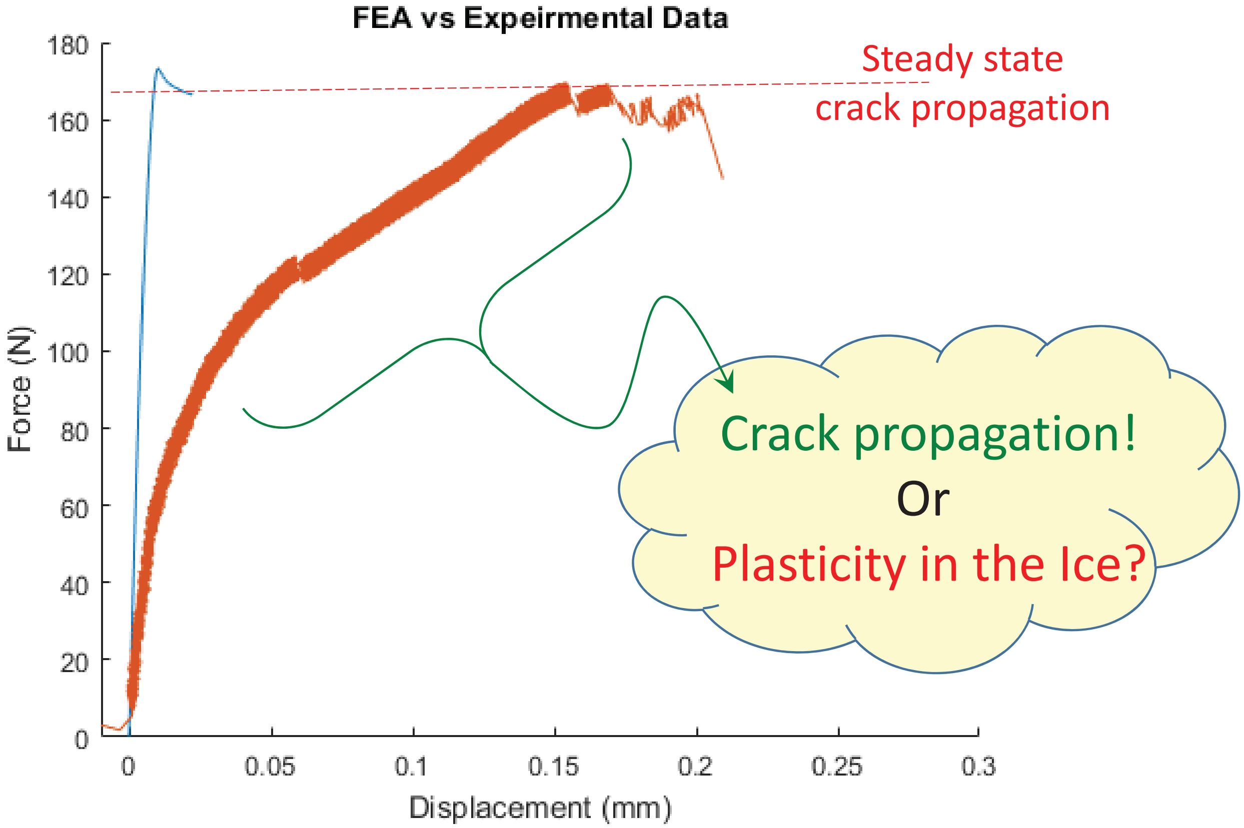
$$G = 1.5\ J/m^2\ ,\ \hat{\sigma} = 0.8\ MPa$$

(e) Blister Test Modeling



$$G = \frac{P^2 3(1 - v_1^2)}{8E_1 \pi^2 h^3}$$

(f) Blister Test Comparison Model & Exp.

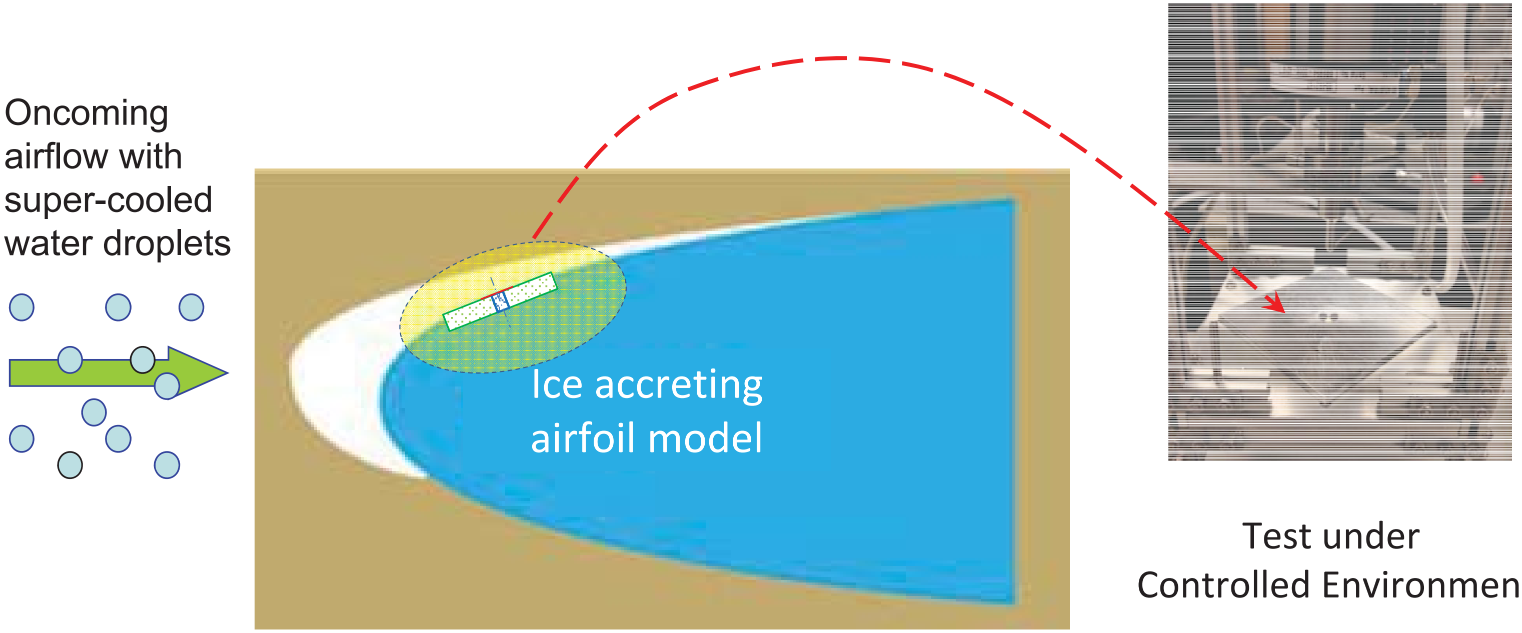


Steady state crack propagation

Crack propagation!
Or
Plasticity in the Ice?

6. Next Step

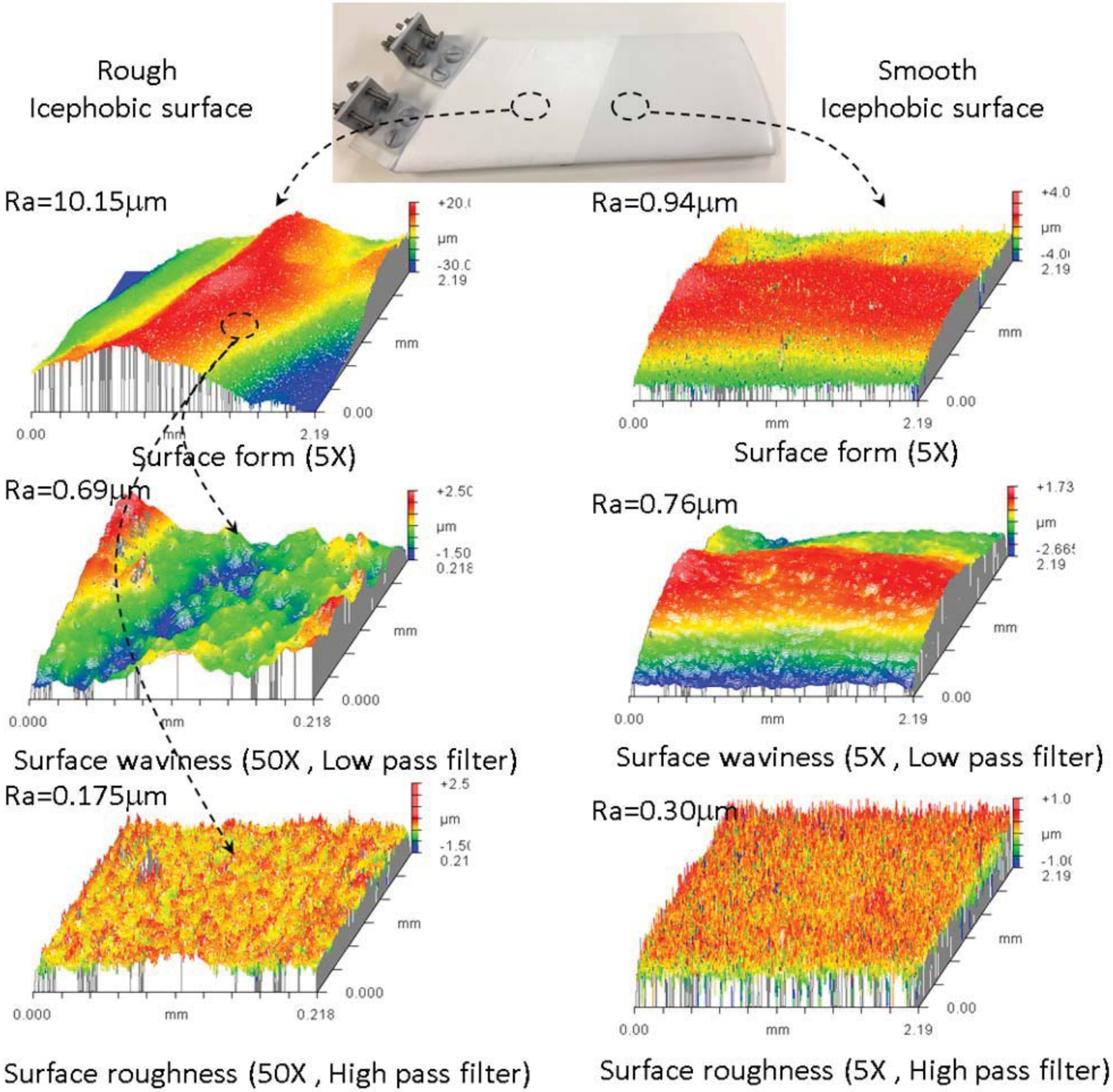
Oncoming
airflow with
super-cooled
water droplets



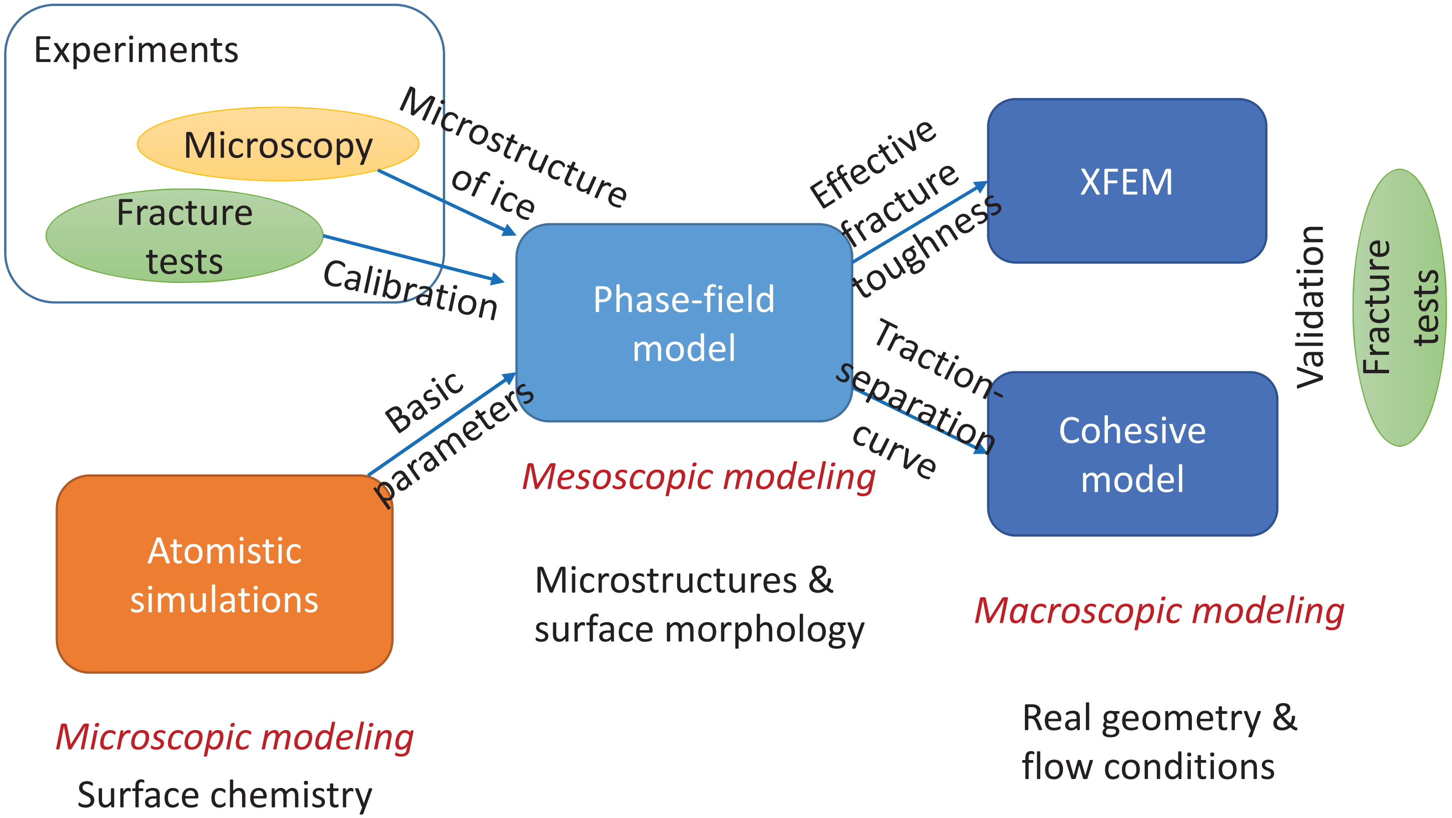
Test under
Controlled Environment

Grow Impact Ice in the Icing Tunnel
(Different flow and WC parameters)

Next Step


Rough
Icephobic surface
Smooth
Icephobic surface
Ra=10.15µm
+20.(
µm
-30.C
2.19
mm
0.00
0.00
mm
2.19
Surface form (5X)
Ra=0.94µm
+4.0
µm
-4.0(
2.19
mm
0.00
0.00
mm
2.19
Surface form (5X)
Ra=0.69µm
+2.5C
µm
-1.50
0.218
mm
0.000
0.000
mm
0.218
Surface waviness (50X , Low pass filter)
Ra=0.76µm
+1.73
µm
-2.66!
2.19
mm
0.00
0.00
mm
2.19
Surface waviness (5X , Low pass filter)
Ra=0.175µm
+2.5
µm
-1.5(
0.21
mm
0.000
0.000
mm
0.218
Surface roughness (50X , High pass filter)
Ra=0.30µm
+1.0
µm
-1.0(
2.19
mm
0.00
0.00
mm
2.19
Surface roughness (5X , High pass filter)

Relation with project components



Experiments

Microscopy

Fracture tests

Microstructure of ice

Calibration

Phase-field model

Basic parameters

Atomistic simulations

Effective fracture toughness

Traction-separation curve

XFEM

Cohesive model

Validation

Fracture tests

Mesoscopic modeling

Microstructures & surface morphology

Macroscopic modeling

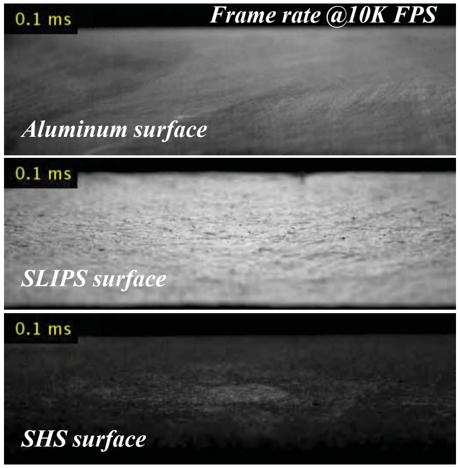
Real geometry & flow conditions

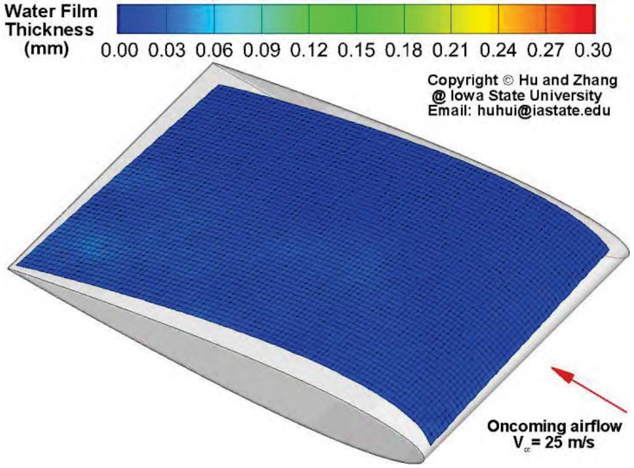
Microscopic modeling

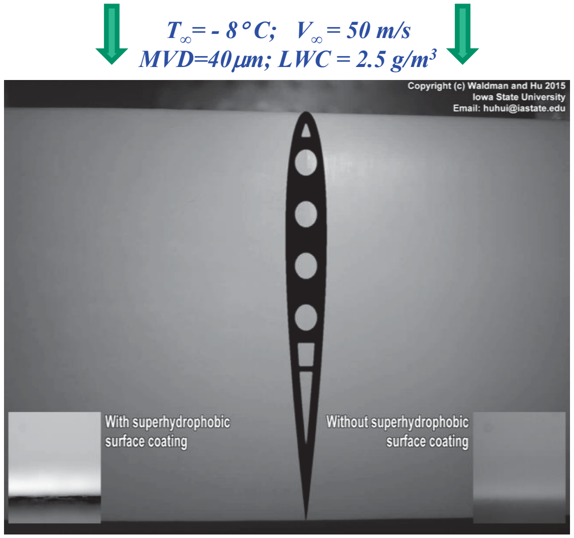
Surface chemistry

7. Ultimate Goal:

1. Characterize the adhesion and cohesion of impact ice accretion over various icephobic materials under different icing conditions.
2. Improve the performance of ice protection systems (IPS) by reducing ice adhesion and improving predictions of ice shedding from aircraft surfaces.







BIO-INSPIRED ICEPHOBIC MATERIALS/COATINGS FOR AIRCRAFT ICING MITIGATION

Dr. Hui HU

Martin C. Jischke Professor and Director

Aircraft Icing Physics and Anti-/De-icing Technology Laboratory

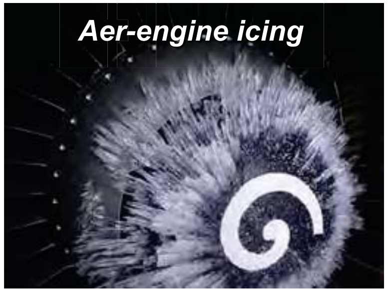
Department of Aerospace Engineering, Iowa State University
2251 Howe Hall, Ames, IA 50011-2271
Email: huhui@iastate.edu

IOWA STATE UNIVERSITY
Aircraft Icing Physics & Anti-/De-icing Technology Laboratory

☐ ISU INITIATIVE FOR ICING PHYSICS AND ANTI-/DE-ICING (I3-PAD)


Aircraft icing


Rotorcraft icing


Aer-engine icing

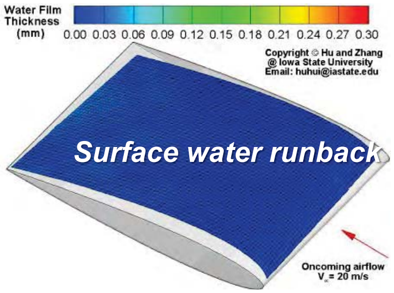

Wind turbine icing


Solar panel icing

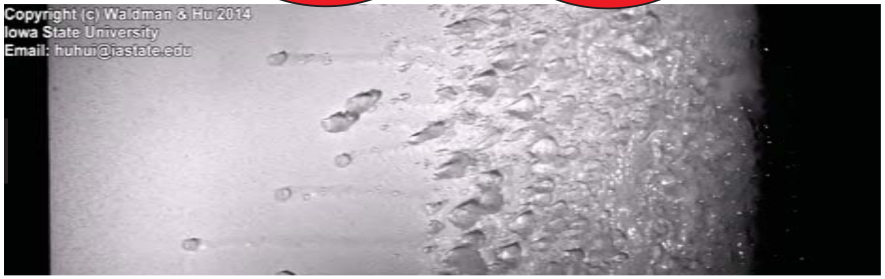
ISU CENTER FOR ICING PHYSICS & ANTI-/DE-ICING TECHNOLOGY

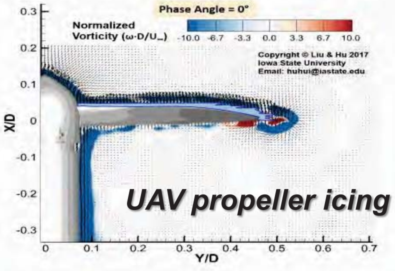
- NDE, MEMS sensors for in-flying icing detection
- Experimental aerodynamics & wind tunnel testing
- CFD & multiphase modeling
- UAS/MAV, Rotorcraft, wind turbine, power lines
- Syetem design and MDO for anti-/de-icing strategy
- Aero-structure designs for icing mitigation & protection.
- Smart materials, Micro & Nano Mechanics
- Super-hydrophobic coatings and surface engineering


Powerline icing



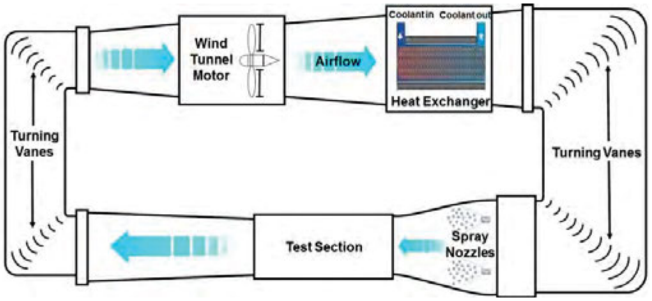
Surface water runback



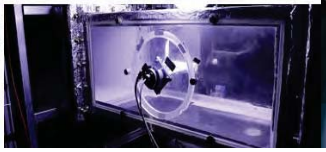


UAV propeller icing

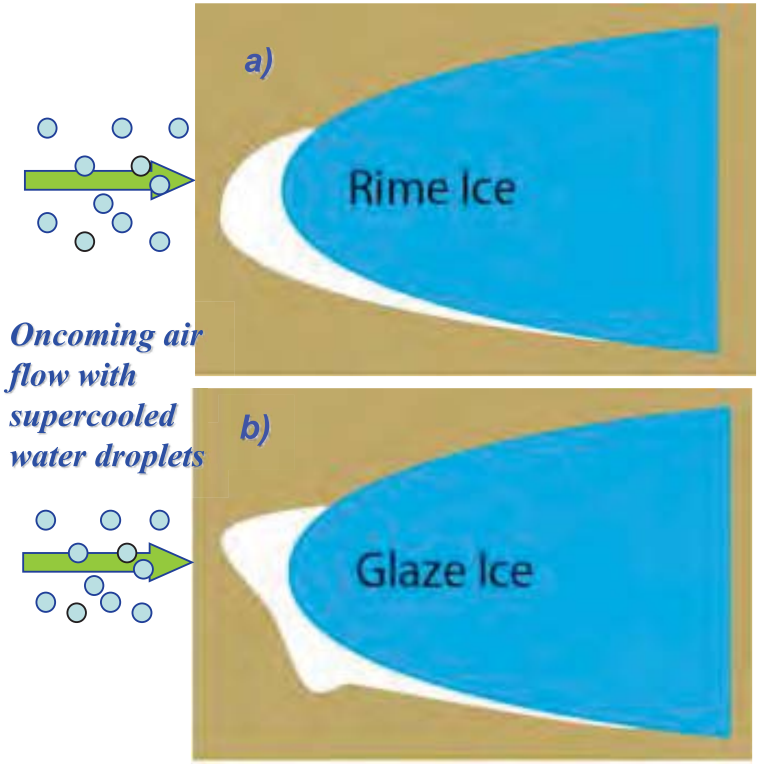
❑ ICING RESEARCH TUNNEL @ IOWA STATE UNIVERSITY (ISU-IRT)



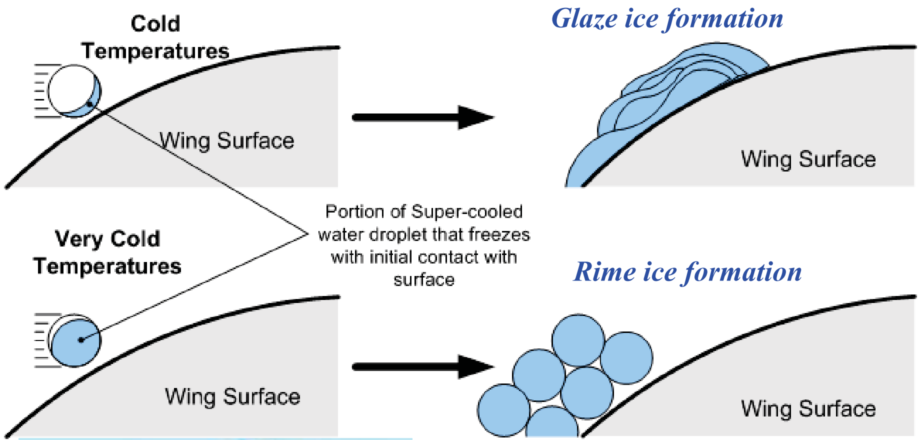
- *ISU Icing Research Tunnel (ISU-IRT),* ***originally donated by UTC Aerospace System (formerly Goodrich Corp.), is a new refurbished, research-grade multi-functional icing tunnel.***
- ***The working parameters of the ISU-IRT include:***
  - ***Test section:*** **0.4m×0.4m×2.0m**
  - ***Airflow Velocity:*** V_∞ **= 5 ~ 100 m/s;**
  - ***Air Temperature:*** T_∞ **= - 25 ℃ ~ 20 ℃;**
  - ***Droplet size:*** $D_{droplet}$ **= 10 ~ 100 μm;**
  - ***Liquid Water Content:*** **LWC = 0.1 ~ 10 g/m^3**
- ***The large LWC range allows ISU-IRT tunnel to be run over a range of conditions from dry rime icing to wet glaze icing.***

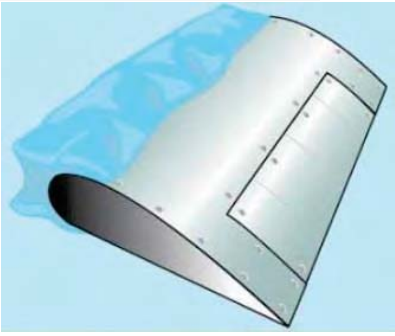


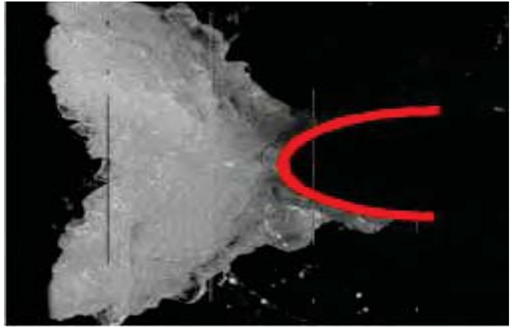
❑ AIRCRAFT ICING PHYSICS: RIME ICE AND GLAZE ICE

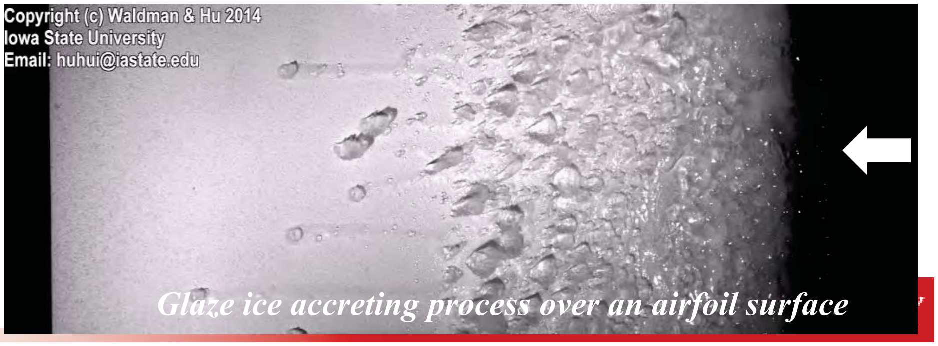


Oncoming air flow with supercooled water droplets







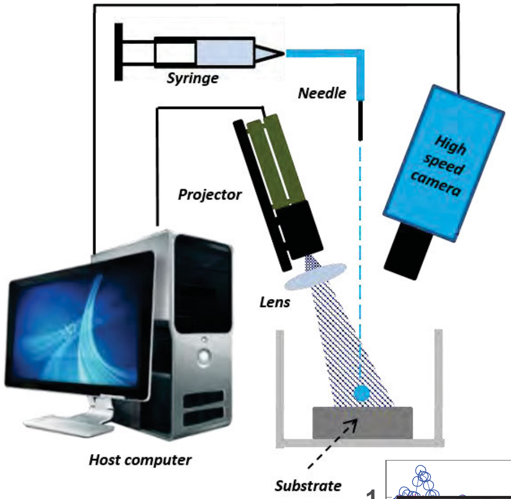


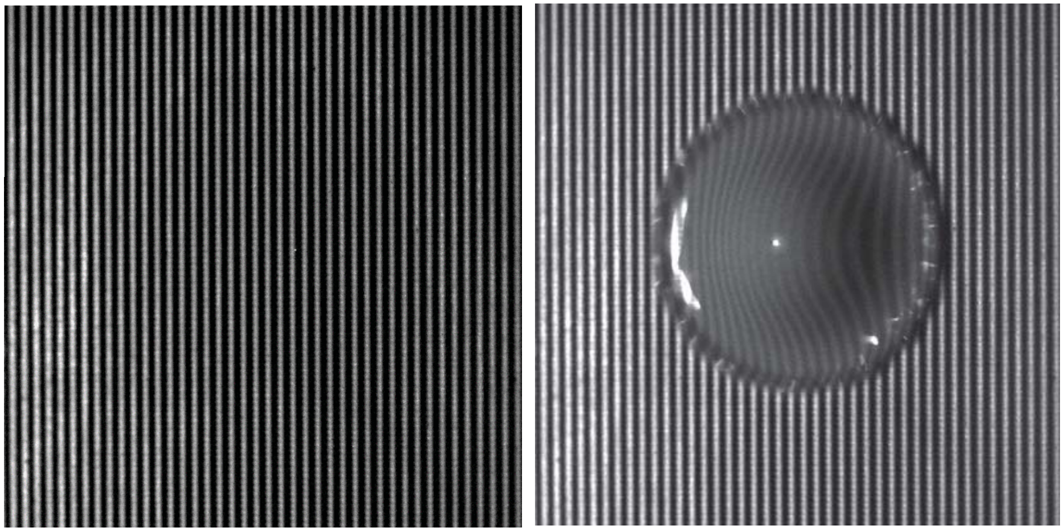
Glaze ice accreting process over an airfoil surface

- ***Glaze ice is the most dangerous type of ice.***
- ***Glaze ice form much more complicated shapes and are difficult to accurately predict.***
- ***Glaze ice is much more difficult to remove once built up on aircraft wings or wind turbine blades.***

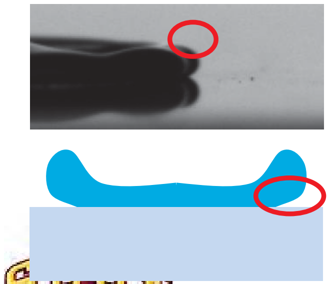
❑ DIP MEASUREMENTS OF SHAPE CHANGES OF IMPINGING DROPLETS

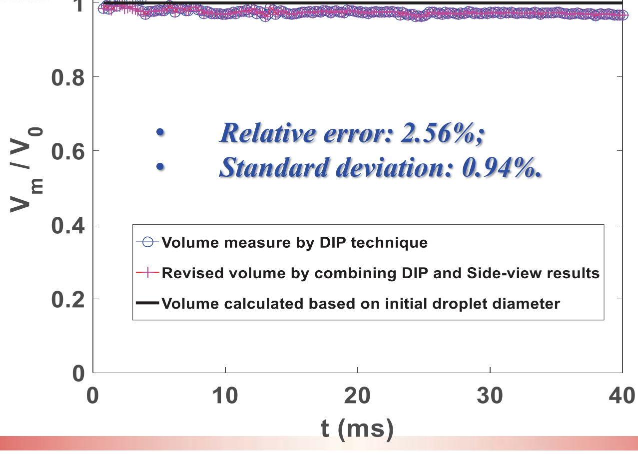
- *Diameter: $D = 2.4mm$*
- *Impact velocity: $V_{impact}=1.60m/s$*
- *Reynolds number: $Re = 4000$*
- *Weber number: $We = 90$*



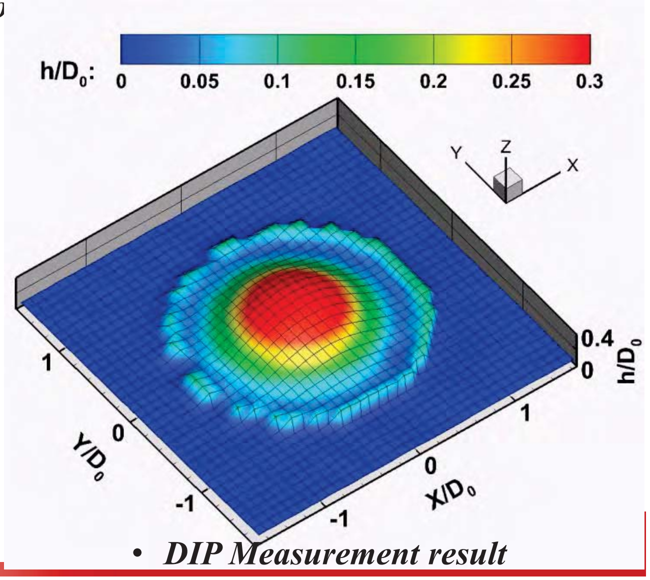


- *Reference image with no*



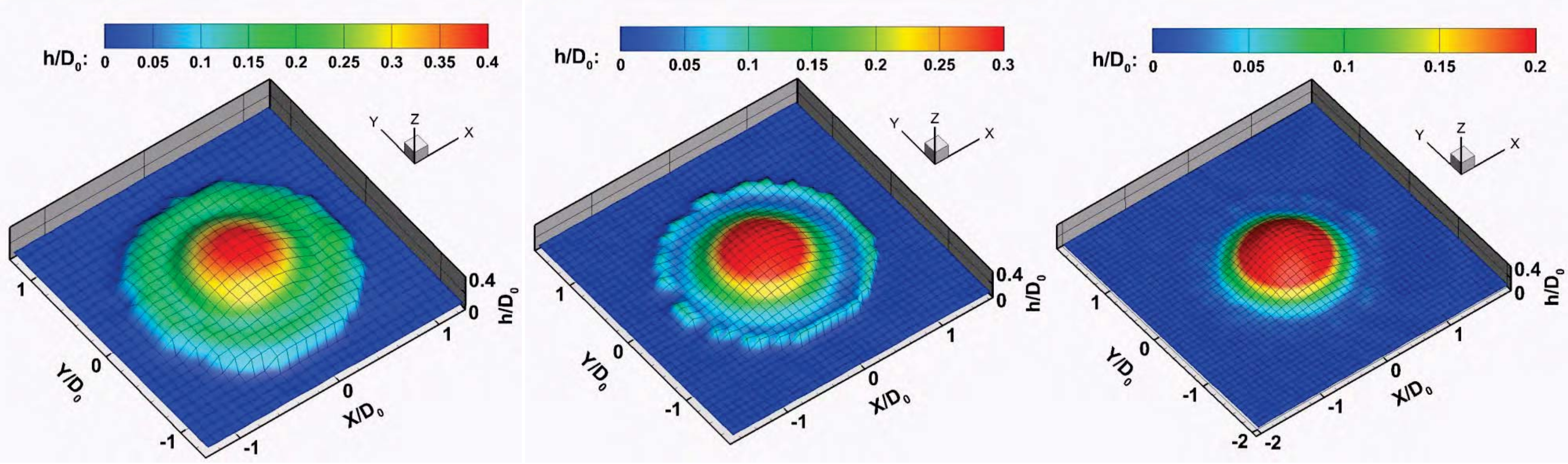


- *Relative error: 2.56%;*
- *Standard deviation: 0.94%.*



- *DIP Measurement result*

QUANTIFICATIONS OF DYNAMICS OF DROPLET IMPINGING PROCESS

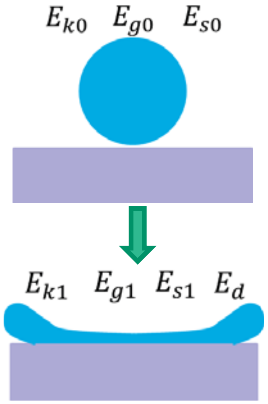


- ***Diameter: 2.4mm,***
- ***Impact velocity: 0.77m/s;***
- ***Re: =1900; We: = 25;***

- ***Diameter: 2.4mm,***
- ***Impact velocity: 1.60m/s;***
- ***Re = 4000; We = 110;***

- ***Diameter: 2.4mm,***
- ***Impact velocity: 2.12m/s;***
- ***Re = 5300; We = 195;***

- ***Energy budget during droplet impact process:***



$$\underbrace{E_{k0} + E_{p0} + E_{s0}}_{\textbf{Before impact}} = \underbrace{E_k + E_p + E_s + W}_{\textbf{During impact}}$$

$$E_{k0} = \frac{1}{2}mv^2 = \frac{\pi}{12}\rho v^2 d_0^{\,3}$$

$$E_{g0} = mgh = \frac{\pi}{12}\rho g d_0^{\,4}$$

$$E_{s0} = \gamma S = \pi d_0^{\,2}\gamma$$

$$E_k = \frac{1}{2}mv^2 \quad E_g = mgh$$

$$W = f(v,\mu)$$

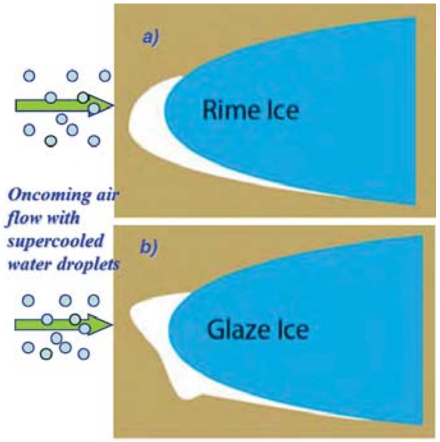
$$\boxed{E_s = \gamma\left[A - \frac{\pi d^2}{4}\cos\theta\right]}$$

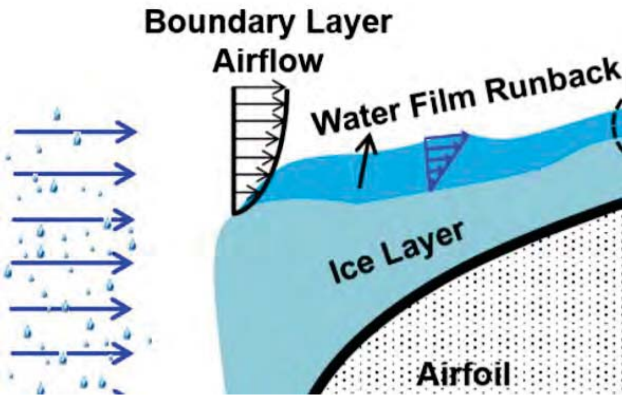
We	***E_0 ($10^{-6}J$)***	***E_{df} ($10^{-6}J$)***	***E_{d1} ($10^{-6}J$)***	***E_{d1}/E_{df}***
25	***3.16***	***2.69***	***2.30***	***85.2%***
110	***10.11***	***9.06***	***8.67***	***95.6%***
195	***16.71***	***14.67***	***14.22***	***96.9%***

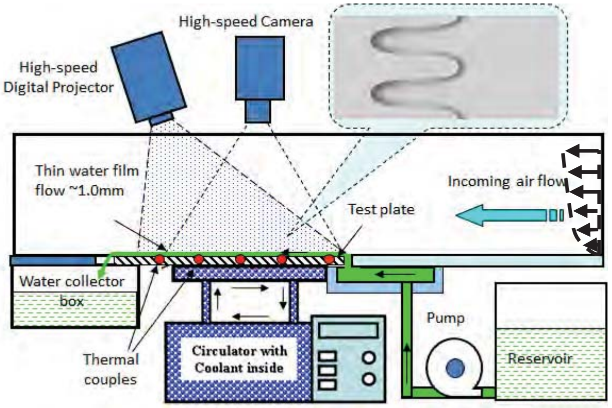
Viscous dissipation function : $\phi \propto \mu U_0^{\;2}$

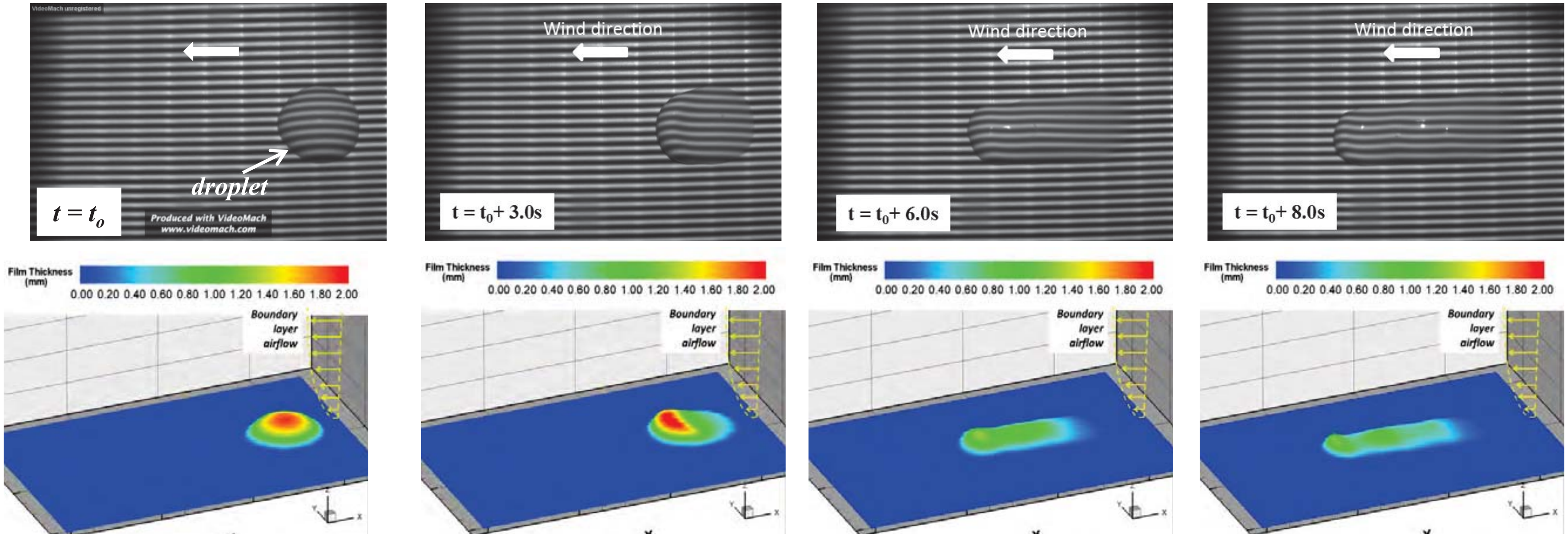
- The energy dissipated during the spreading stage would increase as the increase of the We;
- (S. Chandra & C. T. Avedisian , Proc. R. Soc., 1991)

❑ TRANSIENT BEHAVIOR OF WIND–DRIVEN FILM/RIVULET FLOWS



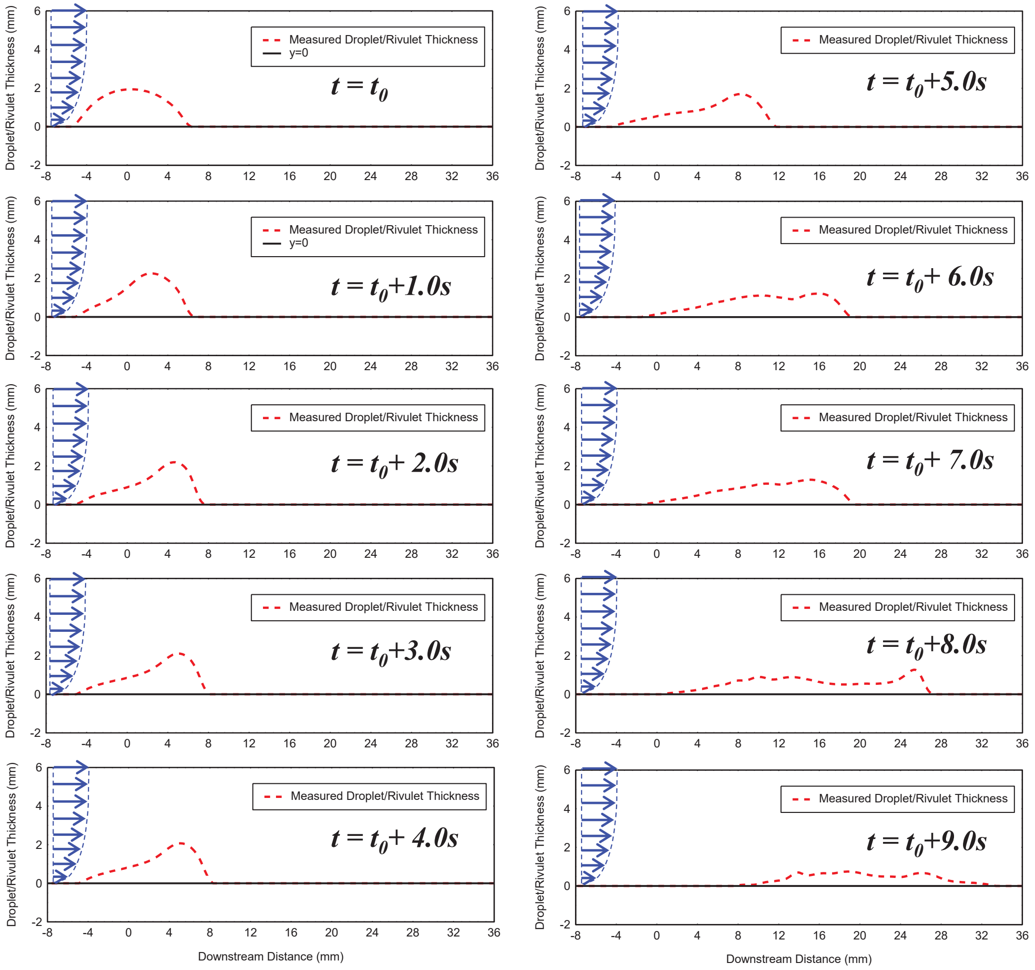


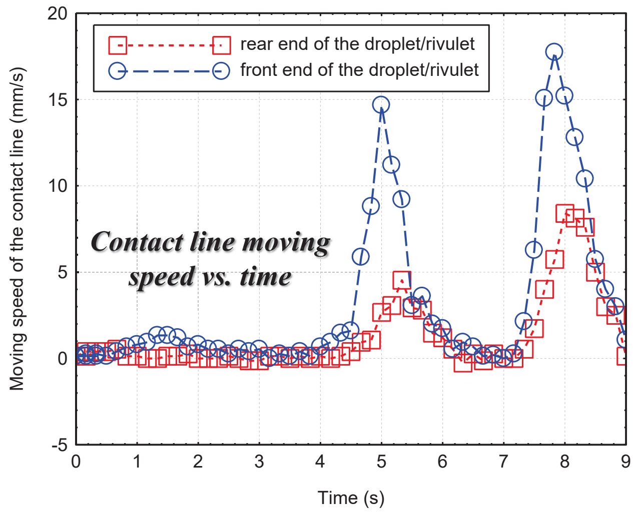




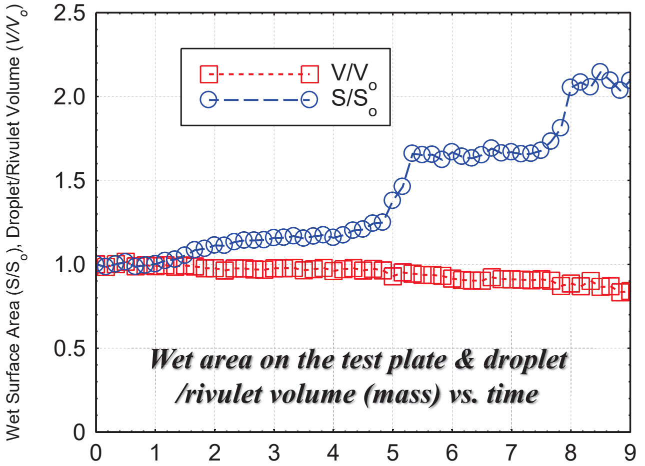
- ***H. Hu, B. Wang, K. Zhang, W. Lohry and S. Zhang,*** *"Quantification of Transient Behavior of Wind-Driven Surface Droplet/Rivulet Flows by using a Digital Fringe Projection Technique", Journal of Visualization, Vol. 18, No.4, pp705-718, 2015*

❑ TIME EVOLUTION OF THE WIND-DRIVEN DROPLET/RIVULET FLOW





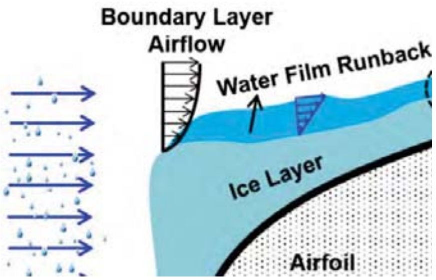
Contact line moving speed vs. time

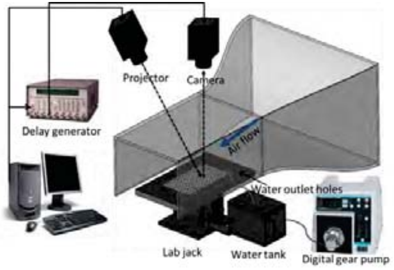


Wet area on the test plate & droplet /rivulet volume (mass) vs. time

- ***H. Hu, B. Wang, K. Zhang, W. Lohry and S. Zhang,*** *"Quantification of Transient Behavior of Wind-Driven Surface Droplet/Rivulet Flows by using a Digital Fringe Projection Technique", Journal of Visualization, Vol. 18, No.4, pp705-718, 2015*

❑ WIND–DRIVEN FILM/RIVULET FLOWS (DRY SURFACE CONDITION)

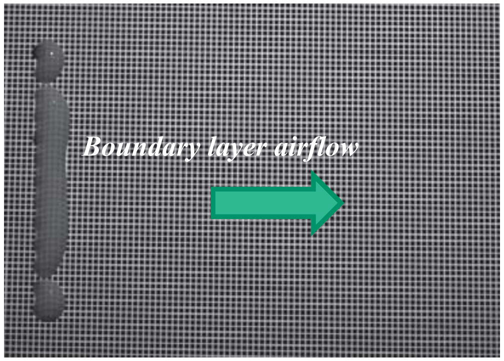


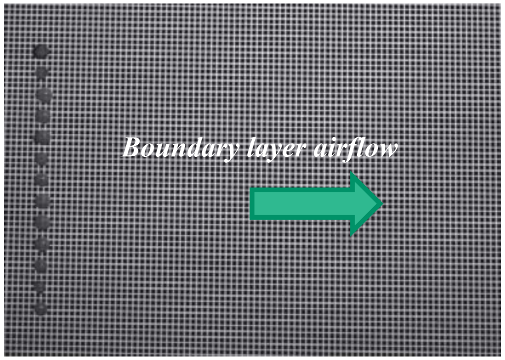


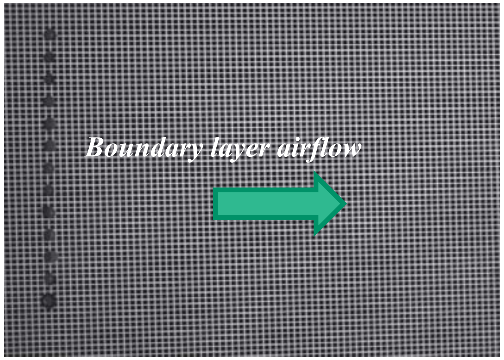
Experiment set up for DIP Measurements

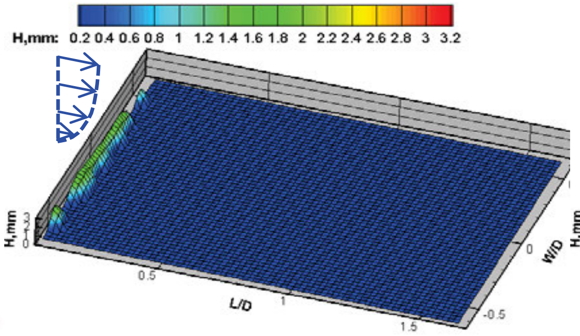
Effects of various important parameters:

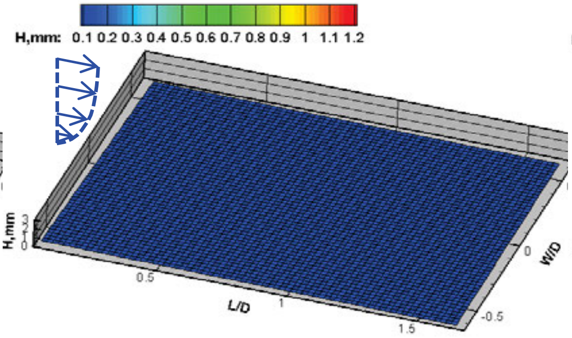
- ***Temperature of the surface***
- ***Thermal conductivity of the subtracts***
- ***Roughness of the test surfaces***
- ***Surface hydrophobicity***
- ***Coatings or nano-structures on the test surfaces***

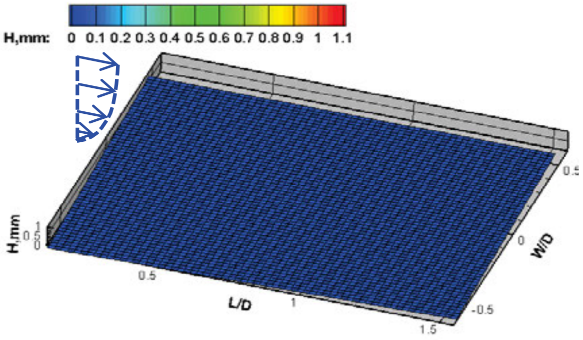












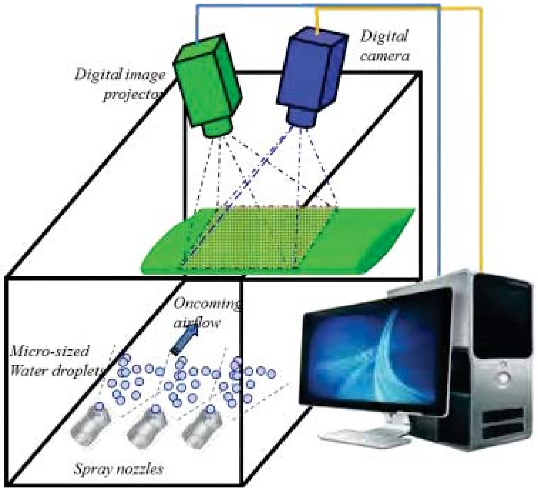
- *Water flow rate:* *Q= 100 ml/min*
- *Free stream airflow:* *V_∞=10m/s*

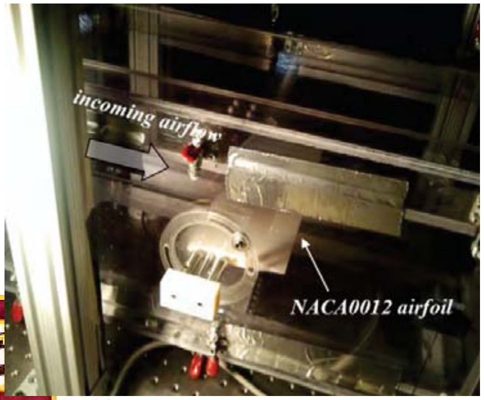
- *Water flow rate:* *Q= 100 ml/min*
- *Free stream airflow:* *V_∞=15m/s*

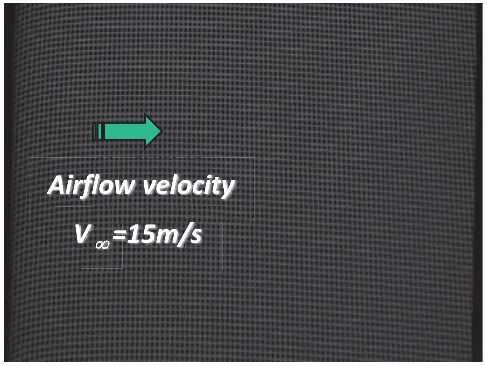
- *Water flow rate:* *Q= 100 ml/min*
- *Free stream airflow:* *V_∞=20m/s*

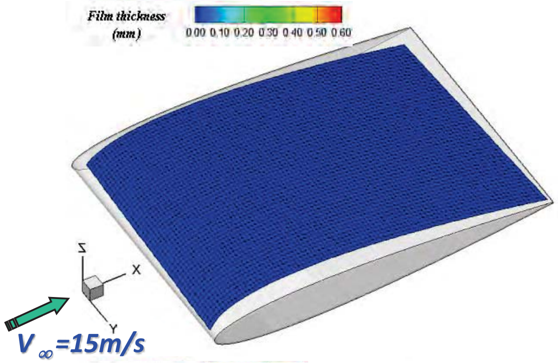
☐ DYNAMIC WATER RUNBACK OVER AN AIRFOIL SURFACE

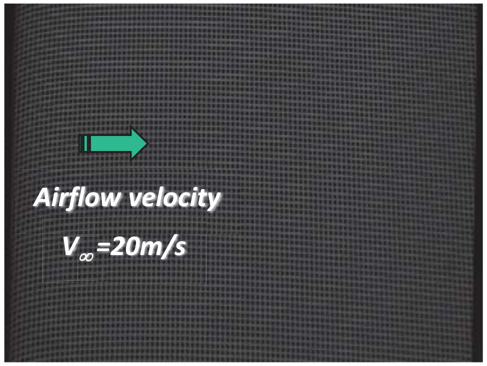
- *Test Conditions:*
  - *Angle of attack:* *α ≈ 0.0 deg.*
  - *Temperature:* *T ≈ 20 °C.*
  - *LWC Level :* *LWC =5.0 g/m³*
  - *Frame rate :* *f = 30 Hz*

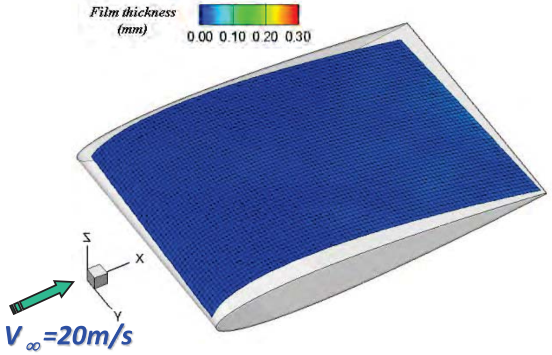


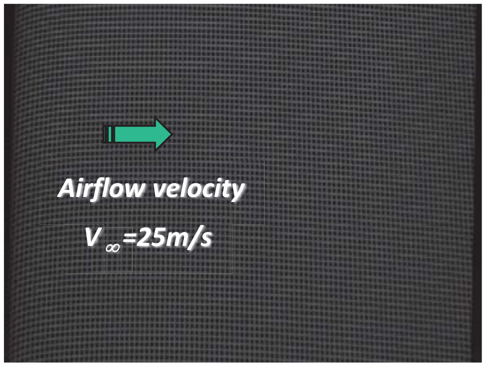


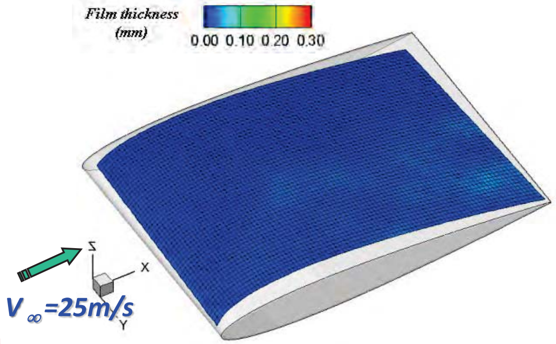






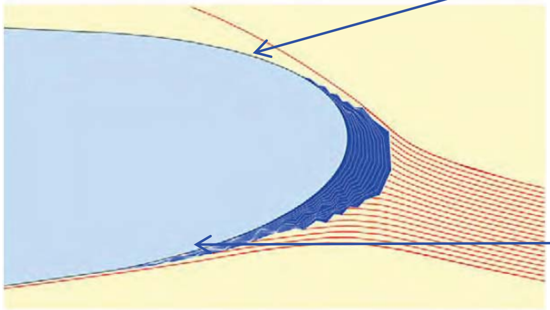


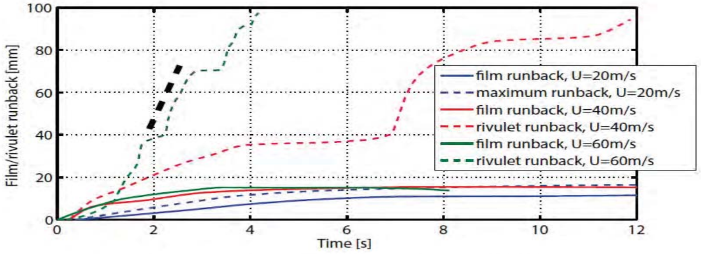


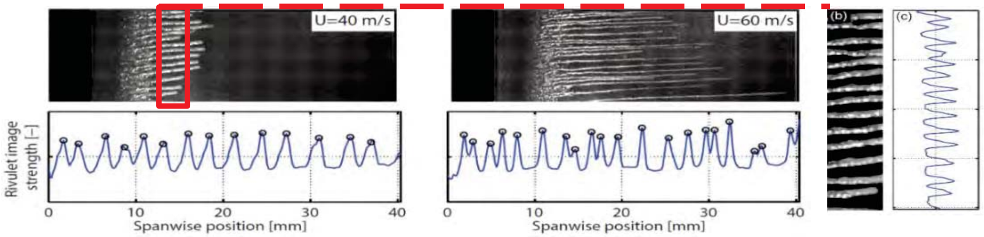


❑ DYNAMIC GLAZE ICE ACCRETION OVER AN AIRFOIL SURFACE

- *Test Conditions:*
    - *Oncoming airflow velocity :* $V_\infty \approx 35$ m/s
    - *Angle of attack of the airfoil:* $\alpha \approx 5$ deg.
    - *Airflow Temperature :* $T \approx -8$ °C.
    - *Liquid water content (LWC) :* LWC =3.0 g/m^3
    - *Image acquisition rate f = 150Hz, 10X replay*





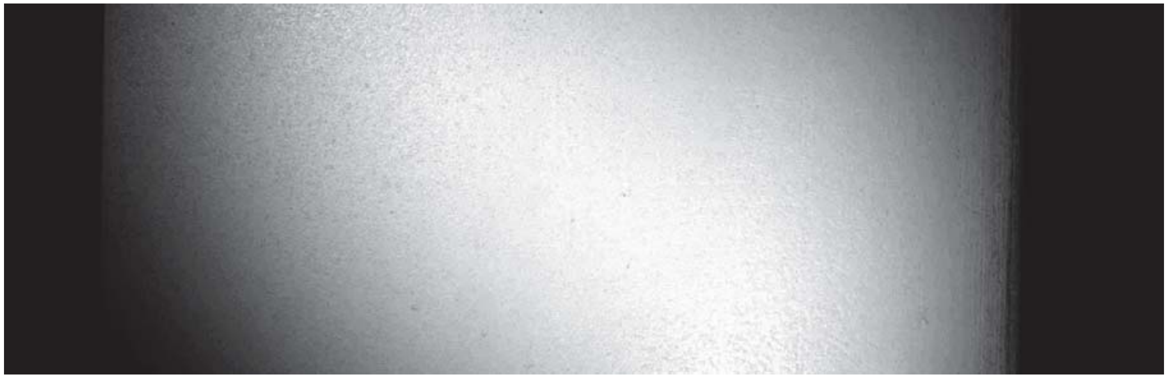


Freestream, U [m/s]	Film runback, u_{film} [mm/s]	Rivulet runback, u_{riv} [mm/s]
20	1.23	2.38
40	3.14	7.96
60	4.66	23.3

Freestream, U [m/s]	Number of rivulets, N [−]	Spacing, δ [mm]	Area fraction, AF [−]	Width, w [mm]
20	0	-	-	-
40	14	2.71 ± 0.60	0.50	1.35
60	17	2.09 ± 0.93	0.29	0.65

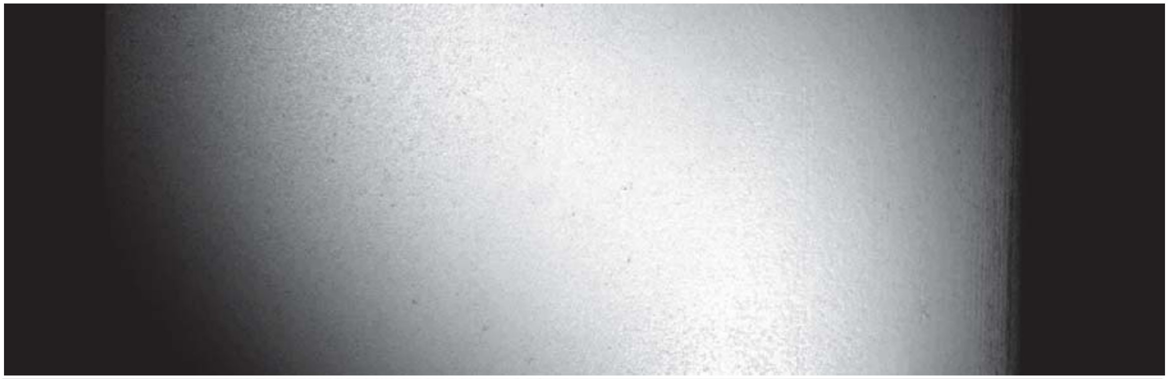
- *(Waldman R. and Hu H., 2016, Journal of Aircraft, Vol.53(2), pp369~377)*

❑ DYNAMIC GLAZE ICE ACCRETION OVER AN AIRFOIL SURFACE



V_∞= 20 m/s

- T_∞ = - 8.0 °C;
- α = 5 °;
- LWC = 1.1 g/m^3



V_∞= 40 m/s

V_∞= 60 m/s

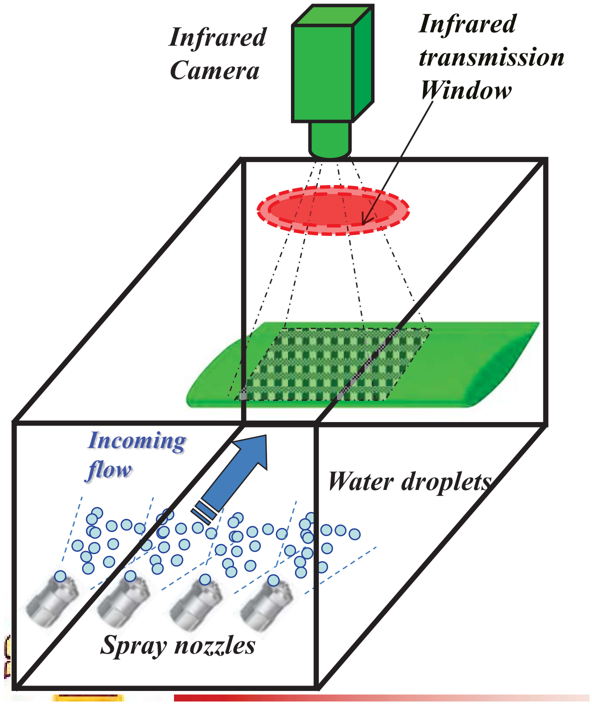
(Waldman R. and Hu H., 2015, *Journal of Aircraft*)

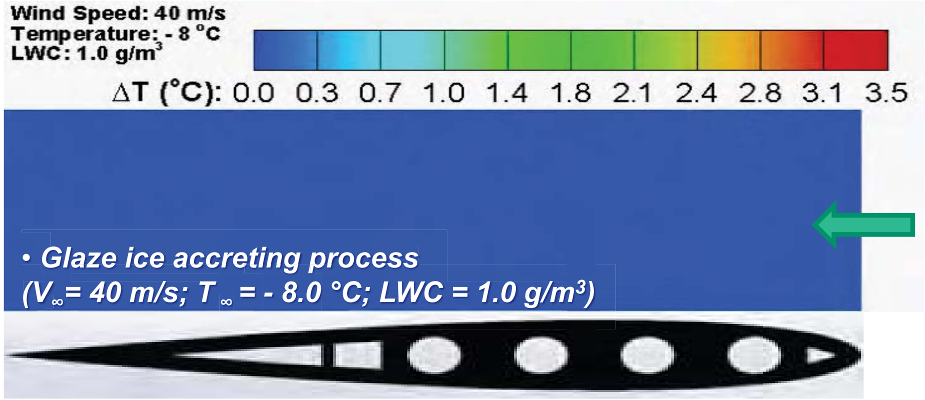
❑ UNSTEADY HEAT TRANSFER PROCESS OVER AN ICE ACCRETING AIRFOIL

Test Conditions:

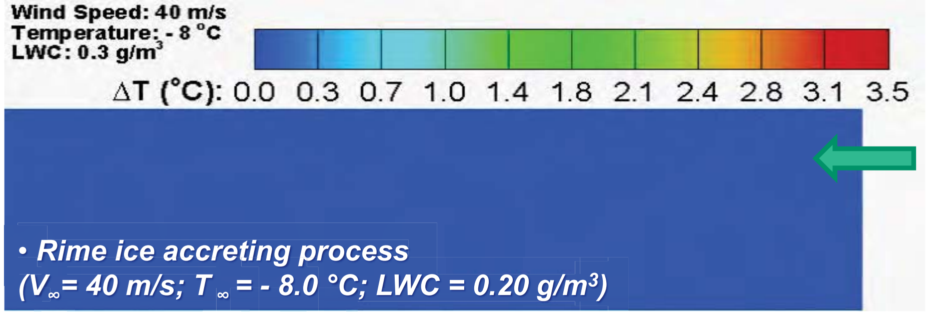
- *Airflow temperature: -4 & -8 °C*
- *Wind speed: 20, 40, 60 m/s*
- *LWC in airflow: 0.3, 1.0, 3.0 g/m³*
- *Angle of Attack: -5°*

Experimental setup for IR thermal imaging

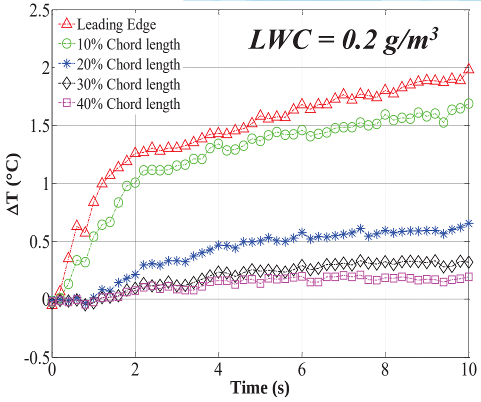


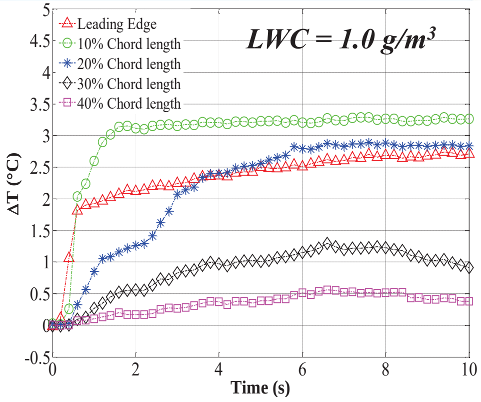


- *Glaze ice accreting process*
(V_∞= 40 m/s; T_∞ = - 8.0 °C; LWC = 1.0 g/m³)

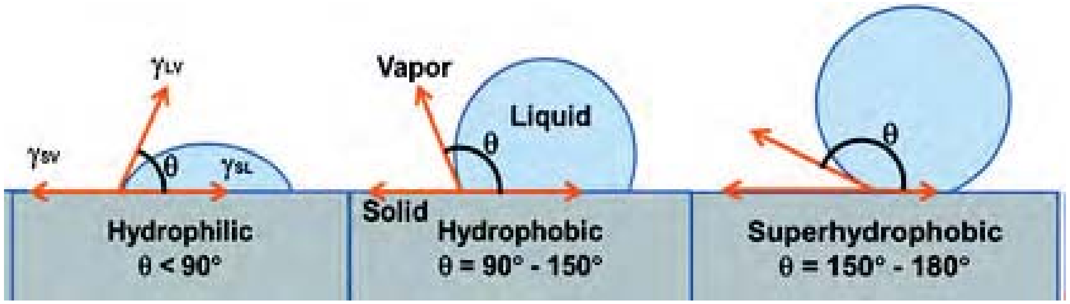


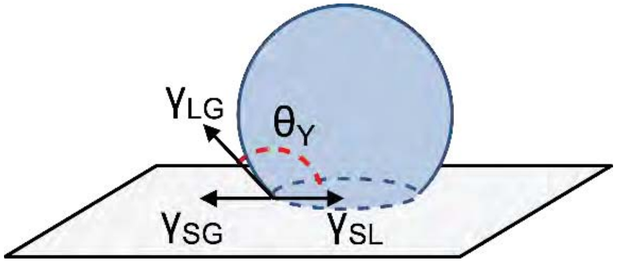
- *Rime ice accreting process*
(V_∞= 40 m/s; T_∞ = - 8.0 °C; LWC = 0.20 g/m³)





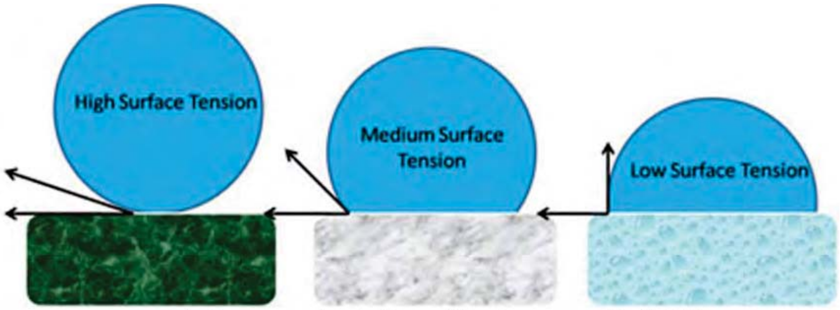
❑ HYDROPHILIC, HYDROPHOBIC, AND SUPER-HYDROPHOBIC SURFACE



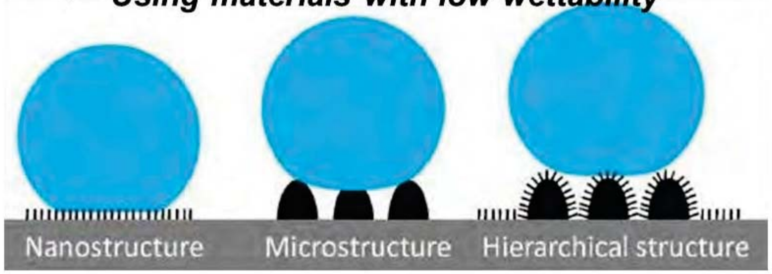


$$\gamma_{LG}\cos\theta_Y = \gamma_{SG} - \gamma_{LS}.$$

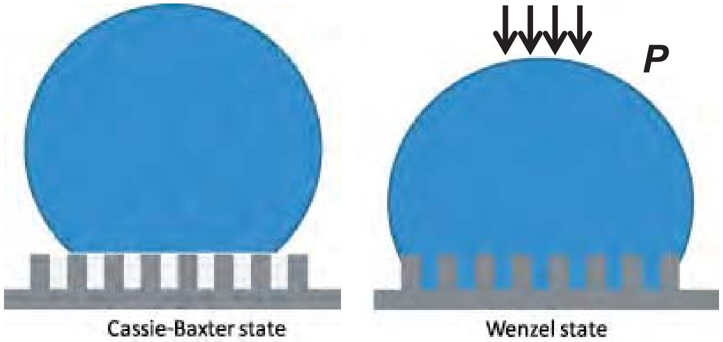
- ***A water droplet over a smooth surface***



- ***Using materials with low wettability***



- ***Making structured surface (creating micro-/nano- structures over the surface)***

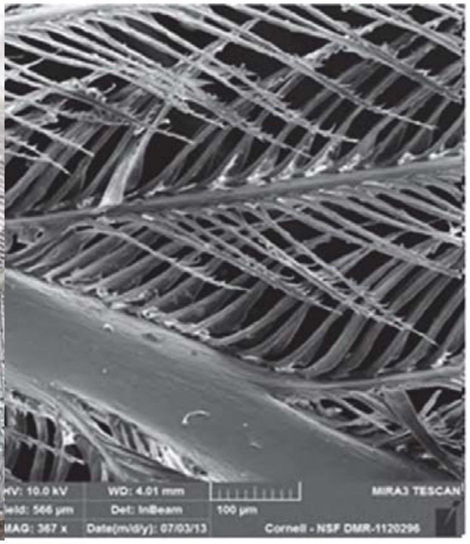


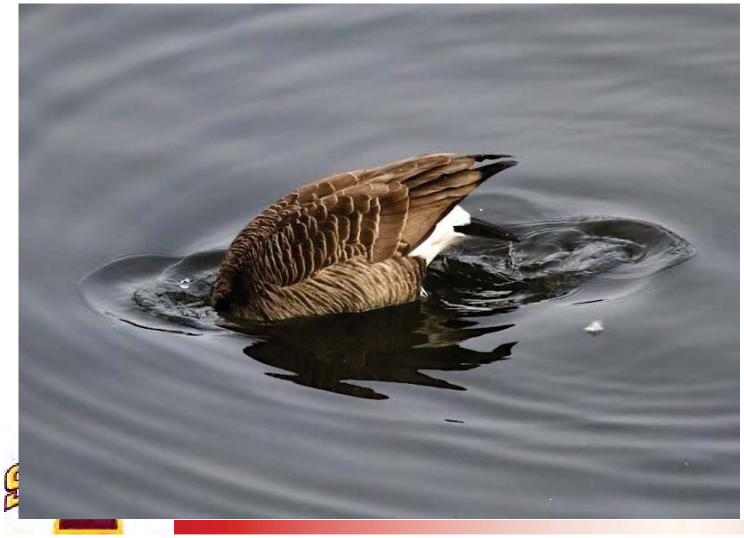
$$\cos\theta_{CB} = \varphi\cos\theta_Y + \varphi - 1 \qquad \cos\theta_W = n\cos\theta_Y$$

- ***A water droplet over a rough surface***

IOWA STATE UNIVERSITY

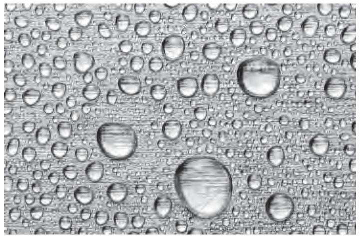
❑ Bird-Feather-Inspired Technology



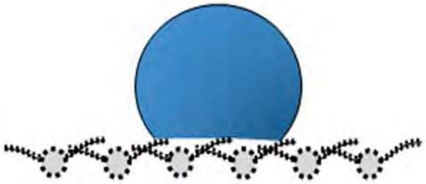


- *Air permeable multi-scale interlaced micro-/nano structures*

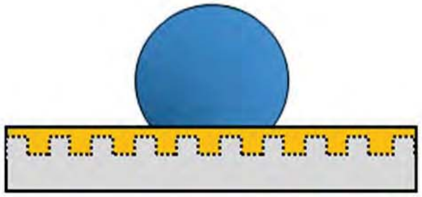
❑ Comparison of Tested Surfaces



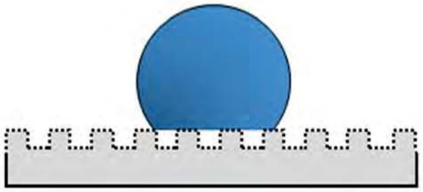
- Hydrophilic (baseline)



- Goose feather

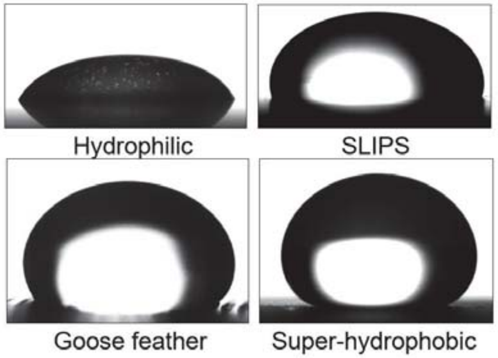


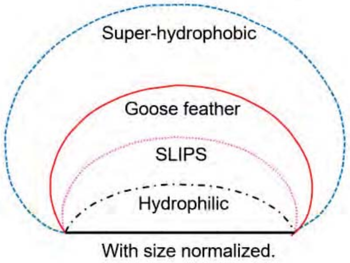
- SLIPS (Pitcher-plant-inspired)

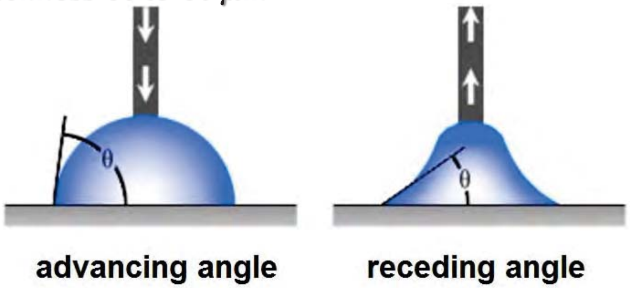


- Hydro-bead SHS (Lotus-leaf-inspired)

- ***DuPont Krytox 103 oil***
- ***Teflon nanofibrous membranes (pore size ~200 nm; thickness 60 to 80 μm***







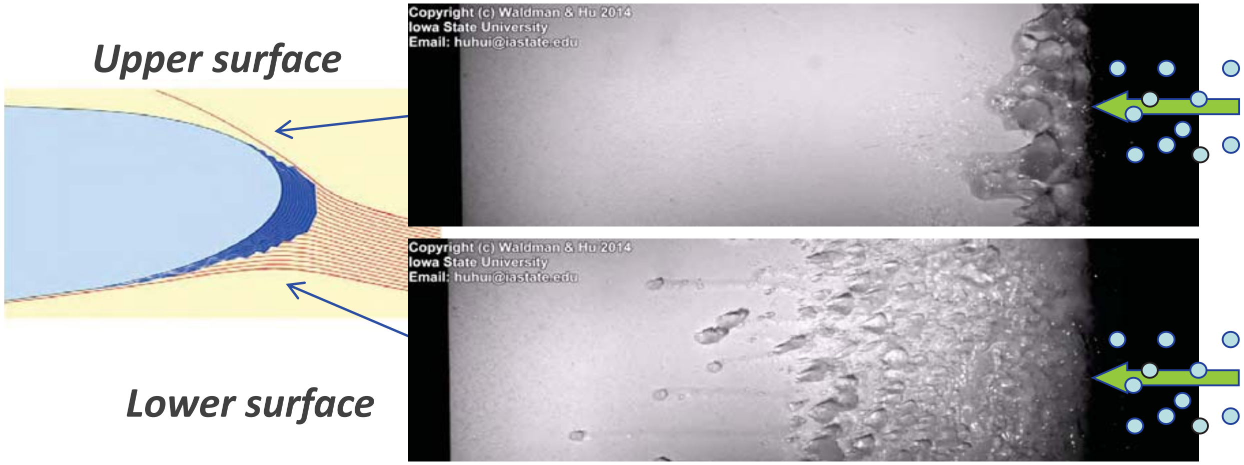
Surfaces	*Static CA*	*Advancing CA*	*Receding CA*	*Hysteresis*
Hydrophilic (baseline)	*30~80*	*70~105*	*15~60*	*<70*
Goose Feather	*75~145*	*142~158*	*70~80*	*<45*
SLIPS	*108~112*	*105~115*	*90~105*	*<15*
Hydro-bead SHS	*155~160*	*156~163*	*151~158*	*<5*

❑ Ice-phobic Coatings for Anti-Frosting vs. Impact Icing

- *Most previous studies were performed based on simple and static tests for anti-frosting applications.*
- *Very little can be found in literature to evaluate the ice-phobic coatings for "impact icing" mitigation pertinent to aircraft icing phenomena, in either dry rime or/and wet glaze icing conditions.*
- *Impact icing is defined as ice formed due to the dynamic collision of super-cooled water droplets onto a surface at a high impact velocity.*

Anti-Frosting application:

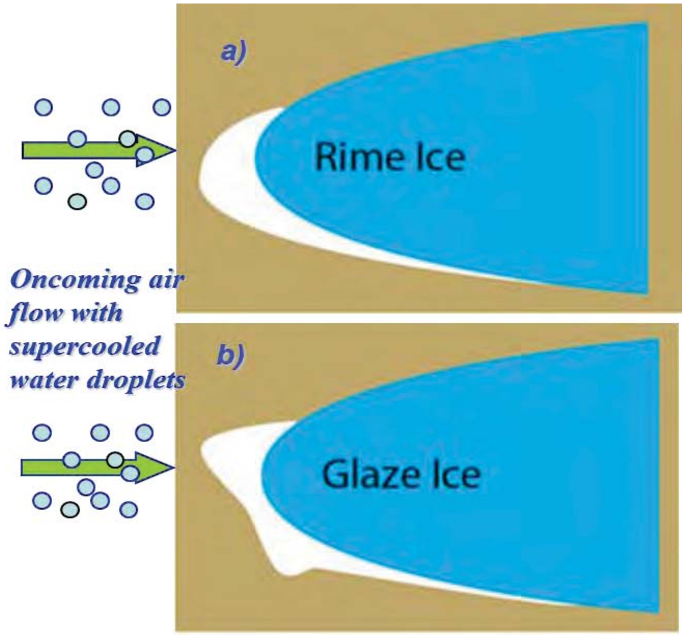
- *Icing process is almost static.*
- *Ice accretion speed is very slow.*



Impact Ice Mitigation:

- *Very short time scale*
- *High-speed impact of water droplets to cause Cassie to Wenzel transition.*
- *Significant effects of wind-driven surface runback process*

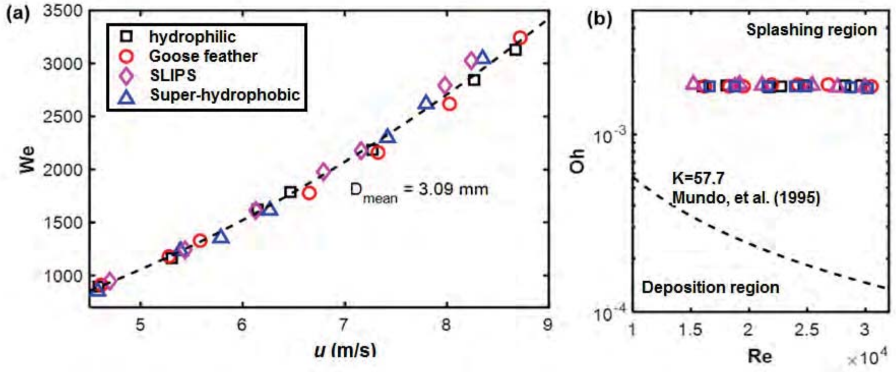
☐ DYNAMIC IMPACTS OF WATER DROPLETS ONTO AIRFOIL SURFACES

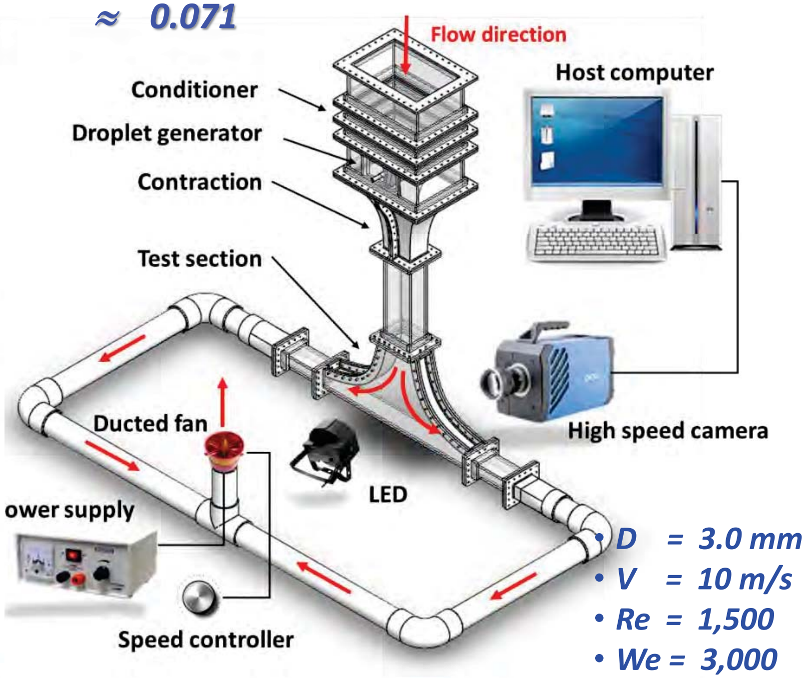


- Weber number $We = \rho D V_{impact}^{2} / \sigma$
- Reynolds number $Re = \rho D V_{impact} / \mu$
- Ohnesorge number $Oh = \mu / \sqrt{\rho \sigma D} = \sqrt{We} / Re$

ρ: *Water density*
μ: *Water viscosity*
σ: *Surface tension*
D: *Droplet diameter*
U: *Impact velocity*

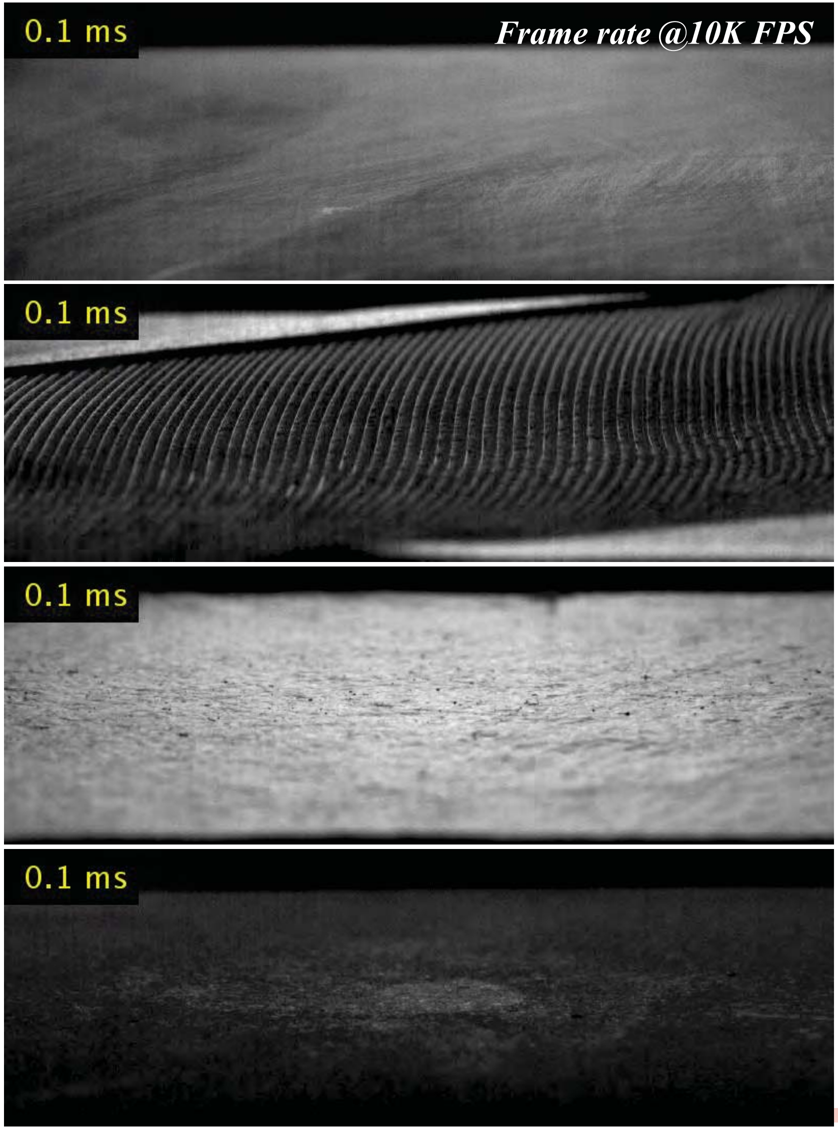
- ***Typical parameter pertinent to aircraft icing phenomena:***
  - ***D = 10 μm***
  - ***V_{impact} = 100 m/s***
  - ***We ≈ 1,250***
  - ***Re ≈ 500***
  - ***Oh ≈ 0.071***





- *D = 3.0 mm*
- *V = 10 m/s*
- *Re = 1,500*
- *We = 3,000*

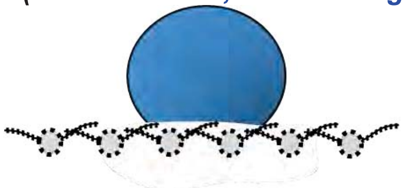
❑ DYNAMIC DROPLET IMPINGEMENT ONTO DIFFERENT SURFACES



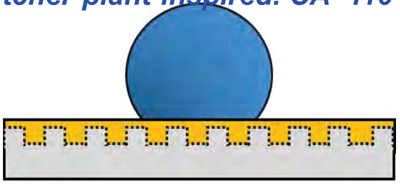
$We \approx 2,000$

Hydrophilic
(Comparison baseline, CA=65 deg..)

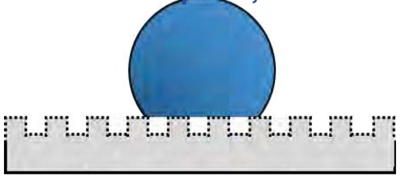
Feather
(Goose feather, CA=130 deg.)



SLIPS
(Pitcher-plant-inspired. CA=110 deg.)



Super-hydrophobic
(Lotus-leaf-inspired, CA=160 deg.)



- ***LQ Ma, HX Li, and H. Hu.*** *An Experimental Study on the Dynamics of Water Droplet Impingement onto Bio-inspired Surfaces with Different Wettability.* ***AIAA-2017-0442****, SciTech2017.*

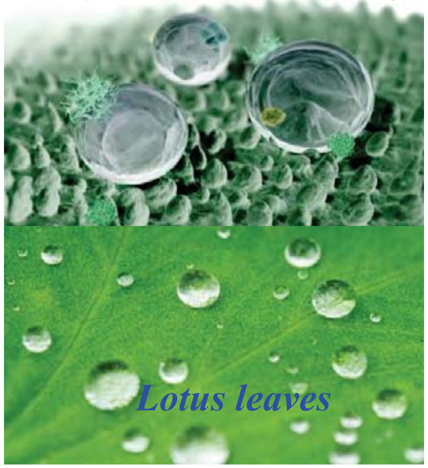
❑ Hydrophilic, Hydrophobic and Superhydrophobic

SLIPS coated surface

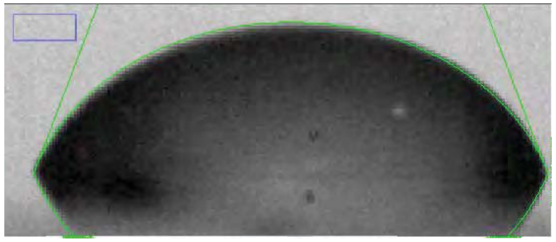
(Wong et al., 2011)

Hydro bead coated surface

(provided by seashell technology)

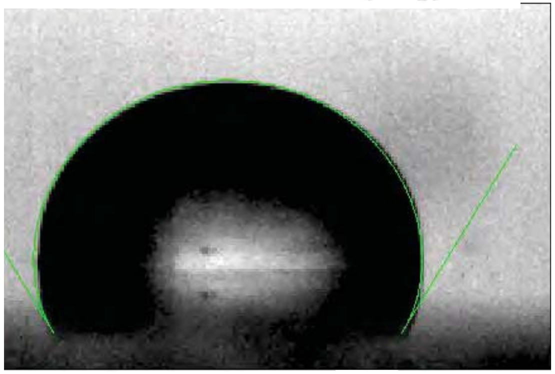


Measured θ ≈ 65 [deg]



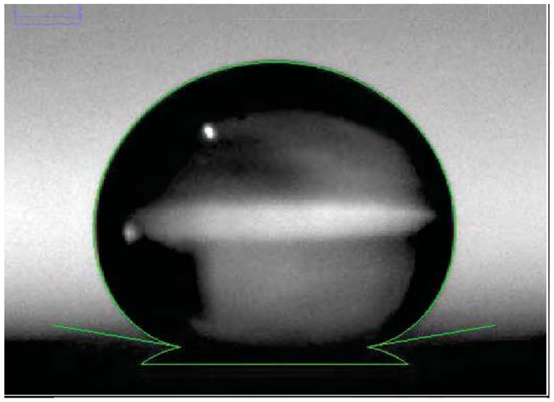
- ***Hydrophilic; θ < 90 º***

Measured θ ≈ 105 [deg]



- ***Hydrophobic; 90 º <θ < 150 º***

Measured θ ≈ 157 [deg]

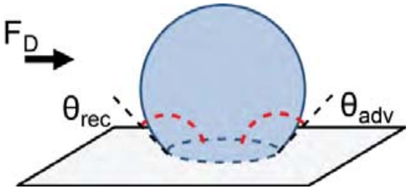


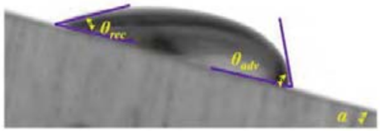
- ***Superhydrophobic; θ > 150 º***

❑ Measuring Advancing and Receding Angles of Water Droplets

- ***According to Quéré et al. (1998):***

$$F_{cap} = \pi R \gamma_{LG} \sin\left(\frac{\theta_{adv}+\theta_{rec}}{2}\right)\left(\cos\left(\theta_{adv}\right)-\cos\left(\theta_{rec}\right)\right)$$

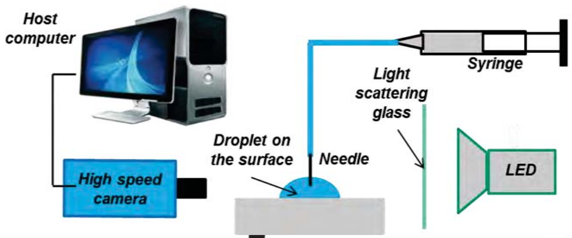


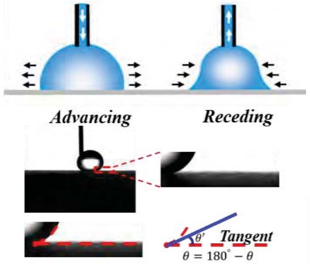


	Advancing contact angle (°)	Receding contact angle (°)	Hysteresis (°)	Ratio of Capillary forces
Hydrophilic (Enamel)	70 ~ 105	15 ~ 60	> 50	1.0
SLIPS	105 ~ 115	90 ~ 105	< 15	~0.25
Superhydrophobic (Hydrobead)	144 ~ 148	141 ~ 145	< 5	~0.04

$$\frac{F_{cap,\,\text{enamel}}}{F_{cap,\,\text{SLIPS}}} \approx \frac{\left[\sin\left(\frac{\theta_{adv}-\theta_{rec}}{2}\right)\sin\left(\frac{\theta_{adv}+\theta_{rec}}{2}\right)\right]_{\text{enamel}}}{\left[\sin\left(\frac{\theta_{adv}-\theta_{rec}}{2}\right)\sin\left(\frac{\theta_{adv}+\theta_{rec}}{2}\right)\right]_{\text{SLIPS}}} \approx 4$$

$$\frac{F_{cap,\,\text{enamel}}}{F_{cap,\,\text{SHP}}} \approx \frac{\left[\sin\left(\frac{\theta_{adv}-\theta_{rec}}{2}\right)\sin\left(\frac{\theta_{adv}+\theta_{rec}}{2}\right)\right]_{\text{enamel}}}{\left[\sin\left(\frac{\theta_{adv}-\theta_{rec}}{2}\right)\sin\left(\frac{\theta_{adv}+\theta_{rec}}{2}\right)\right]_{\text{SHP}}} \approx 25$$



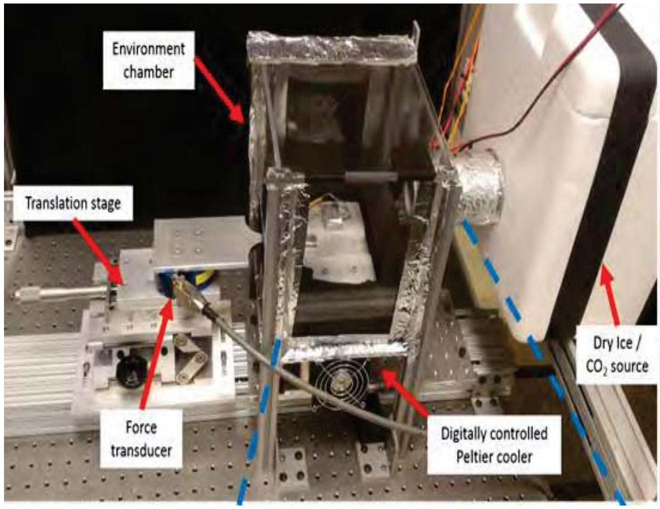


Hydrophilic

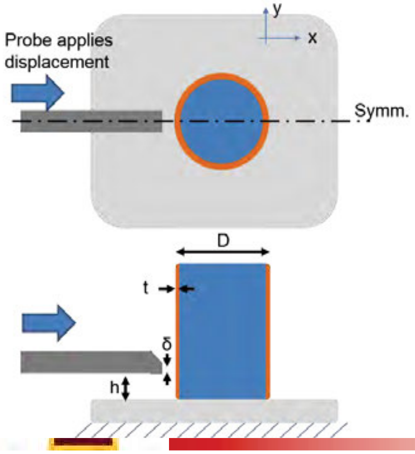
SLIPS

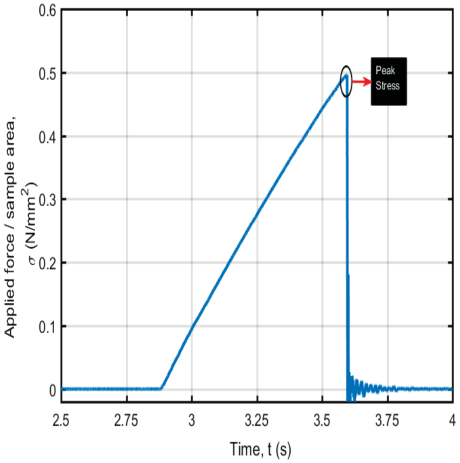
Superhydrophobic coating

☐ Measurements of Ice Adhesion Force over Different Surfaces

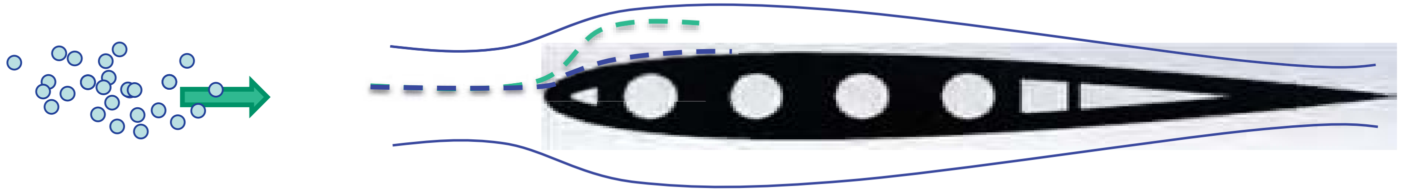


Test surface	Compared Surfaces	Ice adhesion strength @T_{wall} = -10 °C [K Pa]	Std. deviation @ T_{wall} = -10 °C [KPa]
1	Al, 220 Grit	450	70
2	Al, 400 Grit	390	60
3	Al, 1000 Grit	340	40
4	Al, 2000 Grit	300	60
5	Al, mirror finish	130	60
6	Enamel	1,400	130
7	Teflon	420	60
8	Hydrobead SHP	370	90
9	SLIPS	60	10
10	PFA plastic	570	60
11	Stainless steel	550	130
12	NeverWet	420	40



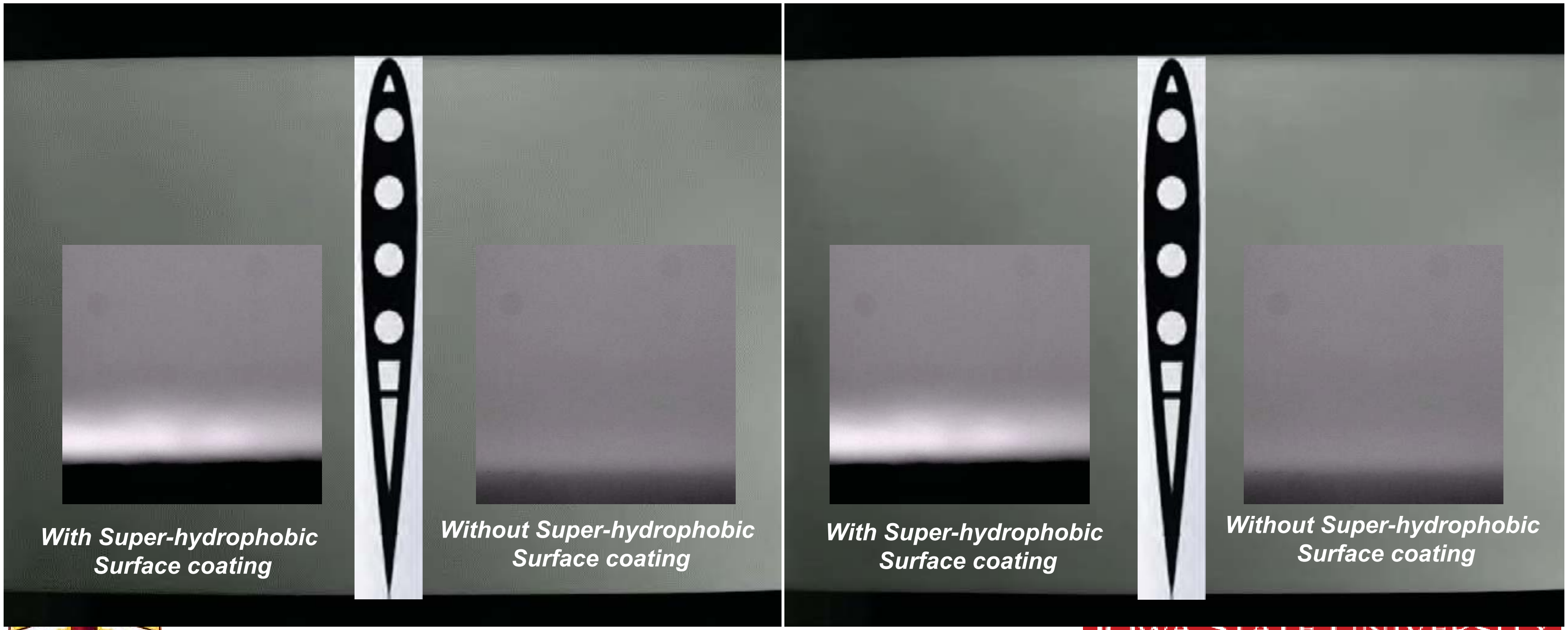


❑ Effects of Bio-Inspired Coatings on Impact Ice Accretion



$T_\infty = -8\,°C$; $V_\infty = 20$ m/s
MVD=40 μm; LWC = 2.5 g/m^3

$T_\infty = -8\,°C$; $V_\infty = 50$ m/s
MVD=40 μm; LWC = 2.5 g/m^3



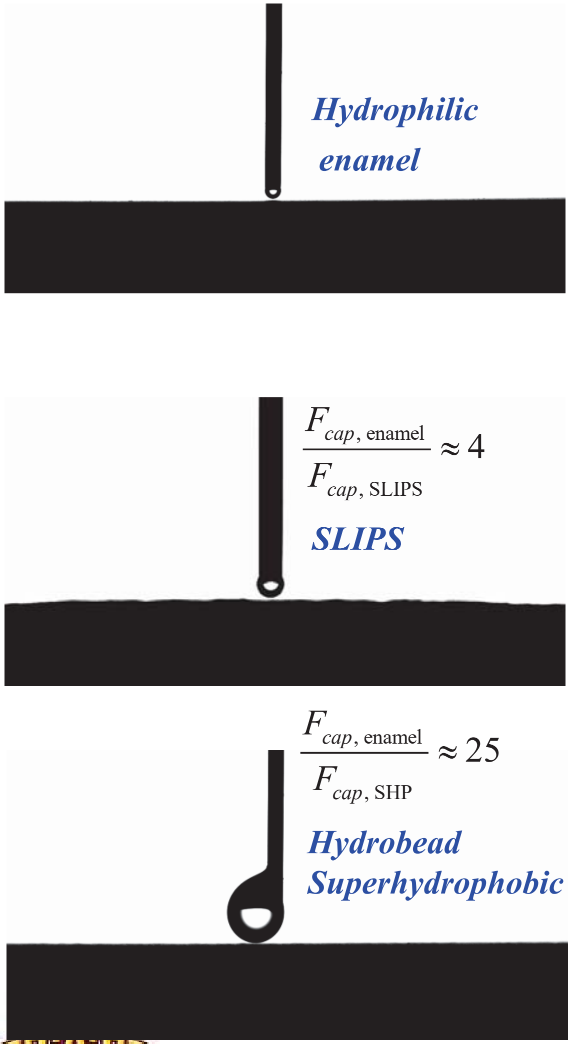
With Super-hydrophobic Surface coating

Without Super-hydrophobic Surface coating

With Super-hydrophobic Surface coating

Without Super-hydrophobic Surface coating

❑ EFFECTS OF BIO-INSPIRED COATINGS ON IMPACT ICE ACCRETION



Hydrophilic enamel

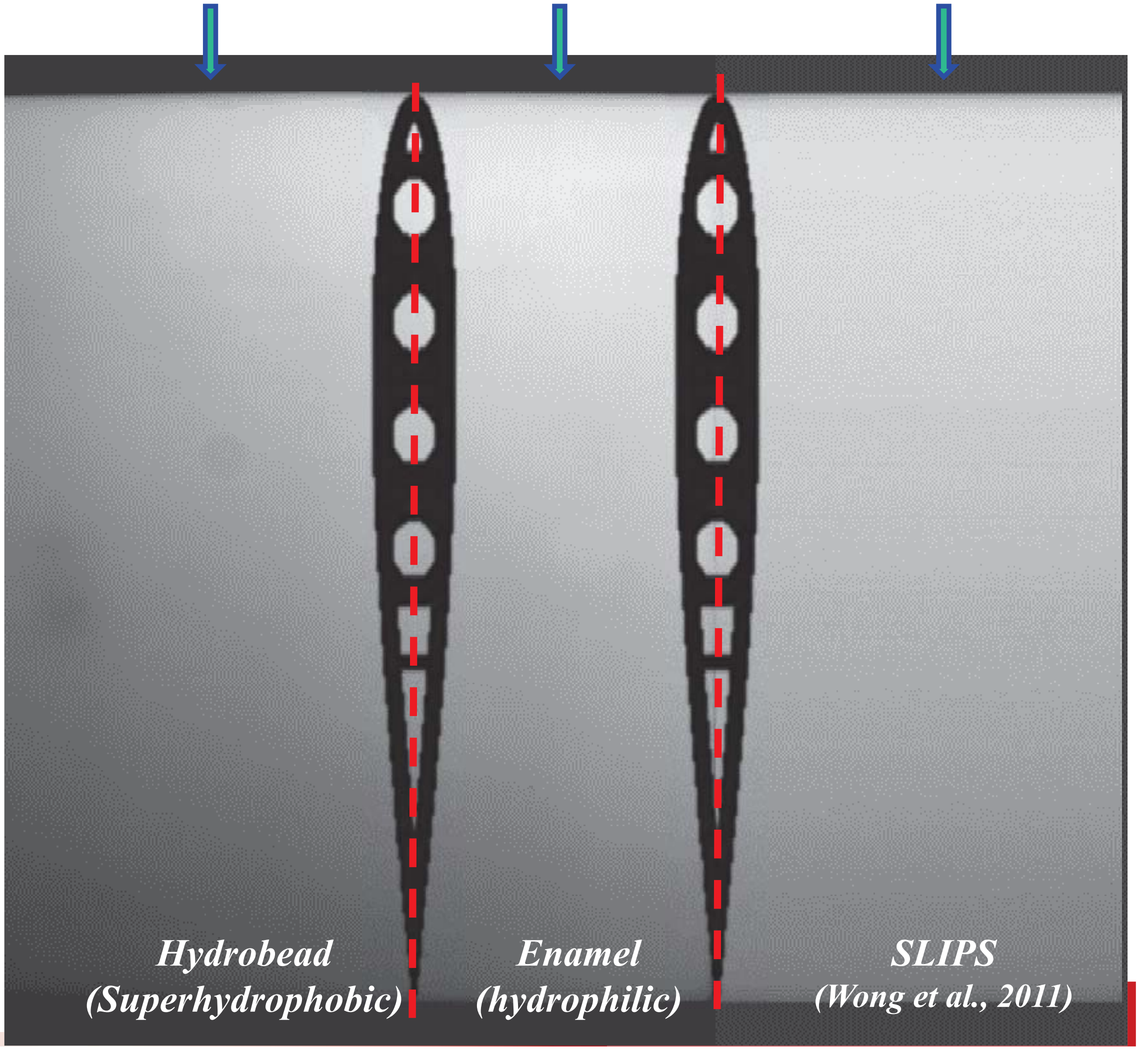
$$\frac{F_{cap,\,\mathrm{enamel}}}{F_{cap,\,\mathrm{SLIPS}}} \approx 4$$

SLIPS

$$\frac{F_{cap,\,\mathrm{enamel}}}{F_{cap,\,\mathrm{SHP}}} \approx 25$$

Hydrobead Superhydrophobic

$T_\infty = -4°C$; $V_\infty = 30$ m/s; MVD=40μm; LWC = 4.0 g/m^3



Hydrobead (Superhydrophobic)

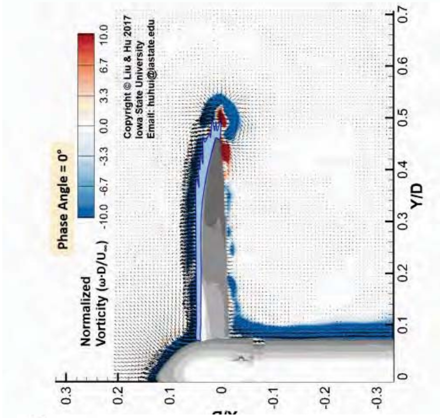
Enamel (hydrophilic)

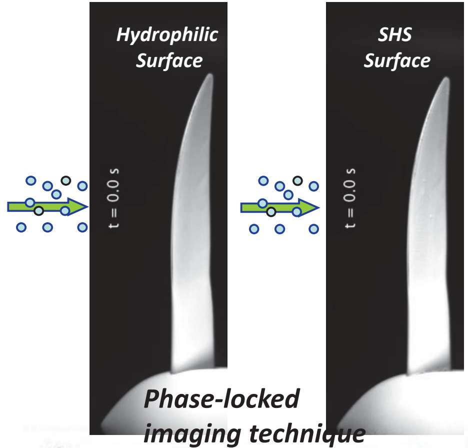
SLIPS (Wong et al., 2011)

❑ DURABILITY TESTING OF BIO-INSPIRED ICEPHOBIC COATINGS

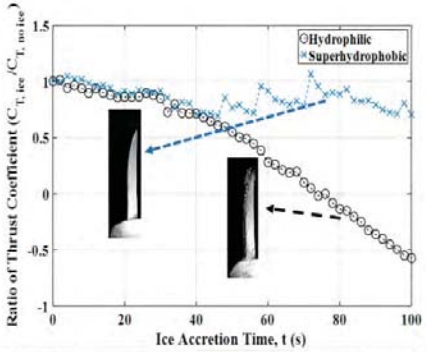
- Puma-AE drone (AeroVironment)

- MQ-9 Reaper (GA-ASI)

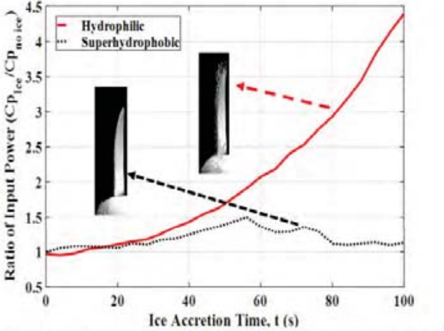




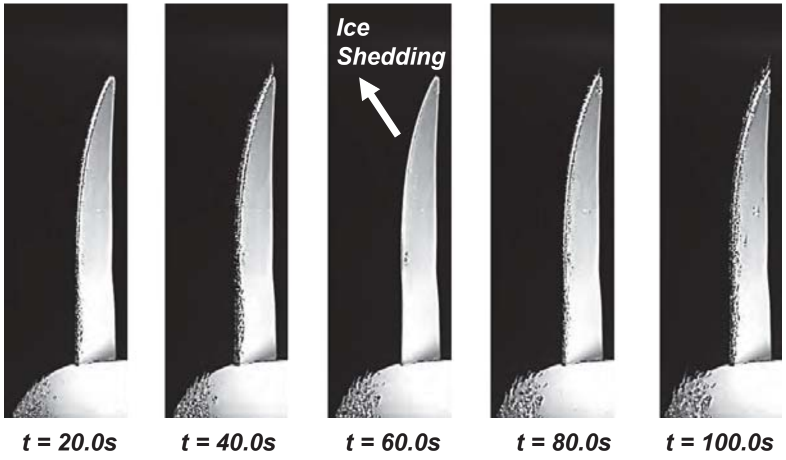
Phase-locked imaging technique



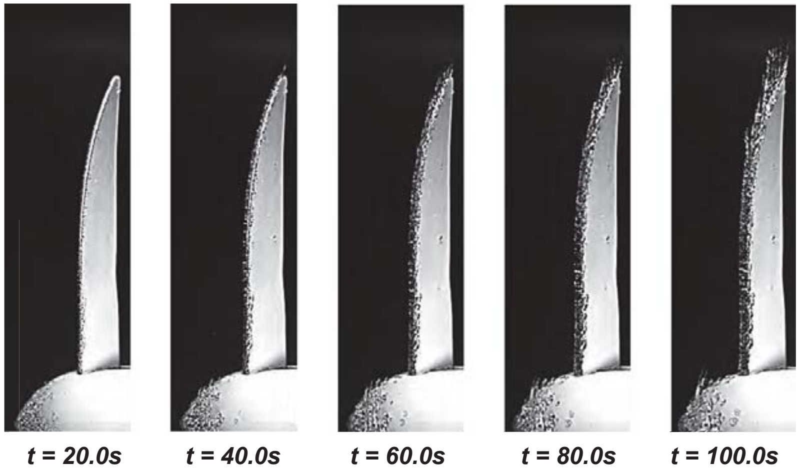
- Thrust loss due to ice accretion



- Extra power consumption due to ice accretion



- ***Right after the coating was applied.***

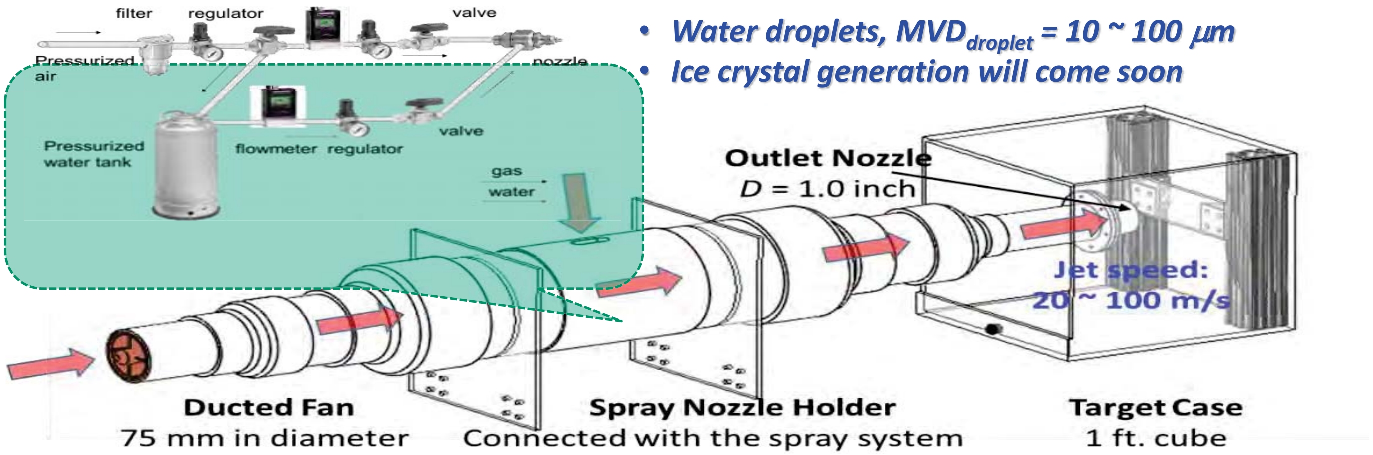


- ***After 15 icing-and-deicing cycles.***

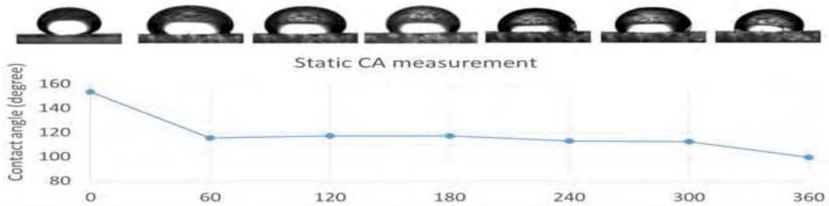
(Each icing-and-deicing would include 120s icing testing in ISU-IRT, and then warm-up to room temperature for de-icing)

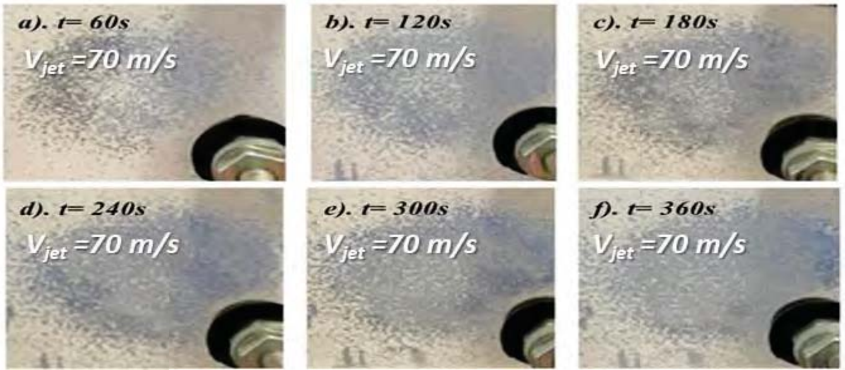
❑ DURABILITY TESTING OF BIO-INSPIRED ICEPHOBIC COATINGS

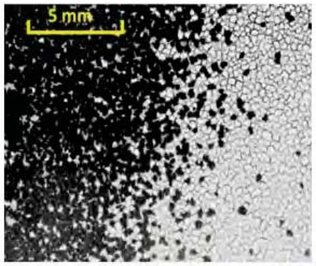
- *Water droplets, MVD$_{droplet}$ = 10 ~ 100 μm*
- *Ice crystal generation will come soon*



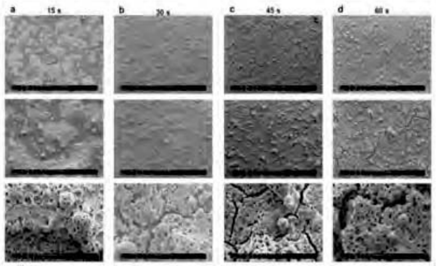
Test rig for durability testing of surface coatings





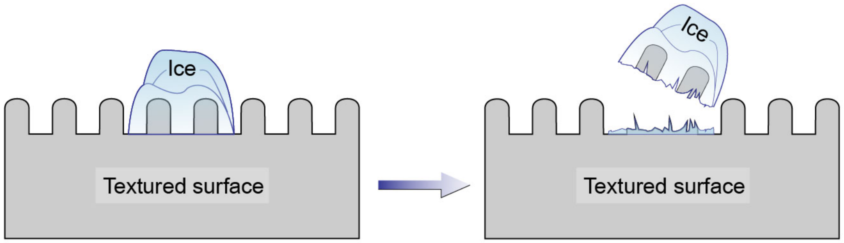


- *Morphology of Hydrobead™ SHS coating on a test substrate. White region with coating covered and the dark region with coating wore out.*

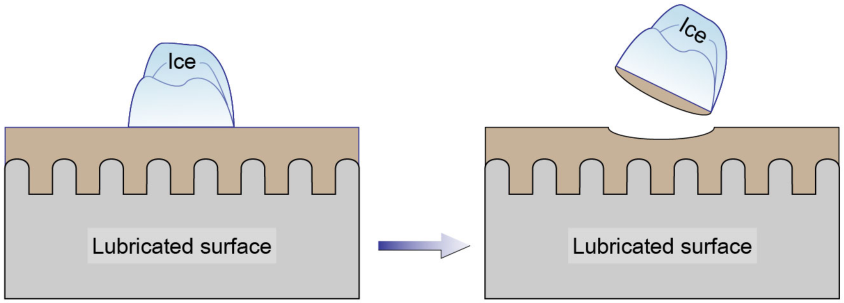


- *SEM images of morphology of Hydrobead™ coating (a) 15s, (b) 30s, (c) 45s, and (d) 60s*

❑ ICEPHOBIC SOFT SURFACES FOR AIRCRAFT ICING MITIGATION?

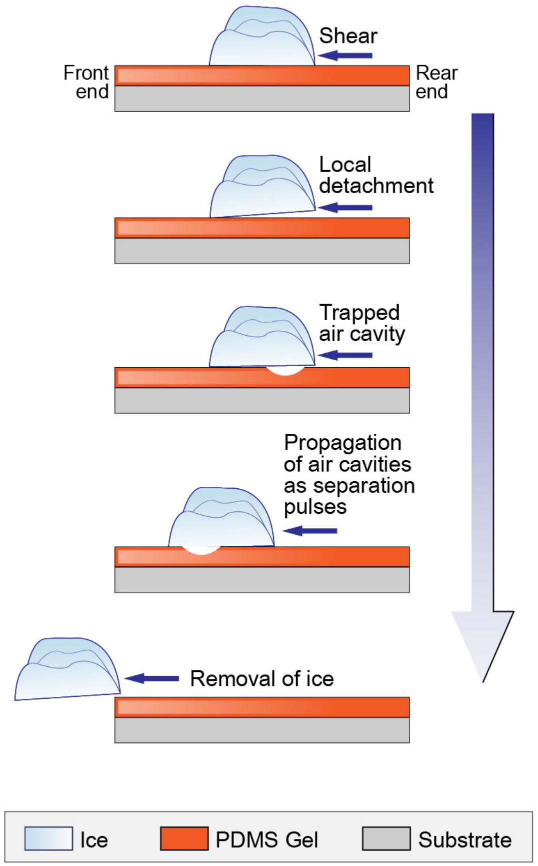


De-icing process causes structural failure for textured icephobic surface



De-icing process sacrifices liquid for lubricated icephobic surface

- Consider mechanical durability, are there any better icephobic materials?
- Icephobic Soft Surfaces?

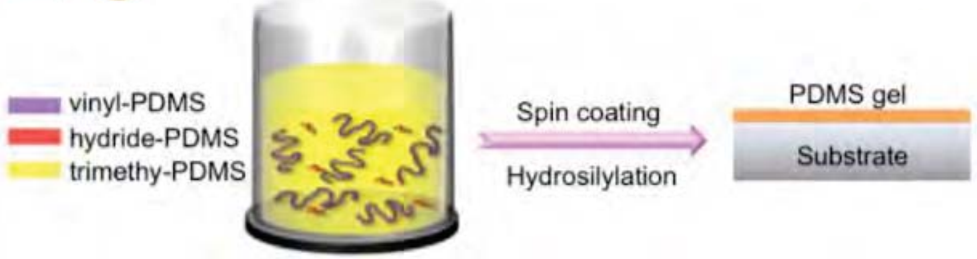


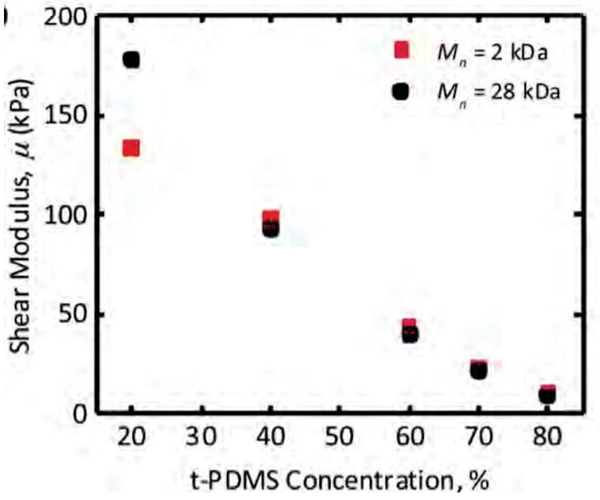
- Schematic illustrating the separation of ice from the PDMS gels via separation pulses.

- *Beemer DL., Wang W, Kota AK. "Durable gels with ultra-low adhesion to ice." Journal of Materials Chemistry A 4.47 (2016): 18253-18258.*

❑ ICEPHOBIC SOFT MATERIAL (PDMS) WITH ADJUSTABLE STIFFNESS

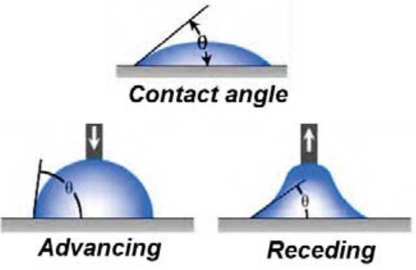
- Research collaboration with Dr. Arun Kota @ Colorado State University





- *The shear modulus of the PDMS gels can be tuned by adding non-active trimethyl-terminated PDMS (t-PDMS) with the concentration from 10% to 80%.*
- *Constant thickness of 500 m of PDMS gels can be ensured by controlling the spin coating speed.*

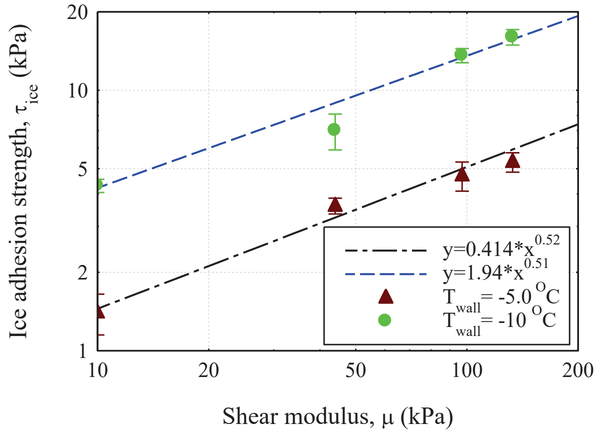
Compared surface	Static contact angle (°) θ_{static}	Advancing contact angle(°) $\theta_{advancing}$	Receding contact angle (°) $\theta_{receding}$	Hysteresis (°) $\Delta\theta=\theta_{advancing}-\theta_{receding}$
Airfoil Surface	65	105	50	55
20% t-PDMS	110	114	78	36
40% t-PDMS	109	116	77	38
60% t-PDMS	108	115	78	37
80% t-PDMS	110	118	80	38



❑ ICEPHOBIC SOFT MATERIAL (PDMS) WITH ADJUSTABLE STIFFNESS

- ***Ice adhesion strength over soft PDMS surface :***

Concentration (%)	T_{wall} = -5 °C		T_{wall} = -10 °C	
	Mean Adhesion Strength (KPa)	Std. deviation (KPa)	Mean Adhesion Strength (KPa)	Std. deviation (KPa)
20	5.3	0.9	16	2.2
40	4.7	1.2	13.6	1.7
60	3.6	0.5	7.0	2.2
80	1.4	0.5	4.3	0.5

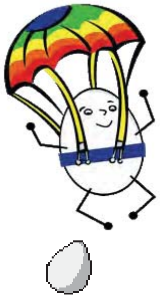


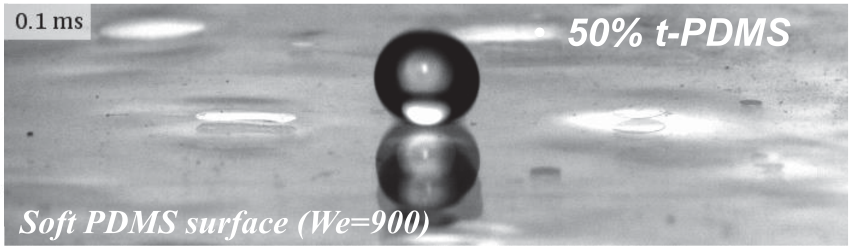
- ***Ice adhesion strength over soft PDMS surface is extremely lower than conventional surfaces.***
- ***Measurement results agree with classical adhesion mechanism ,where τ_{ice} is proportional to $\mu^{0.5}$ (Chaudhury and Kim 2007)***

Compared Surfaces	Ice adhesion strength at T_{wall} = -10 °C [KPa]	Std. deviation @ T_{wall} = -10 °C [KPa]
Al, 220 Grit	450	70
Al, mirror finish	130	60
Enamel	1400	130
Teflon	420	60
Hydrobead SHP	370	90
SLIPS	60	10
PFA plastic	570	60
Stainless steel	550	130
NeverWet	420	40

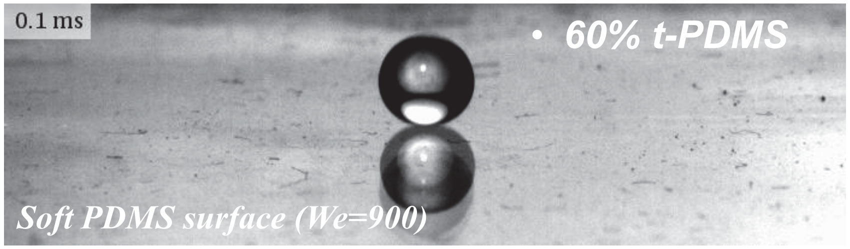
☐ DYNAMIC IMPACTS OF DROPLETS ONTO SOFT PDMS SURFACES

- *Egg Drop Experiment*

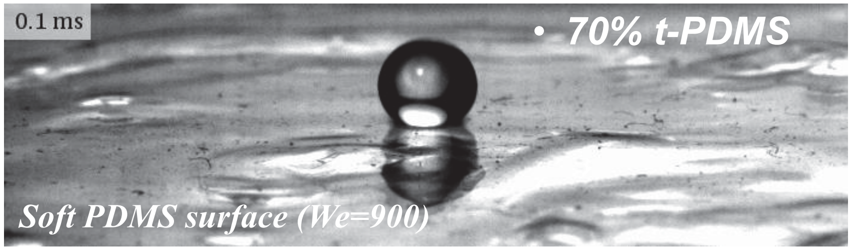




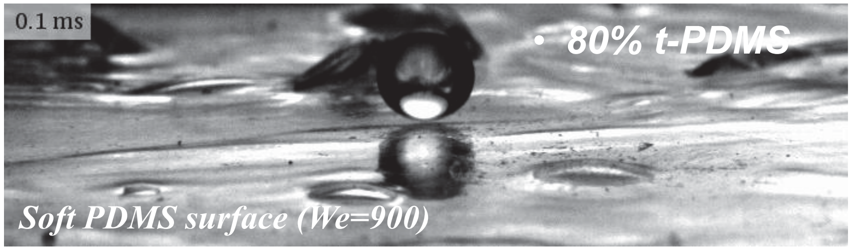
- **50% t-PDMS**



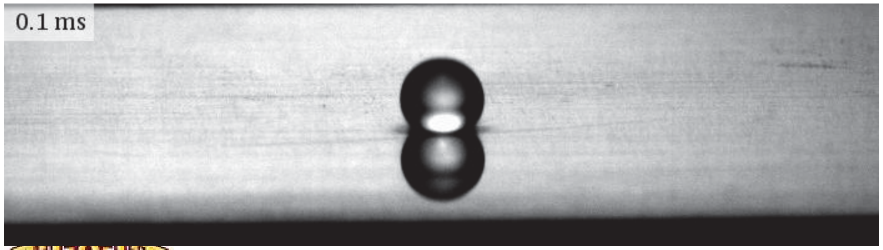
- **60% t-PDMS**



- **70% t-PDMS**

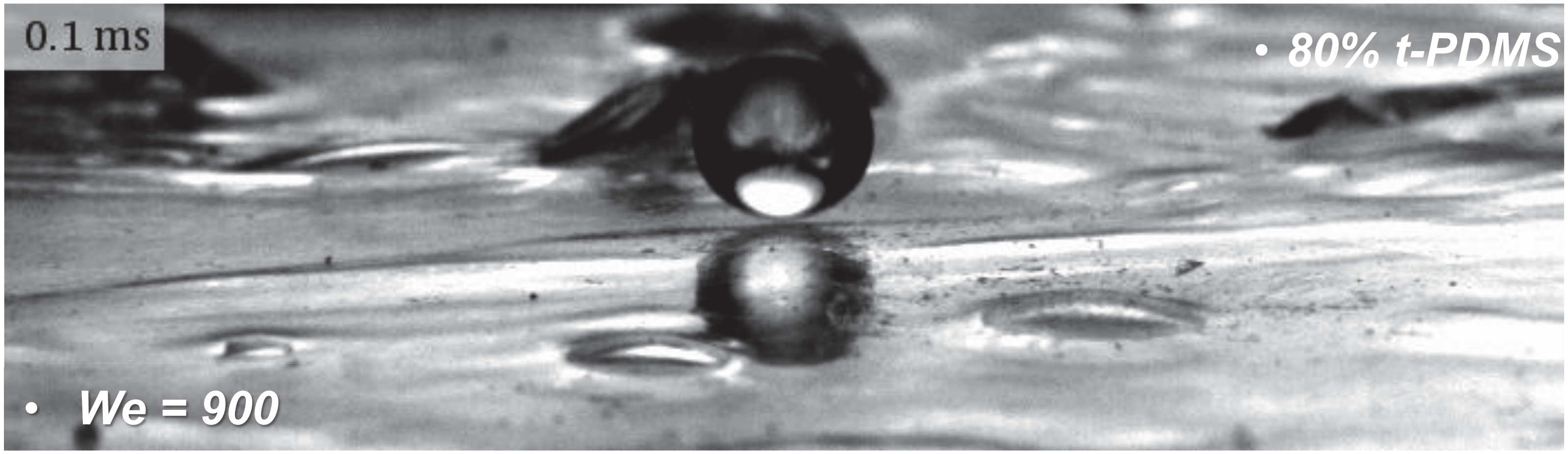


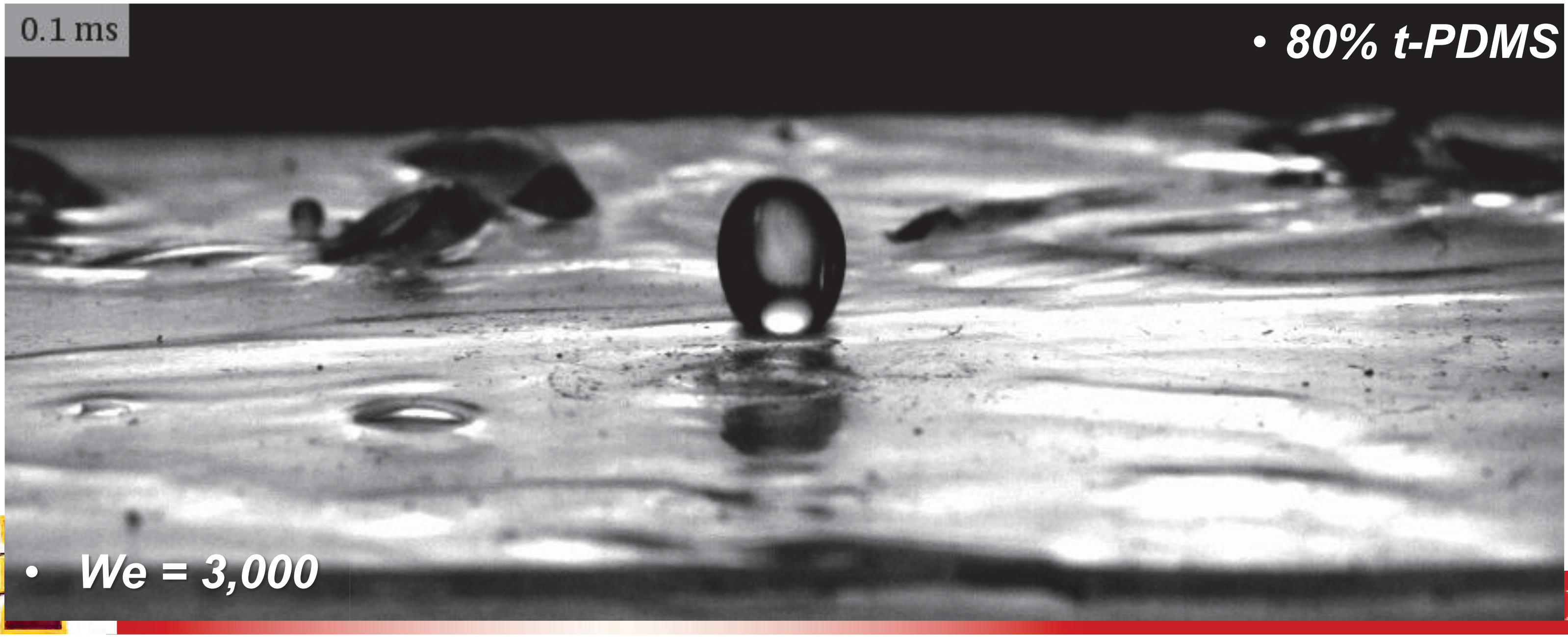
- **80% t-PDMS**



- *Rigid Aluminum Surface (We=900)*

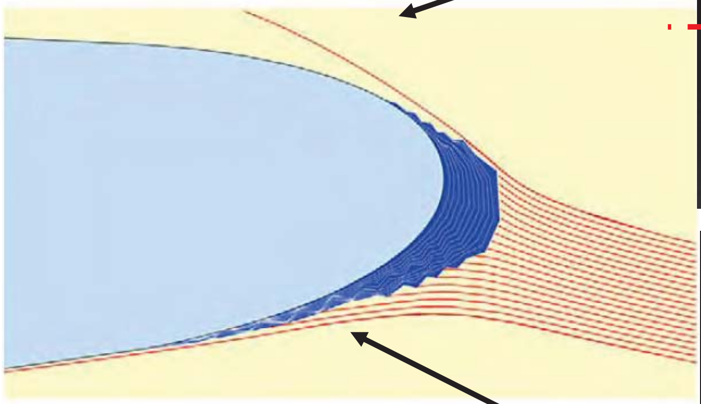
❑ DYNAMIC IMPACTS OF DROPLETS ONTO SOFT PDMS SURFACES

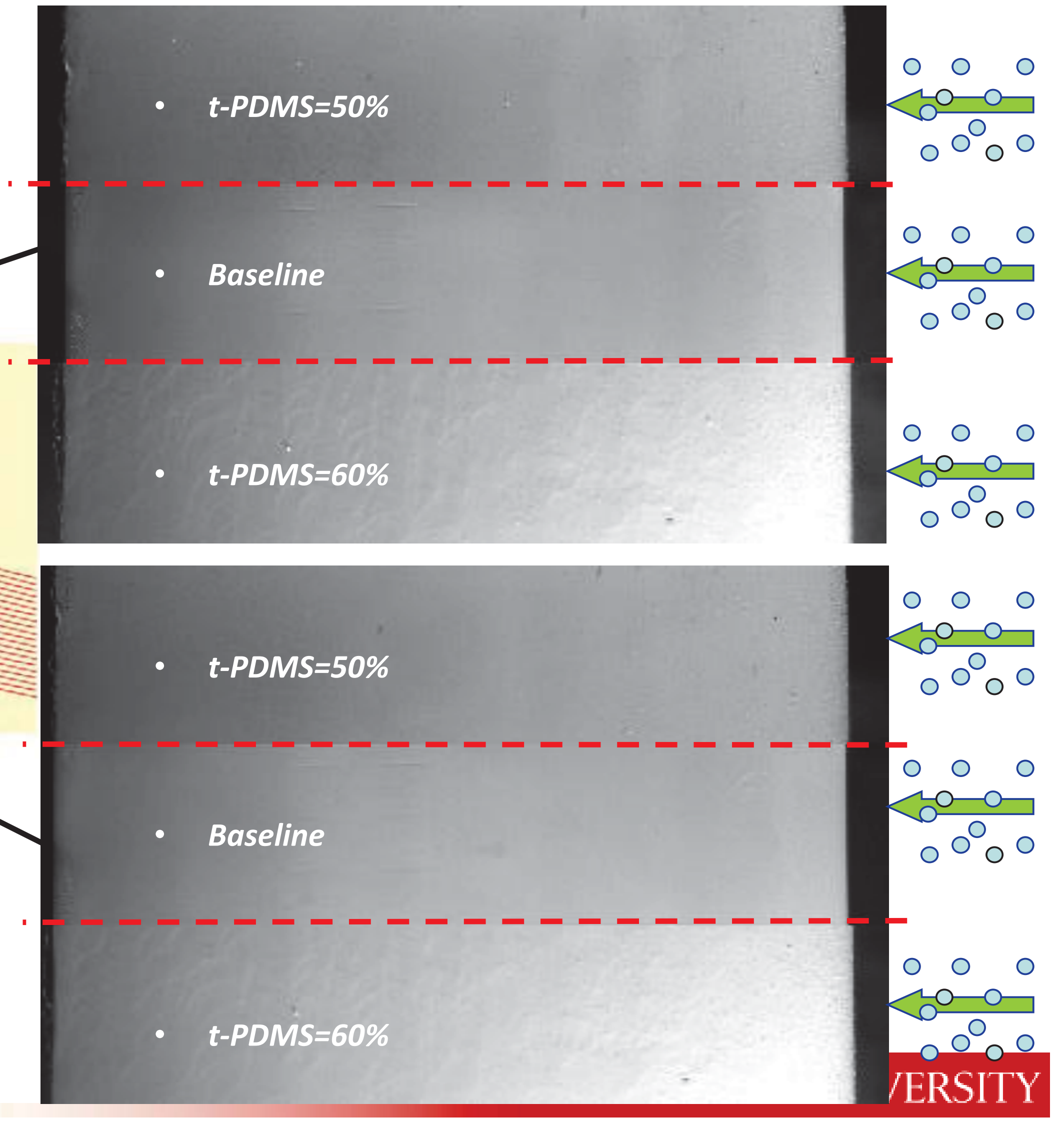




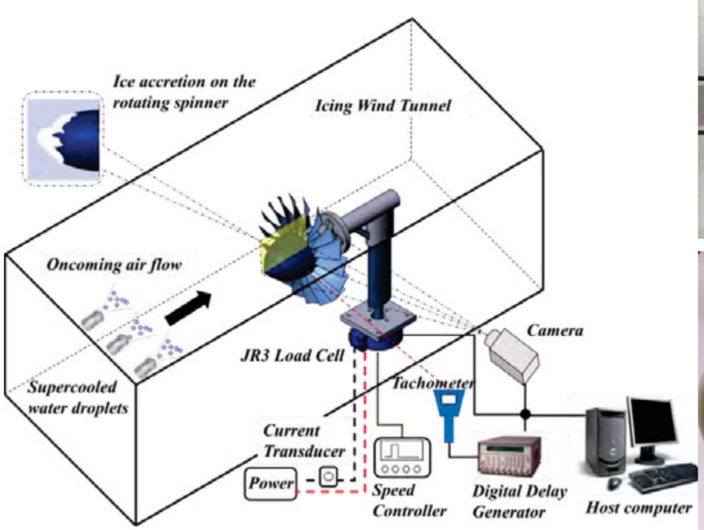
❑ IMPACT ICE ACCRETING PROCESS OVER ICE-PHOBIC SOFT SURFACES

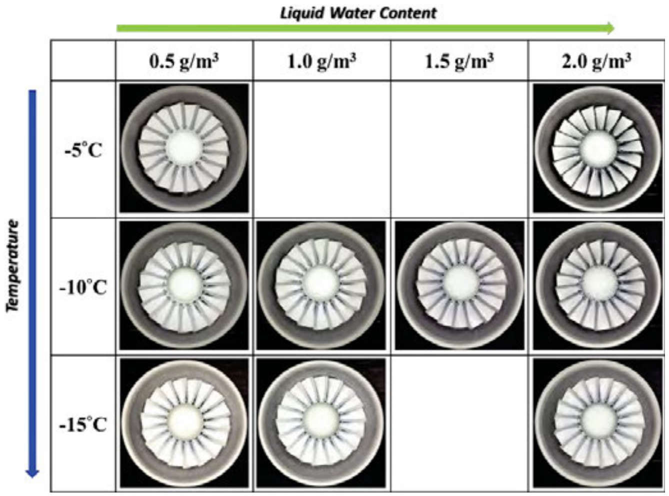
- ***Test Conditions:***
  - $V_\infty = 40\ m/s$
  - $T_\infty = -5.0\ ^\circ C$
  - $LWC = 1.0\ g/m^3$
  - $AOA = 5.0°$

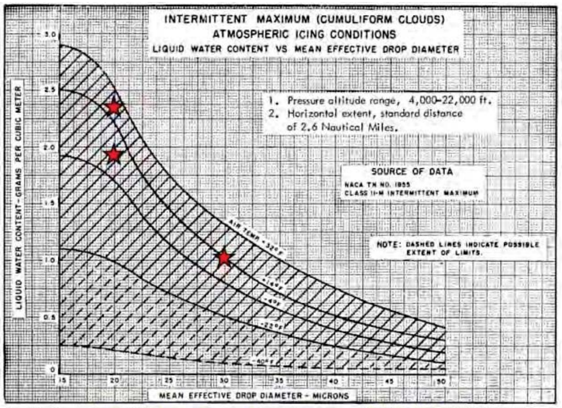




❑ EXPERIMENTAL SETUP FOR AERO-ENGINE ICING STUDY





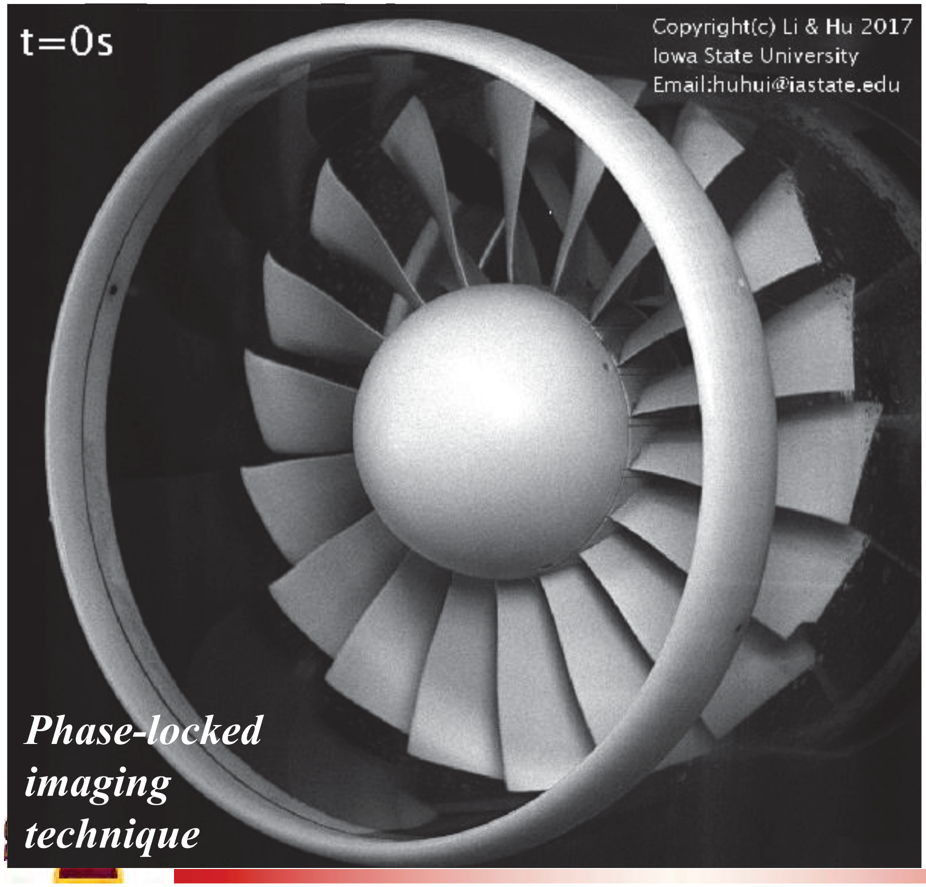


Intermittent maximum atmospheric icing conditions from 14 CFR Part 25 Appendix C[1]

Parameters	CFM 56-2/3 Turbofan	Aero-engine Model
Diameter of (m)	1.52	0.2
Max Rotation Speed (rpm)	5175	4000
Cruising Speed (m/s)	222 (0.74 Ma)	15
Cruising Rotation Speed (rpm)	4900	2500
Temperature Range (°C)	-40 ~ 20	-15 ~ -5
Liquid Water Content (g/m^3)	0.1 ~ 2.0	0.6 ~ 2.4
Advanced Ratio, J	1.80	1.80

Dynamic Ice Accreting Process over Fan Blades

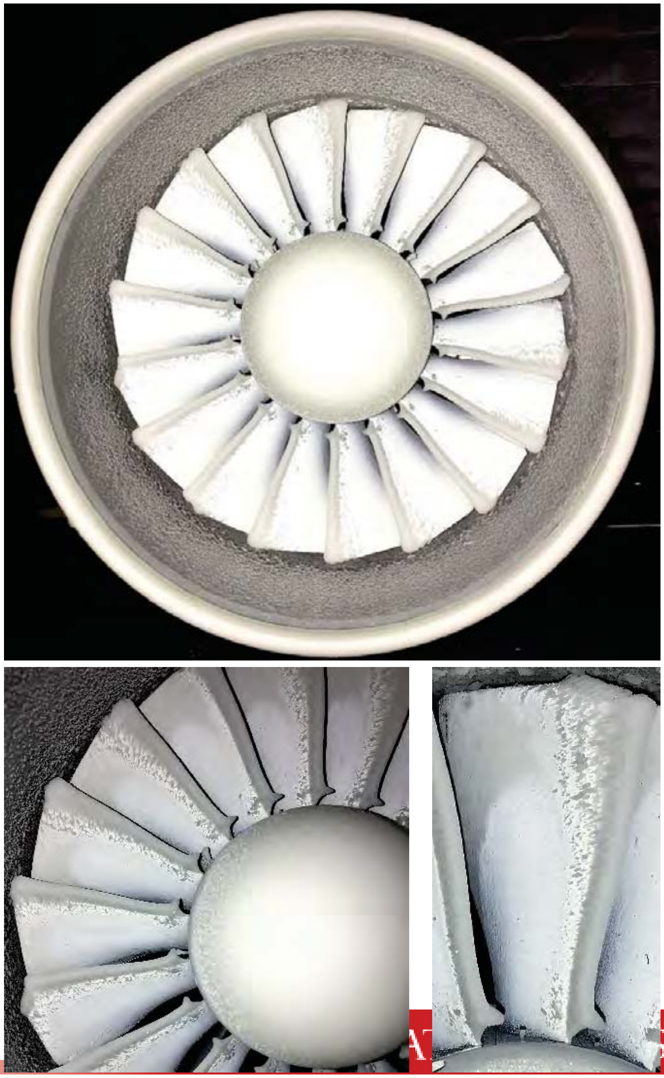
- V_∞ = 15 m/s;
- T_∞ = -15 ℃,
- LWC = 0.5 g/m^3
- Rotation = 2,500 rpm



Copyright(c) Li & Hu 2017
Iowa State University
Email:huhui@iastate.edu

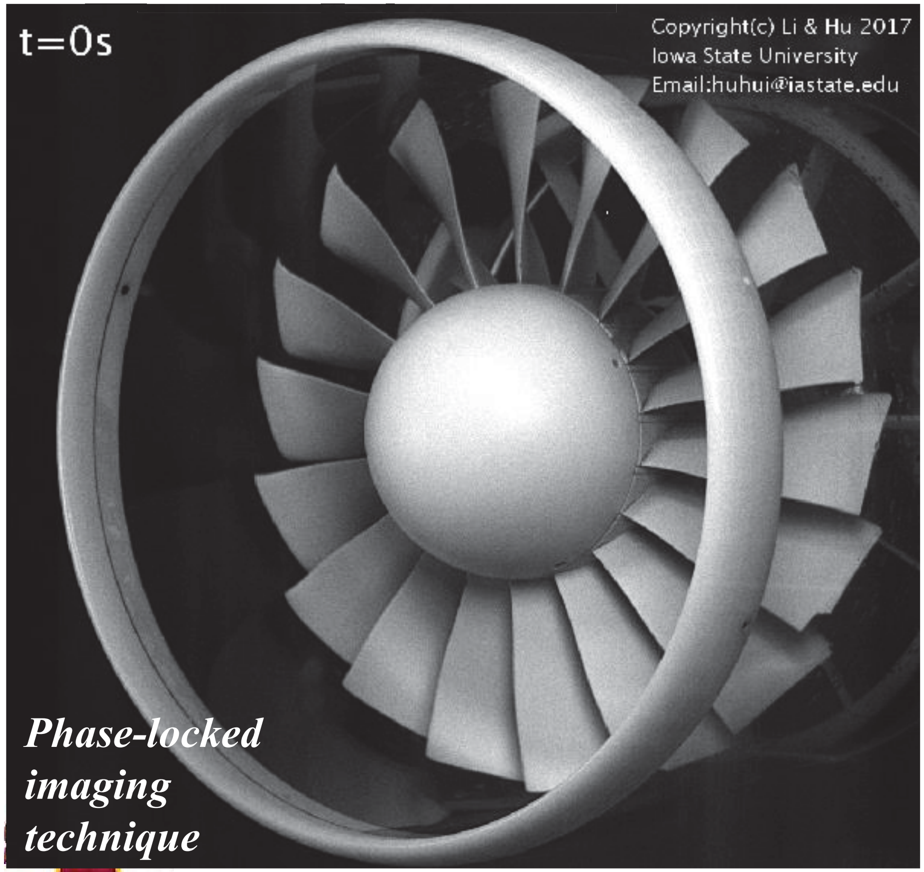
Phase-locked imaging technique

- *Ice shape after 600 seconds of Icing test*



❑ DYNAMIC ICE ACCRETING PROCESS OVER FAN BLADES

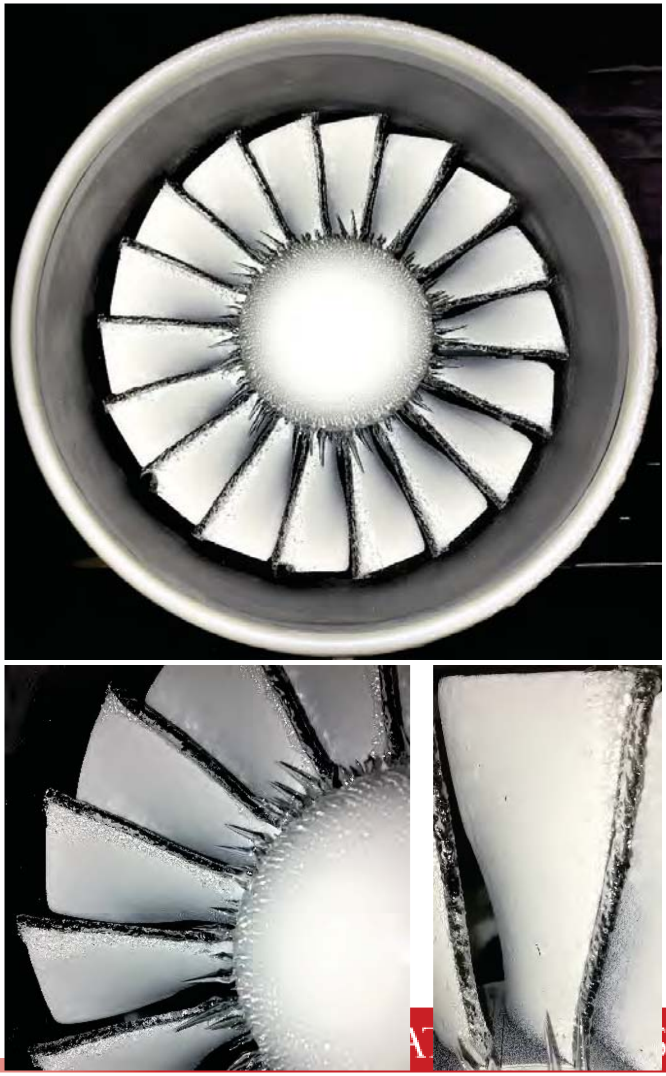
- *V*$_\infty$ = *15 m/s;*
- *T*$_\infty$ = *-5 ℃,*
- *LWC* = *2.0 g/m*^{3}
- *Rotation* = *2,500 rpm*



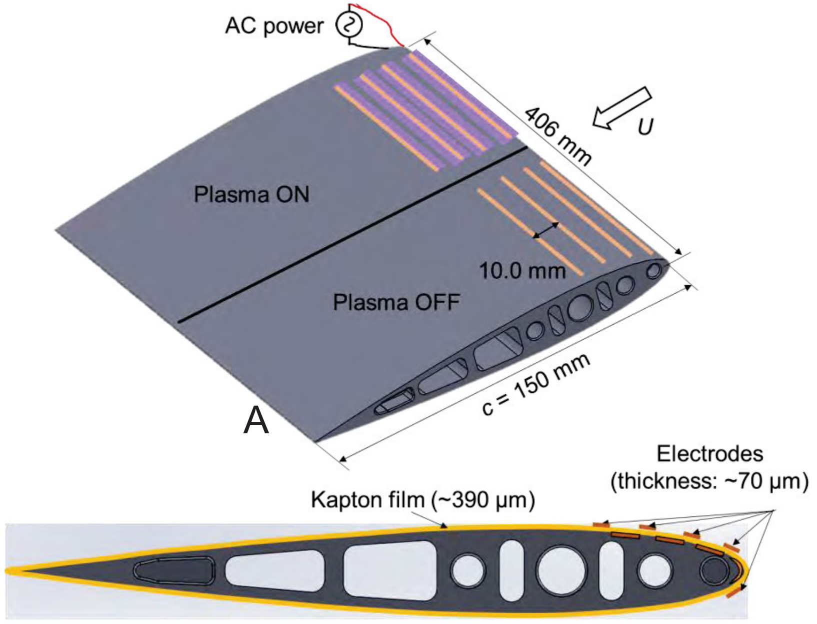
Copyright(c) Li & Hu 2017
Iowa State University
Email:huhui@iastate.edu

Phase-locked imaging technique

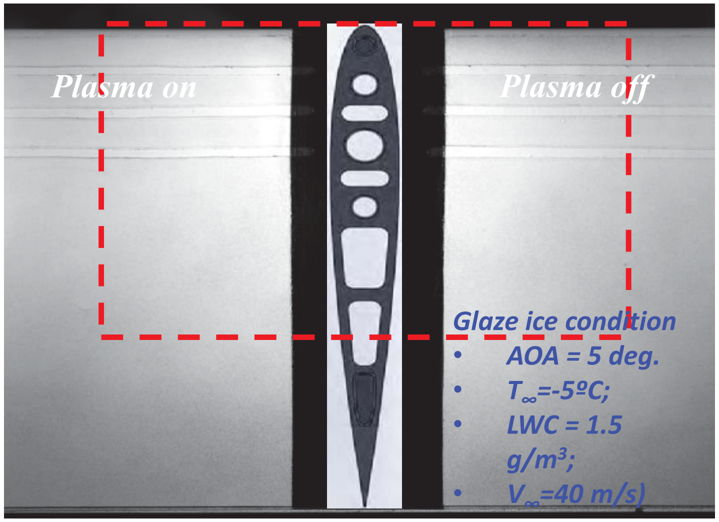
- *Ice shape after 150 seconds of Icing test*



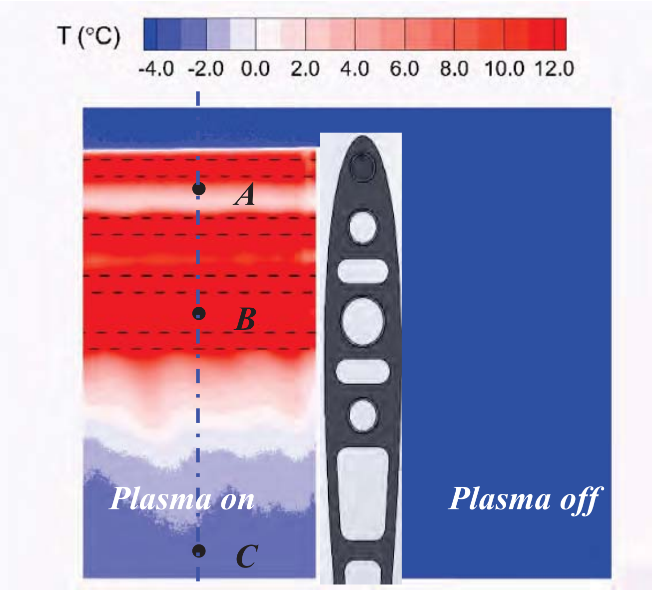
❑ Anti-/De-Icing with DBD Plasma Actuators

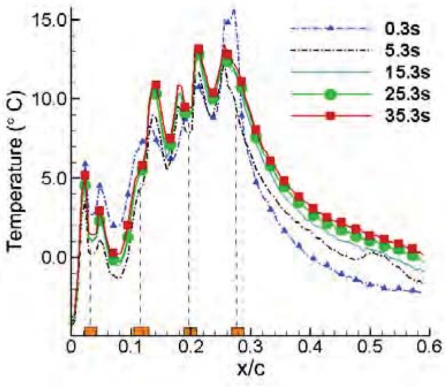


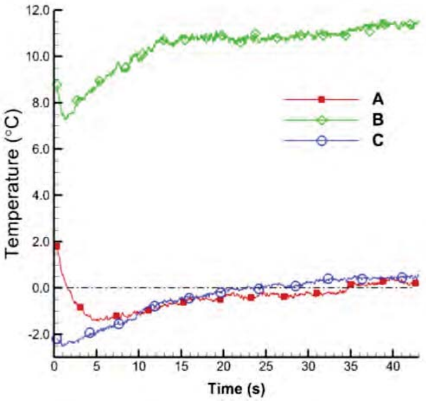
Side View



Glaze ice condition
- *AOA = 5 deg.*
- *T_∞=-5ºC;*
- *LWC = 1.5 g/m³;*
- *V_∞=40 m/s)*







THANK YOU VERY MUCH FOR YOUR TIME!
QUESTIONS?


Aircraft icing


Rotocraft icing


Aero-engine icing


Wind turbine icing


Solar panel icing

Experimental aerodynamics & wind tunnel testing

CFD &multiphase modeling

NDE, MEMS sensors for in-flying icing detection

UAS/MAV, Rotorcraft, wind turbine, power lines

ISU Center for Icing Physics & Anti-/De-icing Technology

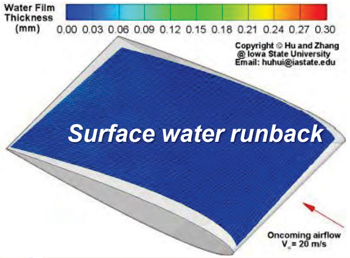
Syetem design and MDO for anti-/de-icing strategy

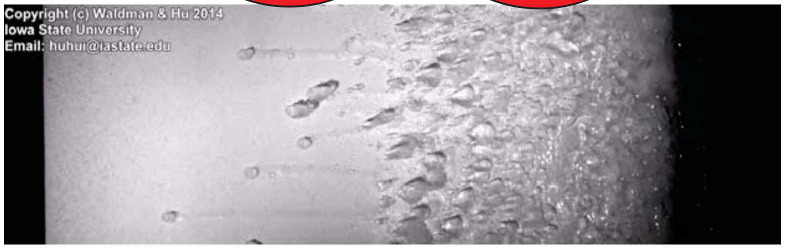
Aero-structure designs for icing mitigation & protection.

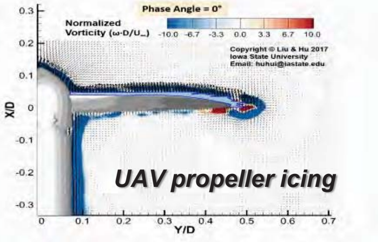
Smart materials, Micro & Nano Mechanics

Super-hydrophobic coatings and surface engineering


Powerline icing


Surface water runback




UAV propeller icing

Durable Low Ice Adhesion Anti-Icing & Ice-Phobic Surfaces

(ONR STTR Program N14A-T013)

10 August 2017

Brief to
NASA Workshop on Low Ice Adhesion Materials

Dr. Ki-Han Kim
Program Officer
Ship Systems and Engineering Research Division (Code 331)
Office of Naval Research

Objectives

- Develop and demonstrate *robust* and *affordable* anti-icing surfaces (prevent ice formation) that are also ice-phobic (reduce ice adhesion to substrates) for superstructure ice protection of surface ships operating in polar regions with no unacceptable ship and environmental impacts

Feb 2011 HMNZS WELLINGTON

Performance Requirements (Major)

- Ice adhesion strength less than 30 kPa that will be proven by independent government laboratory through repeated tests.
- Operate effectively in temperatures down to -30 deg in fresh and salt water.
- Durable and abrasion resistant in simulated operational environments, warm and cold.
- Affordable manufacturing techniques for covering ship superstructures and above-water hull surfaces
- Ease of application to ship structures and other deck equipment, including recoat over existing coating.

Performance Requirements (Additional)

- Resistant to corrosion, mild acids, UV, organisms and organic phosphates
- Operational transparency (>80% in the visible regime)
- Low slipperiness on decks
- Compatibility with current low solar absorbing ship paints
- RF transparency

Participants

Phase I (Aug 2014 – Feb 2015)

- Agiltron Inc. (Woburn, MA) & Dartmouth College (Hanover, NH)
- Luna (Roanoke, VA) & MIT (Cambridge, MA)
- HygraTek & U. of Michigan (Ann Arbor, MI)
- NanoSonic (Pembroke, VA) & Virginia Tech (Blacksburg, VA)

Phase II (Aug 2016 – Dec 2017)

- **HygraTek & U. of Michigan (Ann Arbor, MI)**
- **NanoSonic (Pembroke, VA) & Virginia Tech (Blacksburg, VA)**