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

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

\[ y = 94.777e^{-0.081x} \]
\[ R^2 = 0.129 \]
Data in the Literature - Steel

\[ y = 20.026e^{-0.187x} \]
\[ R^2 = 0.353 \]
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 2\textsuperscript{nd}/3\textsuperscript{rd}
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/m³)
    - Droplet size (median volumetric diameter of 15-40 μm)


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Accreted Ice Types

Glaze/Clear
- 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

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

Mixed
- Variable droplet size
- Combination of glaze and rime ice

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/m³
  - Water droplet mean volumetric diameter of 20 μm
- Ice accumulation and subsequent shedding enabled determination of IASS after data analysis and visual assessment


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

Substituted n-Alkyldimethylsilyl Coatings

non-Hydrogen Bonding

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

Hydrogen Bonding (Donor/Acceptor)

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

Hydrogen Bonding (Acceptor)

C5MEG
Substituted n-Alkyldimethylsilyl Coatings

non-Hydrogen Bonding

Aliphatic
\[ x = 2 \text{ (C3A), 6 (C7A), 10 (C11A)} \]

Hydrogen Bonding (Donor/Acceptor)

Hydroxyl
\[ X = \text{bond, } y = 7 \text{ (C7H), 10 (C10H), 11 (C11H)} \]

Hydrogen Bonding (Acceptor)

C5MEG
Substituted n-Alkyldimethylsilyl Coatings

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**Hydrogen Bonding (Donor/Acceptor)**

- Hydroxyl
  - $X = \text{bond}$, $y = 7$ (C7H), 10 (C10H), 11 (C11H)

**Hydrogen Bonding (Acceptor)**

- C5MEG
Substituted n-Alkyldimethylsilyl Coatings

non-Hydrogen Bonding
Aliphatic
\[ x = 2 \text{ (C3A)}, 6 \text{ (C7A)}, 10 \text{ (C11A)} \]

Hydrogen Bonding (Donor/Acceptor)
Hydroxyl
\[ X = \text{bond}, \ y = 7 \text{ (C7H)}, 10 \text{ (C10H)}, 11 \text{ (C11H)} \]

Hydrogen Bonding (Acceptor)
C5MEG
Surface Properties of Neat Substituted n-Alkylidimethylsilyl Coatings

<table>
<thead>
<tr>
<th>Surface</th>
<th>Mean Roughness (Ra), µm</th>
<th>Receding Water Contact Angle, °</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Avg</td>
<td>Stnd Dev</td>
</tr>
<tr>
<td>Control</td>
<td>0.326</td>
<td>0.048</td>
</tr>
<tr>
<td>non-Hydrogen-Bonding</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C3A</td>
<td>0.324</td>
<td>0.078</td>
</tr>
<tr>
<td>C7A</td>
<td>0.282</td>
<td>0.105</td>
</tr>
<tr>
<td>C11A</td>
<td>0.702</td>
<td>0.298</td>
</tr>
<tr>
<td>Hydrogen Bonding (Donor/Acceptor)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C7H</td>
<td>0.512</td>
<td>0.013</td>
</tr>
<tr>
<td>C10H</td>
<td>0.708</td>
<td>0.100</td>
</tr>
<tr>
<td>C11H</td>
<td>0.320</td>
<td>0.040</td>
</tr>
<tr>
<td>Hydrogen Bonding (Acceptor)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C5MEG</td>
<td>0.390</td>
<td>0.199</td>
</tr>
</tbody>
</table>

ASTM A480: Finish #7
Ice Adhesion Shear Strength of Substituted n-Alkyldimethylsilyl Coatings

![Graph showing the ice adhesion shear strength for various coatings at different temperatures. The x-axis represents the coatings, and the y-axis represents the IASS (Ice Adhesion Shear Stress) in kPa. The graph includes data for -16°C, -12°C, and -8°C.]
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

![Graph showing Adhesion Reduction Factor vs Temperature](graph.png)

- **C3A**
- Control

**Chemical Structures:**
- Aluminum
- C3A

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Substituted n-Alkyldimethylsilyl Coatings: non-Hydrogen Bonding

\[ \text{C3A} + 4 (-\text{CH}_2-) \text{ groups} = \text{C7A} \]
Substituted n-Alkyldimethylsilyl Coatings: non-Hydrogen Bonding

C7A + 4 (–CH₂-) groups = C11A
Substituted n-Alkyldimethylsilyl Coatings: Hydrogen Bonding (Donor/Acceptor)

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)

Change hydrogen atom on terminal methyl group 
(i.e., non-hydrogen bonding) to a hydroxyl group
[i.e., hydrogen bonding (donor/acceptor)]
Substituted n-Alkyl(dimethyl)silyl Coatings: Hydrogen Bonding (Donor/Acceptor)

C7H + 3 (–CH2–) groups = C10H
Substituted n-Alkyldimethylsilyl Coatings: Hydrogen Bonding (Donor/Acceptor)

C10H + 1 (–CH2–) group = C11H
Comparison of non-Hydrogen Bonding & Hydrogen Bonding (Donor/Acceptor) Coatings

![Graph showing comparison of adhesion reduction factor vs. temperature for C7A and control. The graph indicates that the adhesion reduction factor decreases as the temperature decreases, with C7A showing a greater decrease than the control.]
Comparison of non-Hydrogen Bonding & Hydrogen Bonding (Donor/Acceptor) Coatings

![Graph showing Adhesion Reduction Factor vs Temperature]

- C7A
- C7H
- Control

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

Temperature, °C

Adhesion Reduction Factor

C7A
C7H
Control

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

C7 + 4 (–CH2-) groups = C11

Adhesion Reduction Factor vs Temperature, °C

C11A
Control

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

C7 + 4 (–CH₂-) groups = C11

Adhesion Reduction Factor

Temperature, °C

C11A

C11H

Control

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

![Graph showing Adhesion Reduction Factor vs Temperature for C11A, C11H, and Control samples.](image)

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

Temperature, °C

Adhesion Reduction Factor

- C7A
- C7H
- Control
Comparison of non-Hydrogen Bonding & Hydrogen Bonding (Donor/Acceptor) Coatings

![Graph showing adhesion reduction factor vs. temperature for different coatings.](image)

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

![Graph showing adhesion reduction factor vs. temperature for C11A and control samples.](image-url)
Comparison of non-Hydrogen Bonding, Hydrogen Bonding (Donor/Acceptor), & Hydrogen Bonding (Acceptor) Coatings

![Graph showing adhesion reduction factor vs temperature for C11A, C10H, and Control coatings.](image)

Temperature, °C

Adhesion Reduction Factor

- C11A
- C10H
- Control

NASA Workshop on Low Ice Adhesion Materials, 10 August 2017
Comparison of non-Hydrogen Bonding, Hydrogen Bonding (Donor/Acceptor), & Hydrogen Bonding (Acceptor) Coatings

![Graph showing adhesion reduction factor vs. temperature for C11A, C10H, and Control coatings.](image)

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

Adhesion Reduction Factor vs. Temperature, °C

- C11A
- C5MEG
- C10H
- Control

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

Temperature, °C

Adhesion Reduction Factor

- C11A
- C5MEG
- C10H
- Control

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Best Performing Coating from Each Class

![Graph showing adhesion reduction factor vs. temperature for different coatings.]
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
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* non-Hydrogen Bonding
  - Performance dependent upon alkyl chain length
  - Moderate alkyl chain length (C7A) exhibited the best performance
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- **non-Hydrogen Bonding**
  - Performance dependent upon alkyl chain length
  - Moderate alkyl chain length (C7A) exhibited the best performance

- **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)

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

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

※ 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

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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

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Ice Adhesion Shear Strength

\[ \text{Area} = [\text{Thickness} \times (H2 + H4)] + [0.5 \times \text{Thickness} \times (H1 - H2)] + [0.5 \times \text{Thickness} \times (H3 - H4)] \]

Ice Adhesion Shear Strength (IASS) = \( \frac{F_c}{\text{Area}} \)

\( F_c = m_{\text{ice}} r \omega^2 = \frac{m_{\text{ice}} v^2}{r} \)

\( v = r \omega = r \times \text{rpm} \times \frac{2\pi}{60} \text{ s} \)

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

Adhesion Reduction Factor (ARF)
Non-Hydrogen Bonding ARF at -12°C

\[ y = -0.0504x^2 + 0.6272x - 0.2405 \]

\[ R^2 = 1 \]
Non-Hydrogen Bonding ARF at -12°C

\[ y = -0.0504x^2 + 0.6272x - 0.2405 \]

\[ R^2 = 1 \]
Hydrogen Bonding (Donor/Acceptor) ARF at -12°C

\[ y = 0.2466x - 0.6631 \]
\[ R^2 = 0.99972 \]
Hydrogen Bonding (Donor/Acceptor) ARF at -12°C

\[ y = 0.2466x - 0.6631 \]
\[ R^2 = 0.99972 \]
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
  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.
Multiscale Materials & Process Design

- Establishment of Process-Structure-Property relationship
Materials Design Process

- Roughness ($S$) – Adhesion ($P$) relationship establishment

Optimizations: $\min(\text{Adhesion}), \max(\text{Durability})$
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
- Metamodelling and statistical machine learning
- Multi-objective optimization
Research Challenges & Opportunities – Design Parameter Identification

Lack of Fundamental Understanding
- Roughness
- Surface energy
- Adhesion

Identification of Correlations
- Structure descriptor
- Property characterization

Structure-Property Relationship

Test & Evaluation Standards
- Adhesion test protocols
- Durability test protocols
- Terminology
- Classification

Standardization

Ontology

Ontology

Short-Term Long-Term

NASA/CP—2019-219576
Research Challenges & Opportunities
– Modeling & Simulation

Size & Time Scales  Multiscale Simulation Integration  Multiphysics Modeling

- Quantum mechanics
- Mechanical properties
- Molecular dynamics
- Multiphase fluid flow
- Mesoscale simulation
- Crystallization
- Chemical

Prediction Credibility  Sensitivity Analysis  Verification, Validation & Accreditation  Reliable Simulation

- Local sensitivity analysis
- Verification
- Global sensitivity analysis
- Validation
- Verifiable simulation
- Accreditation

Short-Term  Long-Term

NASA/CP—2019-219576
Direct Probe of Surface Nucleation

\[ \chi_{\text{eff}}^{(2)} = \chi_{\text{eff,IR}}^{(2)} + \chi_{\text{eff,Vis}}^{(2)} + \sum_{q} \frac{A_q}{\omega - \omega_q - i\Gamma_q} \]

[Dhinojwala et al. 2012]

Visible

Infrared

Vacuum chamber

Sapphire prism

Heating/cooling stage

SFG

Water/ice

[Graphical representation of experimental setup]

[Graphical data representation with contact angle vs. freezing temperature]
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

Reliable molecular dynamics simulation

Macroscale simulation of droplet impact and ice formation
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?

DREAM BIG

Materials by Design
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

- 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)
# Background & Motivation

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

- 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
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
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  – Evaluation of superhydrophobic materials
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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 waterproofing

5 ft.
Schematic of Rotor Blade
**Adhesion Strength Measurements**

10 ft.

- Beam Fairing
- Test Coupon
- Ice Shield
Adhesion Strength Measurements

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

\[ \tau = \frac{L_{\text{ice}}}{A_s} \]
**Typical Voltage Output During Test**

- Voltage at 0 RPM
- Voltage at Operational RPM

- Spool Up
- Icing Cloud On
- Spool Down

\[ \tau = \frac{L_{\text{ice}}}{A_s} \]
Area Calculation

\[ \tau = \frac{L_{\text{ice}}}{A_s} \]
# Experimental Uncertainty

<table>
<thead>
<tr>
<th>Researcher</th>
<th>Test Type</th>
<th>% deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soltis</td>
<td>Rotor beam</td>
<td>20</td>
</tr>
<tr>
<td>Brouwers</td>
<td>Rotor beam</td>
<td>23</td>
</tr>
<tr>
<td>Hassan</td>
<td>Vibrating beam</td>
<td>40</td>
</tr>
<tr>
<td>Laforte</td>
<td>Centrifuge</td>
<td>18</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>NASA Glenn Icing Research Tunnel Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>MVD</td>
</tr>
<tr>
<td>LWC</td>
</tr>
</tbody>
</table>
Presentation Outline

- Background & Objectives
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- Conclusions
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 \( \mu \text{m} \), 30 \( \mu \text{m} \), 40 \( \mu \text{m} \)

Temperature
-5°C, -10°C, -15°C

Material Surface Characteristics

Surface roughness
20 \( \mu \text{in} \ Ra \), 50 \( \mu \text{in} \ Ra \), 100 \( \mu \text{in} \ Ra \)

Grain direction
0°, 90°

Test Material
Stainless steel 430
Impact velocity 70 m/sec
Effect of Cloud Density

- Over the FAA LWC icing envelope, ice adhesion strength is constant.
- Higher LWC (not to exceed 2 g/m³) might be used to reduce testing time.
Effect of Cloud Density

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).

- 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).
Ice adhesion strength is linearly dependant with ambient temperature.

- 600% reduction in adhesion strength from -15 °C to -5 °C.
**Effect of Particle Size**

- Ice adhesion strength is linearly dependant with MVD
- 52% change in adhesion strength from 20 μm to 40 μm
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
### Environmental and Surface Effects Summary

<table>
<thead>
<tr>
<th>Properties</th>
<th>% Change Over Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>LWC</td>
<td>2</td>
</tr>
<tr>
<td>MVD</td>
<td>52</td>
</tr>
<tr>
<td>Temperature</td>
<td>600</td>
</tr>
<tr>
<td>Surface Roughness</td>
<td>246</td>
</tr>
<tr>
<td>Grain Direction</td>
<td>14</td>
</tr>
</tbody>
</table>

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

Presentation Outline

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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

Brittle erosion

Ductile erosion

Shephard 3/16/12
Motivation

Hypothesis

- Ice adheres to the substrate surface due to mechanical clamping

- Increasing surface roughness increases adhesion strength supporting the hypothesis
Erosion Resistant Material Testing

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 (\( \mu \text{in} \ Ra \)): 20, 50, 100
- Temperature (°C): -8, -12, -16
- MVD (\( \mu \text{m} \)): 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: 15\( \mu \text{m} \)
- Titanium grade 2 substrate

29.2 \( \mu \text{in} \ Ra \)  
62.3 \( \mu \text{in} \ Ra \)  
120 \( \mu \text{in} \ Ra \)
Impact Ice Adhesion Strength for Stainless Steel 430

- Extrapolation predicts to within standard deviation of experiment
Ice Adhesion Comparison

- Un-optimized coatings have higher adhesion strength than uncoated material
- Low surface roughness decreases ice adhesion...
Ice Adhesion Comparison

Aluminum 6061

50 nm Ra @-8°C

1700 nm Ra @ -8°C

Pre Shedding  Post Shedding
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

<table>
<thead>
<tr>
<th>Laser Ablation Level</th>
<th>Ra (um)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.35 W</td>
<td>1.13</td>
</tr>
<tr>
<td>0.6 W</td>
<td>1.95</td>
</tr>
<tr>
<td>1.2 W</td>
<td>5.11</td>
</tr>
</tbody>
</table>
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
Varying Laser Ablation Intensities Create Varying Topographies
1.2 W Ablated Sample

Average Slot Depth—22.5 μm

Average Distance between Slots—75 μm
Results for Varying Surface Substrates

Adhesion Strength Comparison

No Ablation
Results for Varying Surface Substrates

Adhesion Strength Comparison

0.35W Ablation

0.35W Ablation
Results for Varying Surface Substrates

Adhesion Strength Comparison

0.6W Ablation

0.35W Ablation (Ra 1.13)

0.6W Ablation (Ra 1.95)
Results for Varying Surface Substrates

Adhesion Strength Comparison

- Pristine (Ra 0.01)
- 0.35W Ablation (Ra 1.13)
- 0.6W Ablation (Ra 1.95)
- 1.2W Ablation (Ra 5.11)

1.2W Ablation

Distance (µm)

Height (µm)
Results for Varying Surface Substrates

Increased Surface Roughness Corresponds to Increased Adhesion Strength
Modeling based on Surface Roughness?

Shear Force, $S$

Friction Force, $F_f$

$-S + F_f + F_f = 0$

$F_f = \mu N_t$

$S = 2\mu N$

Stress strain relationship

$$E = \frac{\sigma}{\varepsilon}$$

Thermal Coefficient of Thermal Expansion

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

\[ S = 2 \mu E A_0 \alpha \Delta T \]

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

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

Temperature Dependency

---

**Static Coefficient of Friction of Ice on Epoxy Substrate**

\[ S = 2 \mu \varepsilon A_0 \alpha \Delta T \]

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

Ablation Digitization

1. Obtain cross-sectional view using Scanning Electron Microscopy (SEM)
2. Digitize ablated surface
3. Input (x,y) coordinates into computer model
The digitized data is input into Matlab, and the slope is calculated between every point.
Adhesion Strength is calculated for the valley and plateaus.
An average error reduction to less than 9%.
Presentation Outline

• Background & Objectives
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  – Evaluation of superhydrophobic materials
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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

Never Wet (-8 deg C)

- SS 20 μm RA
- Nano Water Guard (-8 deg C)
  - SS 20 μm RA
  - AL 20 μm RA

Hydrobead (-8 deg C)

- SS 20 μm RA

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/m³

~1.5 psi at -12°C
New Testing Technique Being Explored
Issues with the Technique

- 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.

Original CG used for strain gauge calibration

New CG after ice accretion

Complex bending moment is hard to account for due to ice shape
Flat Surface Ice Accretion

- 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
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)
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
Acknowledgements

Project team

• Dr. Dong Meng, MSU Swalm School of Chemical Engineering
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• 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

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Title: Multiscale, Physics-Based Modeling of Impact Ice Adhesion
Cooperative agreement: NNX16AN20A
NASA TM: Eric Kreeger
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
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4. Ice Adhesion Stress: Predictive Models
5. Summary
Motivation

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

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

\[
F_{AD} = \begin{cases} 
14583(T_{MP} - T_s) & 267.7K \leq T_s \leq T_{MP} \\
34475(1.5(267.3 - T_s) + 4) & 258.15K \leq T_s \leq 267.7K \\
34475(3.79(258.15 - T_s) + 12.5) & T_s \leq 258.15K 
\end{cases}
\]
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.
Challenges of Predicting Impact Ice Adhesion

- **Mesoscale**: Continuum Mechanics (FEM)
- **Microscale**: CG MD
- **Atomistic scale**: MD
- **Ab initio**: QM

**Water dimers, clusters**

**H₂O-substrate interactions; interfacial structure**

**Crystal defects; structural anisotropy; substrate structure**

**Ice density; structure porosity**

Scale:
- pm
- nm
- um
- mm
- m
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

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

\( \varphi_{m_n} \) \((n = 1, N_m)\) 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.
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
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4. Ice Adhesion Stress: Predictive Models
5. Summary
EU Horizon 2020 project “Phobic2Ice“

Super-IcePhobic Surfaces to Prevent Ice Formation on Aircraft

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

<table>
<thead>
<tr>
<th>Expected Results</th>
<th>Why it matters</th>
<th>SoA &amp; Gaps – Used &amp; developed technologies</th>
</tr>
</thead>
<tbody>
<tr>
<td>• List of use cases, substrate materials, definition of technical requirements and specifications</td>
<td>• Energy efficient anti-/de-icing systems</td>
<td>• Numerical Ice accretion tools need constant amelioration. No satisfactory tool exists combining aerodynamics, thermodynamics, and material properties</td>
</tr>
<tr>
<td>• Modelling and simulation of ice accretion</td>
<td>• Icing-detection sensors &amp; early warning systems</td>
<td>• No fabrication technology for obtaining a durable, erosion resilient icephobic coating is currently existing</td>
</tr>
<tr>
<td>• Coating development</td>
<td>• Ice accretion models depending on material properties</td>
<td></td>
</tr>
<tr>
<td>• Characterization and testing, development of ice detection sensors</td>
<td></td>
<td></td>
</tr>
<tr>
<td>• Coating application on prototype component &amp; full-scale testing</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Coating Development

Icing Simulations

Wind Tunnel Testing

www.phobic2ice.com
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
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, $\tau_{int}$, is calculated by

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

- $\varepsilon_{EF-al}$: 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

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Material</th>
<th>Surface Treatment</th>
<th>Water CA</th>
<th>Water RoA</th>
<th>Surface Roughness, $R_a / R_z$ (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>Al 2024</td>
<td>Polished + TSA$^1$</td>
<td>60°</td>
<td>&gt;90°</td>
<td>0.01 / 0.16</td>
</tr>
<tr>
<td>1</td>
<td>Al 2024</td>
<td>Polished + TSA + Episurf$^2$</td>
<td>122°</td>
<td>&gt;90°</td>
<td>0.02 / 0.16</td>
</tr>
<tr>
<td>0</td>
<td>Ti6Al4V</td>
<td>Anodized$^3$ + Episurf</td>
<td>155°</td>
<td>20°</td>
<td>0.58 / 4.40</td>
</tr>
</tbody>
</table>

1. Tartaric Sulphuric Acid Anodizing  
2. Commercially available perfluoropolyether phosphonate compound in a HFO solvent  
3. TiO$_2$-Nanotube Layer

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

### Samples

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Material</th>
<th>Surface Treatment</th>
<th>Water CA</th>
<th>Water RoA</th>
<th>Surface Roughness, $R_a / R_z$ (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>Al 2024</td>
<td>Polished + TSA$^1$</td>
<td>60°</td>
<td>&gt;90°</td>
<td>0.01 / 0.16</td>
</tr>
<tr>
<td>1</td>
<td>Al 2024</td>
<td>Polished + TSA + Episurf$^2$</td>
<td>122°</td>
<td>&gt;90°</td>
<td>0.02 / 0.16</td>
</tr>
<tr>
<td>0</td>
<td>Ti6Al4V</td>
<td>Anodized$^3$ + Episurf</td>
<td>155°</td>
<td>20°</td>
<td>0.58 / 4.40</td>
</tr>
</tbody>
</table>

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

<table>
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<td>1.00</td>
</tr>
<tr>
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<td>0.55</td>
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The Icing time was sufficient to obtain the desired ice thickness.
Preliminary Results

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<td>(g/m³)</td>
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## Preliminary Results

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1.60 mm

\[ h_{\text{ice}} \]
## Preliminary Results

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Reference (Sample 3): Superhydrophobic (Sample 0)
Preliminary Results

Strain gauge data analysis – identification of crack initiation
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
Preliminary Results

Strain gauge data analysis with HSC video

CRACK INITIATION (look closely)
Preliminary Results

Strain gauge data analysis with HSC video
Preliminary Results

Strain gauge data analysis with HSC video
Preliminary Results

Ice Interfacial Shear Strength (kPa)

Icing Condition

All Samples

Reference

Hydrophobic

Superhydrophobic

NASA/CP—2019-219576
Outline of Presentation

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

$$F_{AD} = f(\varphi_{m_1}, \ldots, \varphi_{m_{N_m}})$$

- $\varphi_{m_n}$ ($n = 1, N_m$) 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

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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

- **Ab initio: QM**
  - water dimers, clusters

- **Atomistic scale: MD**
  - crystal defects; structural anisotropy; substrate structure

- **Microscale: CG MD**
  - H₂O-substrate interactions; interfacial structure

- **Mesoscale: Continuum Mechanics (FEM)**
  - ice density; structure porosity
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

\[
F_{AD} = f \left( \phi_{m_1}, \ldots, \phi_{m_{N_m}}, \phi_{\mu_1}, \ldots, \phi_{\mu_{N_{\mu}}}, \phi_{n_1}, \ldots, \phi_{n_{N_n}} \right)
\]

\(\phi_{m_n} (n = 1, N_m)\) represent the relevant macroscale parameters
\(\phi_{\mu_n} (n = 1, N_{\mu})\), represent the relevant mesoscale parameters
\(\phi_{n_n} (n = 1, N_n)\) 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.
Predictive Modeling (1\(\mu\)m-100\(\mu\)m)

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 1\(\mu\)m-100\(\mu\)m, 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.
Preliminary Results: Force Field Calibration Liquid State

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

Preliminary Results: Equilibrated \textit{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 $q_6$ order parameter.
Preliminary Results: Ice Nucleation by ”Seeding”

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

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Summary

Multiscale Modeling of Ice Adhesion

Ab initio: QM

Atomistic scale: MD

Microscale: CG MD

Mesoscale: Continuum Mechanics (FEM)

water dimers, clusters

H₂O-substrate interactions; interfacial structure

crystal defects; structural anisotropy; substrate structure

ice density; structure porosity

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

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

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.
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 \( d(x, t) \)

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

$$\Pi = \int \left\{ [1 - g(d)] W_s(\epsilon) + g(d) W_0 + \frac{a}{2} |\nabla d|^2 \right\} dV$$

- Strain energy
- Damage energy
- Gradient energy
- Fracture energy

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)

\[ r_0 \approx \frac{1}{2\pi} \left( \frac{K_{lc}}{\sigma_y} \right)^2 \approx \frac{1}{2\pi} \left( \frac{100 \text{kPa} \cdot \text{m}^{1/2}}{10 \text{MPa}} \right)^2 \approx 10 \mu\text{m} \]

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

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

Example of cohesive elem + XFEM
5. Design of In Situ Ice Adhesion/Cohesion Measurement System
Background:

Strength vs. Fracture Mechanics View

Brittle Materials,

\[ \text{Energy} = \text{Elastic} + \text{Surface} \]

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

Ductile Materials,

\[ \sigma_c \sqrt{a} \approx \text{Constant} \Rightarrow \sqrt{\frac{2\gamma E}{\pi}} \]

\[ \Gamma \sim 100 \text{kJ/m}^2 \]

Irwin and Orowan; apply it in an effective way,

\[ \Gamma = 2\gamma + W_p : \text{Total Fracture Energy} \]

\[ \sigma_c \sqrt{a} = \sqrt{\frac{\Gamma E}{\pi}} ; \quad \sigma_c : \text{Adhesion Strength} \]

\[ a : \text{Crack Length (roughness)} \]
Intrinsic vs. Effective Adhesion

\[ \Gamma_{\text{eff}} = 2\gamma_0 + W_p(r_p) \]

---

**Total Fracture Energy** = **Intrinsic Cohesive Energy** + **Plastic Dissipation Energy**

(Example for polymer adhesion)

Aerospace Engineering

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Strength Testing

Ice Adhesion Test Results

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)
5.1 Experimental Setup

In situ Characterization
Oncoming airflow with super-cooled water droplets

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

In-Tunnel Growth, Off-Tunnel Testing
5.2 Well Characterized Fracture Experiments

- 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

\[ G_0 = G \bigg|_{a=a_c} \]
Well Characterized Fracture Experiments

\[ G_{ave} = 5.07 \text{ J/m}^2 \]

PVC substrate

\[ G_{ave} = 1.37 \text{ J/m}^2 \]

Acrylic substrate

Ra 0.008 um
Rmax 0.08 um

Ra 0.201 um
Rmax 2.479 um

NASA/CP—2019-219576
5.4 Model Calibration, Cohesive Surfaces

- 2-D 4-node quadrilateral linear plane strain elements (CPE4R).
- Single layer of Cohesive surface (Bilinear CZM).
- Refined element size of 20x20μm
Calibration of Interfacial Cohesive Strength, $\hat{\sigma}$

Bilinear traction-separation curve

$$k_0 = \frac{2G_0}{\delta_{max} \delta_c}$$

$$G_0 = \frac{(1-v^2)K_{IC}^2}{E} = \hat{\sigma} \delta_c / 2$$

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

$G_0 \sim 4 \text{ J/m}^2$

Calibration Scheme

$G_0_{\text{FEM}} \approx G_0_{\text{Experiment}}$

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

Interfacial fracture parameters

Aerospace Engineering

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Estimate of cohesive fracture parameters

\[ P_c \text{ (experiments)} \]

\[ G_0 = 4 \text{ J/m}^2 \]
\[ \hat{\sigma} = 0.1 \text{ MPa} \]
5.4 Parametric Study for the design of experimental apparatus (Modified Blister Test)

\[ a_0 \approx 7.5 \text{ mm} \]

<table>
<thead>
<tr>
<th>Material</th>
<th>E (GPa)</th>
<th>( \nu )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ice</td>
<td>9.8</td>
<td>0.3</td>
</tr>
<tr>
<td>Aluminum</td>
<td>70</td>
<td>0.33</td>
</tr>
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Start of Cohesion Zone

Base
(a) Parametric Study (Loading configuration)
(b) Parametric Study (Crack aspect ratio)

Role of plug diameter, $d$

Role of crack length, $a_0$

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5.5 Implementation of Experimental Apparatus

- Pneumatic Cylinder
- LVTD for Displacement Measurements
- Load Cell
- 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

Machine Compliance

Experimental Results

\[ y = 0.3866x + 10.16 \]

\[ R^2 = 0.8637 \]
(b) SCB Modeling

FEM results vs. Elastic Beam Theory

\[ \delta = f(P, a) = \frac{Pa^3}{3EI} \]

Cohesive parameters:

\[ G = 1.5 \, J / m^2, \quad \hat{\sigma} \approx 1.0 \, MPa \]
(c) SCB Comparison

Experimental Measurement

Experiment and FEM 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
• Test Group (06-28-2017)
• Plate: As received 6061 AL
• Ice thickness: 4 mm
• Temp: -17.5 °C
Blister Test  Experimental Results

\[ P_{\text{max}} = 160.5 \pm 10.3 \text{ (N)} \]
\[ G_{\text{ave}} = 1.56 \pm 0.19 \text{ (J/m}^2\text{)} \]
(e) Blister Test Modeling

\[ \delta = \frac{3(1-v_i^2)PR^2}{4\pi E_1h^3} \]

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.

FEA vs Experimental Data

Steady state crack propagation

Force (N)

Displacement (mm)

Crack propagation!

Or

Plasticity in the Ice?
6. Next Step

Oncoming airflow with super-cooled water droplets

Ice accreting airfoil model

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

Test under Controlled Environment
Next Step

Rough icephobic surface

Ra=10.15μm

Surface form (5X)

Ra=0.69μm

Surface waviness (50X, Low pass filter)

Ra=0.175μm

Surface roughness (50X, High pass filter)

Ra=0.30μm

Smooth icephobic surface

Ra=0.94μm

Surface form (5X)

Ra=0.76μm

Surface waviness (5X, Low pass filter)

Ra=0.30μm

Surface roughness (5X, High pass filter)
Relation with project components

- **Experiments**: Microscopy, Fracture tests
- **Phase-field model**: Calibration, Microstructure of ice
- **Mesoscopic modeling**: Atomistic simulations, Basic parameters
- **Macroscopic modeling**: Surface chemistry, Microstructures & surface morphology
- **XFEM**: Effective fracture toughness
- **Cohesive model**: Traction separation curve
- **Validation**: Fracture tests

Aerospace Engineering
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

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

Aircraft icing  Rotorcraft icing  Aer-engine icing  Wind turbine icing

Solar panel icing  Powerline icing

NDE, MEMS sensors for in-flying icing detection
Experimental aerodynamics & wind tunnel testing
CFD & multiphase modeling
UAS/MAV, Rotorcraft, wind turbine, power lines

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

System 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

Water Film Thickness [in]

normalized

0.0 0.0 0.0 0.0 0.0 0.1 0.15 0.2 0.25 0.3 0.4 0.5

Surface water runback

UAV propeller icing

Copyright © 2019, Iowa State University
Email: WadihHendawi@iastate.edu

Phase Angle = φ°

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Email: WadihHendawi@iastate.edu
• **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: $0.4m \times 0.4m \times 2.0m$
  - Airflow Velocity: $V_{\infty} = 5 \sim 100 \text{ m/s}$
  - Air Temperature: $T_{\infty} = -25 ^\circ \text{C} \sim 20 ^\circ \text{C}$
  - Droplet size: $D_{\text{droplet}} = 10 \sim 100 \mu \text{m}$
  - Liquid Water Content: $LWC = 0.1 \sim 10 \text{ g/m}^3$

• The large LWC range allows ISU-IRT tunnel to be run over a range of conditions from dry rime icing to wet glaze icing.
- **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: \( D = 2.4 \text{mm} \)
- Impact velocity: \( V_{\text{impact}} = 1.60 \text{m/s} \)
- Reynolds number: \( Re = 4000 \)
- Weber number: \( We = 90 \)

*Reference image with no droplets*

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

*Volume measure by DIP technique*
*Revised volume by combining DIP and Side-view results*
*Volume calculated based on initial droplet diameter*

*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:**

\[
E_{k0} + E_{p0} + E_{s0} = E_k + E_p + E_s + W
\]

Before impact

\[
E_{k0} = \frac{1}{2}mv^2 = \frac{\pi}{12} \rho \nu^2 d_0^5
\]

\[
E_{p0} = mgd_0 = \frac{\pi}{12} \rho g d_0^4
\]

\[
E_{s0} = \gamma S = \pi d_0^2 \gamma
\]

During impact

\[
E_k = \frac{1}{2}mv^2
\]

\[
E_p = mgd
\]

\[
W = f(y, \mu)
\]

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

<table>
<thead>
<tr>
<th>We</th>
<th>(E_0) (10^{-6} J)</th>
<th>(E_{df}) (10^{-6} J)</th>
<th>(E_{d1}) (10^{-6} J)</th>
<th>(E_{d1}/E_{df})</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>3.16</td>
<td>2.69</td>
<td>2.30</td>
<td>85.2%</td>
</tr>
<tr>
<td>110</td>
<td>10.11</td>
<td>9.06</td>
<td>8.67</td>
<td>95.6%</td>
</tr>
<tr>
<td>195</td>
<td>16.71</td>
<td>14.67</td>
<td>14.22</td>
<td>96.9%</td>
</tr>
</tbody>
</table>

Viscous dissipation function: \(\phi \propto \mu U_0^2\)

- The energy dissipated during the spreading stage would increase as the increase of the We;

WIND-DRIVEN FILM/RIWLET FLOWS (DRY SURFACE CONDITION)

Effects of various important parameters:
- Temperature of the surface
- Roughness of the test surfaces
- Thermal conductivity of the substrates
- Surface hydrophobicity
- Coatings or nano-structures on the test surfaces

Water flow rate: Q = 100 min
- Free stream airflow: v = 10 m/s
- Free stream airflow: v = 20 m/s

Water flow rate: Q = 100 min
- Free stream airflow: v = 15 m/s
**Test Conditions:**
- **Angle of attack:** $\alpha \approx 0.0 \text{ deg.}$
- **Temperature:** $T \approx 20 \ ^\circ \text{C.}$
- **LWC Level:** $\text{LWC} = 5.0 \ \text{g/m}^3$
- **Frame rate:** $f = 30 \ \text{Hz}$

**Airflow velocity**
- $V_\infty = 15 \text{m/s}$
- $V_\infty = 20 \text{m/s}$
- $V_\infty = 25 \text{m/s}$
• Test Conditions:
  • Oncoming airflow velocity: \( V_\infty \approx 35 \text{ m/s} \)
  • Angle of attack of the airfoil: \( \alpha \approx 5 \text{ deg.} \)
  • Airflow Temperature: \( T \approx -8 \text{ °C} \)
  • Liquid water content (LWC): \( \text{LWC} = 3.0 \text{ g/m}^3 \)
  • Image acquisition rate \( f = 150\text{Hz}, \text{10X replay} \)

**Dynamic Glaze Ice Accretion over an Airfoil Surface**

- $V_\infty = 20 \text{ m/s}$
- $T_\infty = -8.0 \, ^\circ\text{C}$
- $\alpha = 5^\circ$
- $\text{LWC} = 1.1 \, \text{g/m}^3$
- $V_\infty = 40 \text{ m/s}$
- $V_\infty = 60 \text{ m/s}$

**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](image)

- Glaze ice accreting process
  \[V_\infty = 40 \text{ m/s; } T_\infty = -8.0 \text{ °C; } LWC = 1.0 \text{ g/m}^3\]

- Rime ice accreting process
  \[V_\infty = 40 \text{ m/s; } T_\infty = -8.0 \text{ °C; } LWC = 0.20 \text{ g/m}^3\]
HYDROPHILIC, HYDROPHOBIC, AND SUPER-HYDROPHOBIC SURFACE

- A water droplet over a smooth surface

Using materials with low wettability

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

\[ \gamma_{LG} \cos \theta_Y = \gamma_{SG} - \gamma_{LS} \]

Cassie-Baxter state

\[ \cos \theta_{CB} = \varphi \cos \theta_Y + \varphi - 1 \]

Wenzel state

\[ \cos \theta_W = n \cos \theta_Y \]

A water droplet over a rough surface
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)*

### Comparison of Test Surfaces

<table>
<thead>
<tr>
<th>Surfaces</th>
<th>Static CA</th>
<th>Advancing CA</th>
<th>Receding CA</th>
<th>Hysteresis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrophilic (baseline)</td>
<td>30~80</td>
<td>70~105</td>
<td>15~60</td>
<td>&lt;70</td>
</tr>
<tr>
<td>Goose Feather</td>
<td>75~145</td>
<td>142~158</td>
<td>70~80</td>
<td>&lt;45</td>
</tr>
<tr>
<td>SLIPS</td>
<td>108~112</td>
<td>105~115</td>
<td>90~105</td>
<td>&lt;15</td>
</tr>
<tr>
<td>Hydro-bead SHS</td>
<td>155~160</td>
<td>156~163</td>
<td>151~158</td>
<td>&lt;5</td>
</tr>
</tbody>
</table>
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/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

Typical parameter pertinent to aircraft icing phenomena:
- \( D = 10 \, \mu m \)
- \( V_{\text{impact}} = 100 \, m/s \)
- \( We \approx 1,250 \)
- \( Re \approx 500 \)
- \( Oh \approx 0.071 \)

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

- \( \rho \): Water density
- \( \mu \): Water viscosity
- \( \sigma \): Surface tension
- \( D \): Droplet diameter
- \( U \): Impact velocity

Oncoming airflow with supercooled water droplets
Dynamic Droplet Impingement onto Different Surfaces

Frame rate @10K FPS

0.1 ms

0.1 ms

0.1 ms

0.1 ms

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.)

Hydrophilic, Hydrophobic and Superhydrophobic

SLIPS coated surface
(Wong et al., 2011)

Hydro bead coated surface
(provided by seashell technology)

Lotus leaves

- Hydrophilic; $\theta < 90^\circ$
- Hydrophobic; $90^\circ < \theta < 150^\circ$
- Superhydrophobic; $\theta > 150^\circ$
Measuring Advancing and Receding Angles of Water Droplets

According to Quéré et al. (1998):

\[ F_{\text{cap}} = \pi R y_{LG} \sin\left(\frac{\theta_{\text{adv}} + \theta_{\text{rec}}}{2}\right)(\cos(\theta_{\text{adv}}) - \cos(\theta_{\text{rec}})) \]

<table>
<thead>
<tr>
<th></th>
<th>Advancing contact angle (°)</th>
<th>Receding contact angle (°)</th>
<th>Hysteresis (°)</th>
<th>Ratio of Capillary forces</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrophilic (Enamel)</td>
<td>70 ~ 105</td>
<td>15 ~ 60</td>
<td>&gt; 50</td>
<td>1.0</td>
</tr>
<tr>
<td>SLIPS</td>
<td>105 ~ 115</td>
<td>90 ~ 105</td>
<td>&lt; 15</td>
<td>(~0.25)</td>
</tr>
<tr>
<td>Superhydrophobic (Hydrobead)</td>
<td>144 ~ 148</td>
<td>141 ~ 145</td>
<td>&lt; 5</td>
<td>(~0.04)</td>
</tr>
</tbody>
</table>

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

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

Hydrophilic

SLIPS

Superhydrophobic coating
Measurements of Ice Adhesion Force over Different Surfaces

<table>
<thead>
<tr>
<th>Test surface</th>
<th>Compared Surfaces</th>
<th>Ice adhesion strength @ $T_{\text{wall}} = -10 , ^\circ\text{C}$ [KPa]</th>
<th>Std. deviation @ $T_{\text{wall}} = -10 , ^\circ\text{C}$ [KPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Al, 220 Grit</td>
<td>450</td>
<td>70</td>
</tr>
<tr>
<td>2</td>
<td>Al, 400 Grit</td>
<td>390</td>
<td>60</td>
</tr>
<tr>
<td>3</td>
<td>Al, 1000 Grit</td>
<td>340</td>
<td>40</td>
</tr>
<tr>
<td>4</td>
<td>Al, 2000 Grit</td>
<td>300</td>
<td>60</td>
</tr>
<tr>
<td>5</td>
<td>Al, mirror finish</td>
<td>130</td>
<td>60</td>
</tr>
<tr>
<td>6</td>
<td>Enamel</td>
<td>1,400</td>
<td>130</td>
</tr>
<tr>
<td>7</td>
<td>Teflon</td>
<td>420</td>
<td>60</td>
</tr>
<tr>
<td>8</td>
<td>Hydrobead SHP</td>
<td>370</td>
<td>90</td>
</tr>
<tr>
<td>9</td>
<td>SLIPS</td>
<td>60</td>
<td>10</td>
</tr>
<tr>
<td>10</td>
<td>PFA plastic</td>
<td>570</td>
<td>60</td>
</tr>
<tr>
<td>11</td>
<td>Stainless steel</td>
<td>550</td>
<td>130</td>
</tr>
<tr>
<td>12</td>
<td>NeverWet</td>
<td>420</td>
<td>40</td>
</tr>
</tbody>
</table>
Effects of Bio-Inspired Coatings on Impact Ice Accretion

- $T_i = -8^\circ C$
- $V_i = 20 m/s$
- MVD = 40 pm, LWC = 2.5 g/m$^3$

With Super-hydrophobic Surface coating

Without Super-hydrophobic Surface coating
**Effects of Bio-Inspired Coatings on Impact Ice Accretion**

- **Hydrophilic enamel**
- **SLIPS**
  \[
  \frac{F_{\text{cap, enamel}}}{F_{\text{cap, SLIPS}}} \approx 4
  \]
- **Hydrobead Superhydrophobic**
  \[
  \frac{F_{\text{cap, enamel}}}{F_{\text{cap, SHP}}} \approx 25
  \]

**Conditions**:

- \( T_\infty = -4^\circ \text{C} \)
- \( V_\infty = 30 \text{ m/s} \)
- **MVD** = 40 \( \mu \text{m} \)
- **LWC** = 4.0 g/m\(^3\)

**Coatings**:

- **Hydrobead (Superhydrophobic)**
- **Enamel (hydrophilic)**
- **SLIPS** (Wong et al., 2011)
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, $MVD_{\text{droplet}} = 10 \sim 100 \mu m$
- 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.

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.

<table>
<thead>
<tr>
<th>Compared surface</th>
<th>Static contact angle (°) $\theta_{\text{static}}$</th>
<th>Advancing contact angle (°) $\theta_{\text{advancing}}$</th>
<th>Receding contact angle (°) $\theta_{\text{receding}}$</th>
<th>Hysteresis (°) $\Delta\theta = \theta_{\text{advancing}} - \theta_{\text{receding}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Airfoil Surface</td>
<td>65</td>
<td>105</td>
<td>50</td>
<td>55</td>
</tr>
<tr>
<td>20% t-PDMS</td>
<td>110</td>
<td>114</td>
<td>78</td>
<td>36</td>
</tr>
<tr>
<td>40% t-PDMS</td>
<td>109</td>
<td>116</td>
<td>77</td>
<td>38</td>
</tr>
<tr>
<td>60% t-PDMS</td>
<td>108</td>
<td>115</td>
<td>78</td>
<td>37</td>
</tr>
<tr>
<td>80% t-PDMS</td>
<td>110</td>
<td>118</td>
<td>80</td>
<td>38</td>
</tr>
</tbody>
</table>
Icephobic Soft Material (PDMS) with Adjustable Stiffness

- Ice adhesion strength over soft PDMS surface:

<table>
<thead>
<tr>
<th>Concentration (%)</th>
<th>T\text{wall} = -5 °C</th>
<th>T\text{wall} = -10 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean Adhesion Strength (KPa)</td>
<td>Std. deviation (KPa)</td>
</tr>
<tr>
<td>20</td>
<td>5.3</td>
<td>0.9</td>
</tr>
<tr>
<td>40</td>
<td>4.7</td>
<td>1.2</td>
</tr>
<tr>
<td>60</td>
<td>3.6</td>
<td>0.5</td>
</tr>
<tr>
<td>80</td>
<td>1.4</td>
<td>0.5</td>
</tr>
</tbody>
</table>

- Ice adhesion strength over soft PDMS surface is extremely lower than conventional surfaces.

- Measurement results agree with classical adhesion mechanism, where $\tau_{\text{ice}}$ is proportional to $\mu^{0.5}$ (Chaudhury and Kim 2007)

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

Shear modulus, $\mu$ (kPa)
Dynamic Impacts of Droplets onto Soft PDMS Surfaces

- Egg Drop Experiment

- Rigid Aluminum Surface (We=900)

- Soft PDMS surface (We=900)

- 50% t-PDMS

- 60% t-PDMS

- 70% t-PDMS

- 80% t-PDMS
- **Test Conditions:**

  - \( V_\infty = 40 \text{ m/s} \)
  - \( T_\infty = -5.0 \, ^\circ\text{C} \)
  - \( LWC = 1.0 \text{ g/m}^3 \)
  - \( AOA = 5.0^\circ \)
EXPERIMENTAL SETUP FOR AERO-ENGINE ICING STUDY

### Experimental Setup

- **Icing Wind Tunnel**
  - Ice accretion on the rotating spinner
  - Outcoming air flow
  - Supercooled water droplets
  - JR3 Load Cell
  - Current Transducer
  - Speed Controller
  - Tachometer
  - Digital Delay Generator
  - Host computer

### Liquid Water Content

<table>
<thead>
<tr>
<th>Temperature</th>
<th>0.5 g/m³</th>
<th>1.0 g/m³</th>
<th>1.5 g/m³</th>
<th>2.0 g/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>-5°C</td>
<td><img src="image1" alt="Images" /></td>
<td><img src="image2" alt="Images" /></td>
<td><img src="image3" alt="Images" /></td>
<td><img src="image4" alt="Images" /></td>
</tr>
<tr>
<td>-10°C</td>
<td><img src="image5" alt="Images" /></td>
<td><img src="image6" alt="Images" /></td>
<td><img src="image7" alt="Images" /></td>
<td><img src="image8" alt="Images" /></td>
</tr>
<tr>
<td>-15°C</td>
<td><img src="image9" alt="Images" /></td>
<td><img src="image10" alt="Images" /></td>
<td><img src="image11" alt="Images" /></td>
<td><img src="image12" alt="Images" /></td>
</tr>
</tbody>
</table>

### Parameters Comparison

<table>
<thead>
<tr>
<th>Parameters</th>
<th>CFM 56-2/3 Turbofan</th>
<th>Aero-engine Model</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter (m)</td>
<td>1.52</td>
<td>0.2</td>
</tr>
<tr>
<td>Max Rotation Speed (rpm)</td>
<td>5175</td>
<td>4000</td>
</tr>
<tr>
<td>Cruising Speed (m/s)</td>
<td>222 (0.74 Ma)</td>
<td>15</td>
</tr>
<tr>
<td>Cruising Rotation Speed (rpm)</td>
<td>4900</td>
<td>2500</td>
</tr>
<tr>
<td>Temperature Range (°C)</td>
<td>-40 ~ 20</td>
<td>-15 ~ -5</td>
</tr>
<tr>
<td>Liquid Water Content (g/m³)</td>
<td>0.1 ~ 2.0</td>
<td>0.6 ~ 2.4</td>
</tr>
<tr>
<td>Advanced Ratio, J</td>
<td>1.80</td>
<td>1.80</td>
</tr>
</tbody>
</table>

### Intermittent Maximum Atmospheric Icing Conditions

Intermittent maximum atmospheric icing conditions from 14 CFR Part 25 Appendix C[1]
**Dynamic Ice Accreting Process over Fan Blades**

- $V_\infty = 15\, \text{m/s}$
- $T_\infty = -15\, \text{°C}$
- LWC = 0.5 g/m$^3$
- Rotation = 2,500 rpm

*Ice shape after 600 seconds of Icing test*

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Phase-locked imaging technique

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Iowa State University
Email: huhiu@iastate.edu
**DYNAMIC ICE ACCRETING PROCESS OVER FAN BLADES**

- $V_\infty = 15 \text{ m/s}$
- $T_\infty = -5 \degree \text{C}$
- $\text{LWC} = 2.0 \text{ g/m}^3$
- Rotation = 2,500 rpm

*Ice shape after 150 seconds of Icing test*

*Phase-locked imaging technique*
**Anti-/De-Icing with DBD Plasma Actuators**

- **Glaze ice condition**
  - AOA = 5 deg.
  - $T_\infty = -5^\circ C$
  - LWC = 1.5 g/m$^3$
  - $V_\infty = 40$ m/s

- Electrodes (thickness: ~70 µm)
- Kapton film (~390 µm)
- AC power

**Graphs:**
- Temperature vs. x/c
- Temperature vs. Time

**Images:**
- Plasma on
- Plasma off
THANK YOU VERY MUCH FOR YOUR TIME!

QUESTIONS?

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

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