Applicant (Principal Investigator)

Hallie Boyer Chelmo, Assistant Professor
University of North Dakota, Grand Forks, ND
College of Engineering and Mines
Mechanical Engineering

Collaborator (co-Principal Investigator)

Jeremiah Neubert, Professor
University of North Dakota, Grand Forks, ND
College of Engineering and Mines
Mechanical Engineering

Project Science Collaborators

David Delene, Research Professor
Department of Atmospheric Sciences
University of North Dakota, Grand Forks, North Dakota

Jerome Schmidt
Marine Meteorology Division
Naval Research Laboratory
Monterey California

Proposed Title

Novel Characterization of the Liquid Layer Associated with Ice Crystal Aggregates Found in Cirrus Clouds

Topic Number: 3

Program Officer: Dr. Brett Pokines

Abstract

Hypersonic vehicles encounter ice crystal aggregates on their flight paths through tropical cirrus clouds. The ice aggregates cause significant damage to the nose tips of these vehicles and potentially alter flight trajectories. The damage potential is directly related to ice aggregate size and mass, yet there is a lack of reliable laboratory techniques and theoretical models to consistently predict whether these aggregates act as large particles or clusters of monomers that disintegrate. The thickness and interfacial tensions of the liquid layer on the surface of ice vary with cloud conditions and potentially dictate the strength of the aggregated ice crystals. The proposed research will develop (1) experimental and (2) theoretical techniques to characterize the interfaces of ice crystals.

(1) ***Tracking Size, Phase, and Morphology of Frozen Aqueous Droplets Using Optical Tweezers.*** A cold optical tweezers for trapping individual crystals and measuring the thickness of the liquid layer under controlled conditions.
(2) Thermodynamic Model of Liquid Layer Thickness as a Function of Interfacial Tension. Adsorption isotherm and statistical mechanical modeling at the ice-vapor interface to predict tensions and thickness of the liquid layer.

Recent airborne field campaigns using a Particle Habit Imaging and Polar Scattering (PHIPS) probe reported stereographic images of these aggregates in tropical cirrus clouds. The PHIPS probe was on board the North Dakota Citation Research Aircraft in 2019 as part of a Navy sponsored CapeEx19 field project. These observations do not inform the relevant cloud conditions that support ice aggregate formation. Results from this work will characterize the melted layer on the surface of ice, relate atmospheric observations of ice particles from the PHIPS probe to the impact on hypersonic vehicles, and improve model predictions of the effects of cloud conditions on ice aggregate strength.

Program Description Narrative

Ice aggregates form in tropical cirrus clouds and can damage hypersonic vehicles by deforming their nose tips and affecting their trajectory (***Figure 1***). The liquid layer present on the ice surface may play a key role in forming the ice bonds that hold the aggregates together, thereby dictating the size and mass.[1] The aggregates' size and mass are directly related to the damage potential of cirrus particles; therefore, to understand the impact of chain-like ice aggregates observed in tropical cirrus clouds, we need to understand how they interact with shock waves and interfere with hypersonic systems. Understanding these interactions requires knowing if the chains break apart and act like individual ice crystals or stay together across the shock wave and hence are particles with a large mass. These chain-like ice aggregates are composed of many ice crystal monomers, or single crystal types, jointed or aggregated together in various quasi-linear chains. Current literature focuses on observations of where these aggregates are found using airborne field campaigns;[2] however, there is a shortage of reliable laboratory techniques and theoretical models to consistently predict whether these aggregates act as one single massive particle or a cluster of monomers that break apart in high speed flow.

The primary scientific approach proposed here is to use single-particle laboratory techniques to probe the liquid layer on ice crystals and use a complementary thermodynamic model to measure and predict these layers' thicknesses. Ice aggregates in cirrus clouds were recently imaged using a PHIPS probe on board the North Dakota Citation Research Aircraft during the summer of 2019, as part of a Navy sponsored CapeEx19 field project. These images will be useful when compared to the experimental and theoretical results of this work.[3] High-resolution stereographic PHIPS images show an abundance of ice aggregate chains, yet how they are generated and their impacts on hypersonic systems are poorly understood. Characterization of the liquid surface layer is not only crucial for the formation of chain-like ice aggregates, but likely impacts the scattering of electrostatic radiation back to targets, such as in the case of C-band radars.[4]

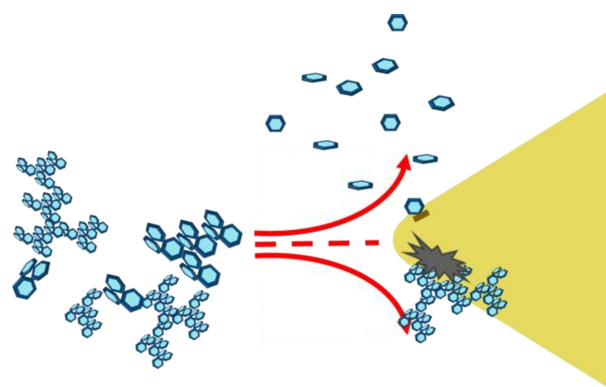


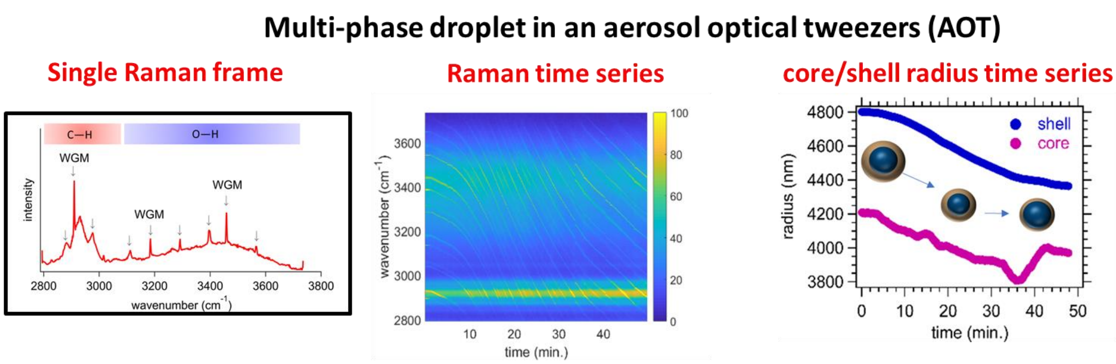
Figure 1: Schematic of ice aggregates impacting a missile in a cirrus cloud. As the vehicle travels through cirrus clouds, the chain-like ice aggregates may break apart or stick together and act as one large particle. These inertial particles damage the front of the vehicles and may alter their trajectory.

We will develop new techniques to characterize ice crystal monomers' interfaces and relate our results to the atmospheric observations of ice particles from the PHIPS probe, thereby examining these particles' impact on hypersonic vehicles. The ice-vapor interface is composed of a pre-melted liquid layer, arising from the disruption of the 3D network of hydrogen bonds at the interface.[5] Surface area, surface tension, and surface charge all affect how well the ice crystals stick together, and therefore potentially dictate the angle at which particles stick together and the complex morphology that results from aggregation.

Tracking Size, Phase, and Morphology of Frozen Aqueous Droplets Using Optical Tweezers

Optical tweezers use a tightly focused laser beam to trap dielectric particles and stably levitate them. A custom Cold Droplet Optical Tweezers (CDOT) will be built to trap individual supermicron droplets, freeze them, and measure the liquid layer's thickness under different conditions. The enclosed chamber and indefinite, stable levitation are ideal for controlling the environmental conditions (temperature, humidity) of the chamber. The experiments will likely reveal effects that have not been observed *in situ*, such as possible hysteresis during temperature and relative humidity changes.

Recent work by the Applicant presented a breakthrough method to probe thermodynamic droplet properties in real-time using an optical tweezers.[6] These tweezers can also probe multi-phase particles undergoing phase changes (***Figure 2***). Trapping is achieved through a balance between scattering forces and the gradient of light in the beam waist, resulting in a perfectly spherical shape of liquid droplets since there is no contact with a substrate. Raman spectra are simultaneously retrieved from the trapping laser, enabling the real-time obtainment of chemical information for compounds with Raman-active vibrational signatures. An important feature of the spectra is the spontaneous "Whispering Gallery Modes (WGMs)" superimposed on the unchanging Raman vibrational modes. These modes are surface resonances that shift sensitively when the droplet changes size and refractive index, and are driven by interactions with the surrounding gas phase, such as the condensation of water or other vapor species. Analysis of WGMs from Mie scattering theory enables highly accurate measurements of droplet radius.



- Representative Raman spectrum of an aqueous-organic acid droplet trapped in the AOT.
- Single frame taken from time series (middle)

- Time series of the droplet slowly shrinking in the AOT due to reducing RH.
- WGMs shift sensitively in response to droplet changes in size, refractive index, and shell thickness.

- The core-shell droplet shrinks slowly due to droplet equilibrating with the humid air as RH is lowered.

Figure 2: Recent experiments demonstrate the possibility of tracking the radius and shell thickness of a core-shell droplet in the aerosol optical tweezers (AOT). This technique will be adapted to measuring shell thickness of the liquid layer included on an ice crystal's surface and explore morphologies of the liquid layer.

Thermodynamic Model of Liquid Layer Thickness as a Function of Interfacial Tension

Theoretical development will apply a framework of adsorption isotherms and statistical mechanics at the interface from recent work by the Applicant[7] to predict tensions and thickness of the liquid layer and its interfaces. Interfacial tensions are dependent on thickness, as well as long-range and short-range molecular interactions. The model will advance our ability to predict the structure of ice aggregates and equip existing simulations with the ability to treat ice aggregates as a system of crystals held together in certain conformations.

In ***Figure 3***, the blue circles are water molecules, which act as adsorption sites in the ice layer. Liquid waters then adsorb in lattice layers of increasing disorder. Expressions for entropy and free energy will be derived using partition functions from statistical mechanics. A partition function can be written for each layer.

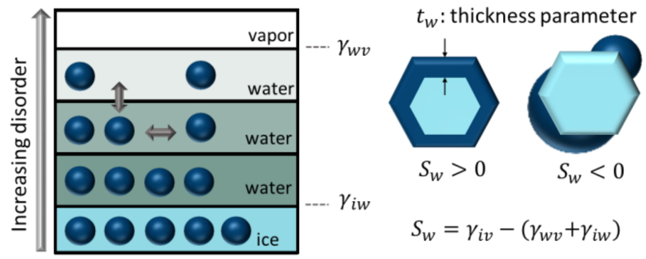


Figure 3: Multilayer adsorption isotherms (left) model thermodynamic properties such as interfacial tensions and spreading coefficients (S_w) and thickness of the liquid layer (right), following the PI's recent work on surface tensions of complex water droplets (Boyer et al. 2017).[7]

Liquid layer thickness is governed by ice crystal surface area and shape, as well as surrounding conditions (temperature, relative humidity).[1] Previous experimental and theoretical work has been combined to understand the ice-air interface from a molecular perspective using spectroscopy and ab initio simulations.[8] Here, we will use in-situ airborne observations to constrain the liquid layer thickness and laboratory experiments with optical tweezers to develop a new thermodynamic model using the multilayer adsorption isotherm in ***Figure 3***.

Summary

Large ice particles can deform the nose tips of hypersonic aircraft and re-entry vehicles, though little is known about their formation and even the cloud conditions that promote it. In order to predict damage to high-speed vehicles, we need to know what happens on impact, as well as understand the environment encountered during flight of vehicles that fly through cirrus clouds at high speeds. Existing models use a liquid equivalent diameter for ice particles. Particle size (diameter) and velocity are derived from shock tunnel work from the 1970's.[9] Mass, velocity, and impact angle change when the particle hits the solid surface. The proposed research will develop novel experimental and theoretical techniques to characterize the interfaces of ice crystal monomers, including optical tweezers and adsorption isotherm-based models. How these ice aggregates form is not well understood, though several explanations are offered, including how the interfaces are composed of nanoscale or quasi-liquid layers which enable partial coalescence, or surface tensions that bond the monomers together.

Bibliography

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(2) Lawson, R. P.; Woods, S.; Jensen, E.; Erfani, E.; Gurganus, C.; Gallagher, M.; Connolly, P.; Whiteway, J.; Baran, A. J.; May, P.; et al. A Review of Ice Particle Shapes in Cirrus Formed In Situ and in Anvils. *J. Geophys. Res. Atmos.* **2019**, *124* (17–18), 10049–10090. https://doi.org/10.1029/2018JD030122.

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(8) Tang, F.; Ohto, T.; Sun, S.; Jéré, J.; Rouxel, J. R.; Imoto, S.; Backus, E. H. G.; Mukamel, S.; Bonn, M.; Nagata, Y. Molecular Structure and Modeling of Water−Air and Ice−Air Interfaces Monitored by Sum-Frequency Generation. *Chem. Rev.* **2020**, *120*, 3633–3667. https://doi.org/10.1021/acs.chemrev.9b00512.

(9) The Space and Missile Systems Organization, T. A. F. O. of S. R. *ADVANCED REENTRY AEROMECHANICS*; 1978.

Collaboration Composition Statement

Applicant (Principal Investigator): Hallie Boyer Chelmo, Assistant Professor, Mechanical Engineering, College of Engineering and Mines, University of North Dakota, Grand Forks, ND

Collaborator (co-Principal Investigator): Jeremiah Neubert, Professor, Mechanical Engineering, College of Engineering and Mines, University of North Dakota, Grand Forks, ND

This collaboration is an important opportunity for introducing the Applicant to the DoD's basic research priorities and its supportive research ecosystem. The core collaboration consists of Principal Investigator **Hallie Boyer Chelmo** and co-Principal Investigator **Jeremiah Neubert**. The Applicant and Collaborator are both at the University of North Dakota, which is convenient for facilitating the collaboration. The Applicant will lead the project and will use more than 50% of the award for student support, laboratory supplies, conference travel, and publication costs. The Applicant/Principal Investigator is a full-time, tenure-track faculty member who has never served as PI on a prior DoD-funded award. The Collaborator Jeremiah Neubert is a full-time professor with tenure who currently serves as PI on HMMWV Battlefield Common Operating Picture Through Augmented Reality actively since March 20th, 2020.

Jeremiah Neubert will guide the Applicant in future DoD proposal submittals on a programmatic level. He will be supportive of the Applicant in navigating the DoD's grant application, management, and reporting procedures. Even though his expertise in augmented reality is outside the scope of the proposed research, he is well-suited as a mentor for the Applicant based on his recent and continual participation with the DoD's funding calls to spur basic research in DEPSCOR eligible states. He will require funding in the form of salary support for this work.

Project Science Collaborator: David Delene, Research Professor, University of North Dakota

The first project collaborator, David Delene, is also at UND. His input will benefit our greater goal of understanding how ice crystal aggregates are formed, such as those imaged in the CapeEx 19 campaign. He led the recent airborne field campaign on board the North Dakota Citation Research Aircraft during the summer of 2019 as part of a Navy sponsored CapeEx19 field project. These measurements will be essential for evaluating the Applicant's experimental and theoretical results. and how we can mitigate damage caused to aircraft that impact the aggregates. He will require funding in the form of salary and student support for analyzing the thousands of images taken during the campaign.

Project Science Collaborator: Jerome Schmidt, Meteorologist, Naval Research Laboratory,

The second project collaborator, Jerome Schmidt of the Naval Research Lab in California, will require minimal or no funding support. Jerome was the project PI for the CapeEx19 field campaign. His expertise regarding flow fields at the nose-tips of the hypersonic vehicles impacted by ice aggregates will be vital for this project. He will be invited to give an engineering seminar and may require travel support only. His funding needs are therefore minor, and the money will be spent in North Dakota, with an exception if we cover travel costs for him to visit.

Basic Research Statement

19th-century physicist Michael Faraday was the first observer of the thin melted surface layer that comprises the ice-vapor interface. For over a century, research has established that this semi-liquid layer exists at subcooled temperatures, spreading uniformly over ice surfaces; however, this surface layer is far more complicated than initially thought, as new research shows. Instead of a homogeneous, uniformly spread liquid layer as previously thought, there are multiple wetting states and morphology, as well as spatial heterogeneity in the surface layer. The thickness of this layer is a function of interfacial tension of the ice-liquid interface. New physical models are needed to connect the liquid layer's equilibrium states up to the decline of this layer upon contact with ice crystals or aqueous droplets.

This research meets DEPSCoR's aim of improving capabilities at IHE's in eligible states and will increase the chance of future DoD funding at the University of North Dakota. Understanding the surface layer through new experimental methods, such as levitating individual crystals with an optical tweezers and simultaneously retrieving their Raman vibrational spectroscopy, will demonstrate the multiple equilibrium morphological states of the liquid layer and connect it to the importance of ice crystal aggregate formation in tropical cirrus clouds. Further, new theoretical frameworks will be developed, using adsorption isotherms to model the thickness of the liquid layer as a function of interfacial tensions.

The novel experimental and theoretical techniques proposed here include the single-particle optical tweezers experimental platform and the adsorption isotherm-based models. The primary scientific approach proposed here is to develop a low temperature optical tweezers to probe the liquid layer on ice crystals and the complementary thermodynamic model to measure and predict these layers' thicknesses. The liquid aqueous layer at the ice-vapor interface arises from the disruption of the 3D network of hydrogen bonds at the interface. This fundamental knowledge will be applied to ice crystal aggregate formation in cirrus clouds.

Characterization of the liquid surface layer is not only crucial for the formation of chain-like ice aggregates, but likely impacts the scattering of electrostatic radiation back to targets, such as in the case of C-band radars. Surface area, surface tension, and surface charge all affect how well the ice crystals stick together, as well as potentially dictate the physical characteristics of the liquid surface layer and the angle in which particles stick together and the complex morphology that results from aggregation. The liquid layer present on the ice surface may play a key role in forming the ice bonds that hold the aggregates together, thereby dictating the size and mass of ice crystal aggregate structures.

Understanding the impaction of chain-like ice aggregates observed in tropical cirrus clouds on hypersonic vehicles requires knowing if the chains break apart and act like individual ice crystals or stay together across the shock wave and hence are particles with a large mass. These ice aggregates are composed of many ice crystal monomers, or single crystal types jointed or aggregated together in various quasi-linear chains. Current literature focuses on observations of where these aggregates are found using airborne field campaigns. A shortage of reliable laboratory techniques and theoretical models to consistently predict whether these aggregates act as one single massive particle or a cluster of monomers that break apart in high-speed flow needs to be addressed.

BIOGRAPHICAL SKETCH

NAME: Hallie Boyer Chelmo

POSITION TITLE: Assistant Professor

EDUCATION/TRAINING:

Institution and location	Degree	Completion Date	Field of study
Macalester College, St. Paul, MN	BA	05/2008	Physics
University of Minnesota, Minneapolis, MN	PhD	05/2017	Mechanical Engineering
Carnegie Mellon University, Pittsburgh, PA	Postdoc	07/2019	Chemistry

A. Personal Statement

I am an early stage investigator and my role in this project is the Principal Investigator. As an aerosol scientist, I bring my expertise on airborne particle dynamics and a molecular perspective on chemical and physical droplet properties. Microscopic ice crystals exhibit departing features from their bulk counterparts in unexpected and profound ways. These include the dominance of surface properties and processes over the bulk. They also access metastable states, such as supersaturated with respect to solute, accelerated chemical reactions, and varied internal structures. When composed of electrolyte and organic mixtures, core-shell and non-spherical morphologies emerge, drastically changing how ambient droplets evolve in the surrounding air.

This proposal builds on my unique background and expertise in aqueous surfaces (PhD; University of Minnesota)[1] and experimental measurements of thermodynamic properties of individual levitated microscale objects (Postdoc; Carnegie Mellon University). Because micro-droplets have a high surface-to-volume ratio, their surfaces are more important than the bulk, yet remain and important challenge to probe and isolate. I am highly motivated to measure and model complex droplet surfaces, which is what I achieved in the first projects[1]. My work in project [2] has prepared me for the experiments in this proposed work.

(*Author published as Boyer until 2020)

[1] R. E. H. Miles, M. W. J. Glerum, **H. C. Boyer***, J. S. Walker, C. S. Dutcher, B. R. Bzdek,*J. Phys. Chem. A* 2019, *121* (13), 3021–3029.
M. Riva, Y. Chen, Y. Zhang, Z. Lei, N. E. Olson, H.C. Boyer*, et al. *Environ. Sci. Technol* 2019, *53*, 8682–8694.
H. C. Boyer* and C. S. Dutcher, *J. Phys. Chem. A*, 2017, 121, 4733−4742.
H. C. Boyer*, B. R. Bzdek, J. P. Reid, and C. S. Dutcher, *J. Phys. Chem. A*, 2017, *121*,198–205.
H. C. Boyer* and C. S. Dutcher, *J. Phys. Chem. A* 2016, *120* (25), 4368–4375.
H. C. Boyer*, A. Wexler, and C. S. Dutcher, *J. Phys. Chem. Lett.* 2015, *6* (17), 3384–3389.
[2] **H. C. Boyer***, K. Gorkowski and R. C. Sullivan, *Anal. Chem*, 2019, 92, 1089–1096.
R. C. Sullivan, **H. C. Boyer Chelmo**, K. Gorkwoski, H. Beydoun, *Acc. Chem. Res.*, accepted.

B. Professional Experience and Honors

Other experiences and professional memberships

2020	Chair, Aerosol Physics, American Association for Aerosol Research (AAAR)
2020-present	Reviewer, Science Impacts, manuscripts
2020-present	Reviewer, Environmental Science Processes and Impacts, manuscripts
2019-present	Reviewer, Environmental Science and Technology, manuscripts
2017-present	Reviewer, National Oceanic and Atmospheric Administration, proposals
2017-present	Reviewer, Langmuir, manuscripts
2017-present	Reviewer, Physical Chemistry Chemical Physics, manuscripts
2015-present	Member, American Association for Aerosol Research (AAAR)
2016-2017	Member, American Meteorological Society (AMS)

Fellowships & Awards

2019-2021	UND College of Engineering and Mines Dean's Research Professor Award
2017	Invitee to the 14th Atmospheric Chemistry Colloquium for Emerging Senior Scientists (ACCESS XIV)
2017	Best talk award at ACCESS XIV
2016	American Association for Aerosol Research (AAAR) Student Travel Award
2016	Graduate Women in Science (GWIS) Student Travel Award
2014-2017	National Science Foundation Graduate Research Fellowship
2012-2013	UMN Diversity of Views and Experiences (DOVE) Graduate Fellowship
2012	UMN Mechanical Engineering Graduate Fellowship

Non-academic work experience

2010-2012	*Peace Corps Volunteer, Ghana, West Africa.* Taught high school physics.
2008-2010	*Commodities Options Clerk, Chicago Board of Trade, MOE Options, Inc.*

C. Contributions to Science

Thermodynamic modeling of aqueous aerosol surfaces.

Formulated powerful analytical expressions for surface tensions of mixture-containing aqueous solutions, towards improvements in treatment of aerosol surfaces in global models. Extended the model to numerous complex mixtures of varying influences on the surface, especially solutions containing atmospherically relevant compounds.

Spectroscopic measurements of aerosol particle properties in optical tweezers.

Developed a novel *in situ* method for direct, real-time particle thermodynamic property measurements using a contactless, single-particle trapping technique coupled with Raman spectroscopy. Designed a new optical tweezers system with low temperature capabilities to characterize chemical and physical changes in the particle in response to changes in temperature

Biographical sketch: Jeremiah Neubert, Ph.D., E.I.T.

a. Professional Preparation:

Saint Cloud State University	B.S. in Manufacturing Engineering,	1999
University of Wisconsin-Madison	M.S. in Mechanical Engineering	2001
University of Wisconsin-Madison	M.S. in Computer Science	2003
University of Wisconsin-Madison	Ph.D. in Mechanical Engineering	2005
Cambridge University	Postdoctoral Researcher	2005-2007

b. Appointments:

Full Professor of Mech. Engr.	University of North Dakota	2020-present
Associate Professor of Mech. Engr.	University of North Dakota	2012-2020
Assistant Professor of Mech. Engr.	University of North Dakota	2007-2012
Postdoctoral Researcher	Cambridge University	2005-2007
Research Assistant	University of Wisconsin-Madison	1999-2005
NSF GK12 Fellow	University of Wisconsin-Madison	2000-2001

c. Publications most closely related to the proposed project:

I. Rizwan, A. Haque, **J. Neubert**, "Deep learning approaches to biomedical image segmentation", *Informatics in Medicine Unlocked*, 2020

T. Elderini, N. Kaabouch, **J. Neubert**, "Space Occupancy Representation Based on A Bayesian Model for Unmanned Aerial Vehicles." *J Intell Robot Syst*, 97, 399–410 2020.

A. Haque, A. Elsaharti, T. Elderini, M. Elsaharty, **J. Neubert**, "UAV Autonomous Localization Using Macro-Features Matching with a CAD Model," *Sensors*, MDPI, 2020, 20, 743.

T. Elderini, N. Kaabouch, and **J. Neubert**, "Space Occupancy Representation Based on a Baysian Model for Unmanned Aerial Vehicles," *Journal of Intelligent & Robotic Systems*, Springer, 2019. (Impact Factor 2.020)

T. Elderini, N. Kaabouch and **J. Neubert**, "3-D Graphical Representation for Indoor Objects Based on A Bayesian Model," *NAECON 2018 - IEEE National Aerospace and Electronics Conference*, Dayton, OH, 2018, pp. 425-430.

S. S. Roy, A. U. Haque and **J. Neubert**, "Automatic diagnosis of melanoma from dermoscopic image using real-time object detection," *2018 52nd Annual Conference on Information Sciences and Systems (CISS)*, Princeton, NJ, 2018, pp. 1-5.

N. Allen, A. Tabassum, W. Semke and **J. Neubert**, "Repeatability of edge detectors in various environmental conditions," *International Conference on Unmanned Aircraft Systems (ICUAS)*, 2017, pp. 912-919.

d. Previous DOD experience and involvement:

"HMMWV Augmented Reality", Contract Number: W56HZV-19-001, P.I. **Jeremiah Neubert**, Co-P.I. Ron Marsh, and Richard Ferraro, $1.5 million Funded 3/30/2020.

"Unmanned Aerial System Remote Sense and Avoid System and Airborne Payload Analysis and Investigation", United States Air Force UAV Battle Lab, P.I. Mark Askelson, $9.656 million, Funded 9/2007.