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)

Joseph Jewell, Assistant Professor

Purdue University, West Lafayette, IN

College of Engineering

School of Aeronautics and Astronautics

Proposed Title

Investigating the Formation and Impacts of Ice Crystal Aggregates on Hypersonic Vehicles

Topic Number: 14

Program Officer: Dr. Eric Marineau

Abstract

Hypersonic vehicles inevitably encounter ice crystal aggregates on their flight paths through tropical and mid-latitude cirrus clouds, which cover approximately one third of Earth's surface. The ice aggregates can significantly damage the nose tips of these vehicles. The damage potential is directly related to ice aggregate size and mass, yet there is a lack of reliable laboratory techniques to consistently test whether ice aggregates impact as a single large particle or disintegrate into clusters of monomers when traversing a vehicle's shockwave. The broad research goal is to create a new method to assess ice aggregate impacts to hypersonic vehicles. Towards understanding the bonding strength of monomers that make up ice crystal aggregates and how they pass through hypersonic boundary layers, the proposed research will develop complementary laboratory experiments:

(1) Understanding monomer joining process of ice crystal aggregation using a cold, dual electrodynamic balance at UND to stick two individual crystals together and pinpoint their joining ambient conditions and the dependence of crystal structure.

(2) Observing ice crystal monomers and aggregates in the process of transition to turbulence using Purdue's shock tube facility to test how ice crystals interact with a shockwave.

The recently developed Particle Habit Imaging and Polar Scattering (PHIPS) probe was deployed on the North Dakota Citation Research Aircraft in 2019 as part of a Navy sponsored CapeEx19 field project. The PHIPS probe obtained stereographic images of ice crystal aggregates in tropical cirrus clouds that clearly show monomers joints, which are very small, compared to the monomer's size. Results will relate atmospheric observations of ice crystal aggregates to the impact on hypersonic vehicles and improve understanding of the aggregation process.

Objective: We will combine laboratory studies and hypersonic flow physics to test whether observed ice crystal aggregates break apart or stay together after encountering a shock wave.

Program Description Narrative

Hypersonic vehicles encounter atmospheric constituents upon re-entry, including cirrus clouds extending across approximately 25-33% of the Earth's troposphere, and ranging from 4-20 km in altitude.[1] Cirrus clouds grow from aerosol seed particles (ice nuclei), which itself is a poorly constrained process, but once crystals form they can aggregate (join together) to produce larger ice crystal aggregates, sometimes in long chains of individual monomers.[2] These joined monomers (clusters of individual crystals) shown in Figure 1 have significant effects on hypersonic vehicles since they alter flight path trajectories and can damage nose cones.[3–6] However, information regarding these aggregates is severely lacking, including their formation processes, evolution in the atmosphere, and behavior when traversing a shock wave. Additionally, the relevant timescales of each of these processes is unknown, which hinder model development. This broad lack of knowledge inhibits accurate damage potential assessment and flight path models of hypersonic vehicles at high altitudes, which will become increasingly important for space exploration, mitigating missile and plane damage, and creating faster transportation.

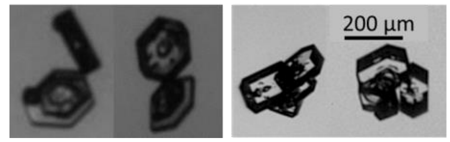

Figure 1: Representative in situ stereo images of small ice crystal aggregates present in most cirrus clouds, obtained with a Particle Habit Imaging and Polar Scattering (PHIPS) probe onboard the North Dakota Citation Research Aircraft during the summer of 2019, which was part of a Navy sponsored CapeEx19 field project.[2]

This problem, as illustrated in Figure 2, has been known about since at least 1977, when it was reported by Lin and Thyson[7], yet little to no progress has been made since recently. There is current focus on dust and aerosol particles;[8,9] however, ice crystals have not yet been investigated and potentially have a larger effect because of the sufficiently larger mass, especially of the chain-like aggregates (Figure 2 inset image) that do not disintegrate. Although we emphasize that ice crystals commonly form a hexagonal shape (Figure 1, left), there are many frozen spherical droplets.[10] The shape of the crystal depends primarily on its growth process. The hexagon shape results from the environmental condition during growth, which controls the growth speed in different dimensions; however, water molecules can homogenously freeze without an ice nuclei at low temperatures (<40 °C)[10], which forms a sphere of ice.

We will harness the detailed knowledge provided by the CapeEx19 field campaign, augment it with ice particle levitation experiments under atmospherically accurate conditions (***Objective #1***), and introduce ice aggregate mimics into Purdue's shock tube facility (years 1-2) and BAM6QT (Boeing/AFOSR Mach-6 Quiet Tunnel) in year 3 (***Objective #2***). From communication with computational modelers Dr. Christoph Brehm (UMD) and Dr. Tom Swartzentruber (UMN), these experiments will support particle impingement and flow simulations, which require geometrical and thermodynamic constraints. For example, heat transfer, diffusion, and phase change all affect the time needed for

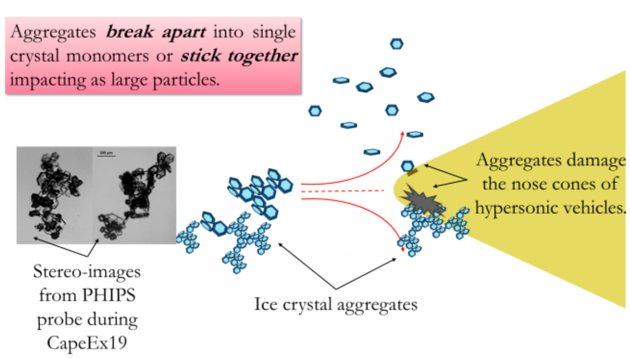

Figure 2: Illustration of ice aggregates impacting a vehicle nose cone. The ice aggregates' damage potential to hypersonic vehicles can be significantly different if they break apart into individual ice crystals or stay together across the harsh thermochemical changes within a shock wave.

ice to impinge, but there is insufficient input data for accurate simulations. Our experiments can provide precisely what they need, and we have been offered a letter of support for a full proposal from Dr. Brehm. This research will not overlap other funded projects, such as the ONR MURI on Multiphase Hypersonics, as they do not include ice crystal aggregates and their bonding strength.

<u>Objective #1:</u> *Understanding the monomer joining process using a dual electrodynamic balance.*

Microparticle levitation techniques are stable, high throughput, and non-destructive; they preserve the crystal interface's integrity and offer precision and control that is difficult to achieve at the microscale.[11,12] The Applicant is an expert in microparticle levitation, especially using optical tweezers.[13] Our newly available electrodynamic method traps and isolates liquid droplets, and can be readily adapted to ice particle structures, using a dual balance quadrupole electrodynamic balance (DBQ-EDB, Figure 3), which is currently used in just one other laboratory for atmospheric chemistry studies. Unlike the tweezers, the DBQ-EDB can load irregularly shaped objects like single ice crystals or aggregates to directly interrogate crystals of the right sizes, which is larger than a liquid-equivalent diameters of 50 μm.[14]

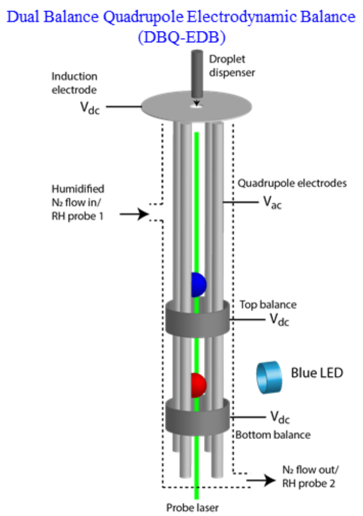

Figure 3: Schematic of the experimental apparatus for trapping ice crystals (blue and red spheres). A freezer will replace the droplet dispenser to introduce ice crystals. The induction electrode charges the crystal to be trapped. The balancing region has a direct current and traps the crystal with electrostatic forces.[22,23] The second, oppositely charged balancing region is 5 cm below the top. One trapped crystal receives a higher net charge, so that when the two trapped crystals are brought together, they remain with a net charge and thus stably trapped. Adapted from Ref. 22.

We will grow ice crystals in a freezer chamber and directly pull them into the trap (replacing the droplet dispenser on top in Figure 3). The chest freezer is temperature controlled and can be set to any crystal growth temperature. Humidity is added to the freezer using a dew point generator and if finer humidity control is necessary, a simple wet cloth on a light bulb with a dimmer switch can be used. The freezer contains a side hole, which allows an access tube to pull the crystals into the DBQ-EDB instrument. The DBQ-EDB will be chilled to maintain the ice crystals. It is also possible to collide (join) the two trapped ice crystals in the DBQ-EDB and image the collision with Raman spectroscopy or bright-field and far-field microscopy. We hypothesize that ice-vapor interfaces, composed of a disordered, pre-melted liquid layer,[15–19] dictate how well the ice crystals stick together, which is a crucial factor in aggregate formation and break-up. The thickness and phase state of the liquid layer depends on temperature and ice crystal habit, or shape. At this point, it cannot be estimated which conditions are most supportive of this process, since cirrus clouds form at a wide range of temperature (-30 to -80 °C).

<u>Objective #2:</u> *Observing the ice crystal monomers and aggregates in the process of transition to turbulence using Purdue's experimental aerodynamics facilities.*

The shock tube at Purdue will enable studies of ice crystals and ice aggregates in shock waves, followed by BAM6QT tests after developing new techniques to couple individual ice crystals and high-speed flows. There are numerous advantages in using the shock tube for initial tests. It is an uncomplicated and inexpensive setup, with easy to control flows and rapid, repeatable generation of one-dimensional shock waves. Because the flow conditions themselves are simple to interpret and model, the flow will be the experimental control, allowing us to observe shape (drag) and phase (ice or water). Dozens of tests will be done in one day, if need be. Shock tubes have enabled improved physical understanding in other areas of hypersonic studies, such as improving predictive power of transition location, disturbance environment, and freestream noise. We expect similarly useful results for the reaction of ice particles to the shock wave.

There are many ice crystal shapes and sizes displayed in the PHIPS images. We will identify 1-3 specific shock conditions, i.e. pressure ratio of the driven and driver fluid sections and repeat them for the different ice crystal habits (shapes) and aggregates (sizes). Modifications can be readily made to the shock tube (Figure 4) to accommodate adding ice particulates and imaging them in the test regions. The shock wave is in the test region, between the compressed fluid and the upstream driven fluid, will show comparison of changes in the ice particles before and after the shock. There is some variety from run-to-run due, but Berridge[20] reports a standard deviation of burst pressure within 10% for all diaphragm materials. These experiments are a logical first step in our hypersonic flow experiments. The next step is to utilize Purdue's BAM6QT, which will reduce the freestream noise level and accurately replicate the disturbances of hypersonic flight.[21]

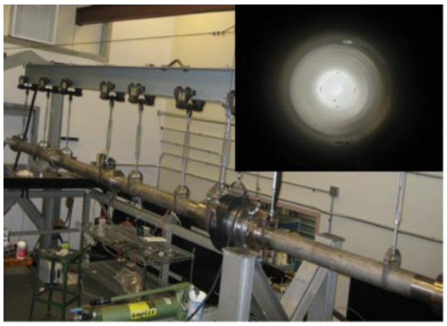

Figure 4: Purdue's 3" shock tube (left picture; Fig 3.1 from ref 21) and inside (inset left picture; Fig. 3.2 from ref 21). Inside is a diaphragm section dividing the driver and driven fluids, with respective section lengths 4' and 12'. Minor modifications such as adding high-speed cameras and micro-positioners to control timing the shock wave formation and ice crystal interaction are possible.

We will first test spherical particles with a similar mass to frozen droplets, such as spherical plastic or glass beads above the liquid equivalent 50 µm diameter, to mimic homogenous freezing ice particles. These simpler systems will make it easier to model drag in flow simulations and impact on hypersonic vehicles. Then we will create 3D printed ice crystals for testing. Three-dimensional printing technology will be used to produce surrogate ice crystal aggregates with densities comparable to ice and shapes like the PHIPS observations (Figure 1 and inset in Figure 2). A student will create computer drawings of the PHIPS aggregates and the design sent to a 3D printing company for production. A micro-positioner will hold the surrogate aggregate for release into the shock tube flow for accurate drag behavior; however, some topics cannot be studied this way: how well the aggregate hold together in the transition to turbulence; phase transitions such as sublimation, melting, and evaporation; and changes in size and morphology due to phase transitions. These phase studies can be completed during laboratory work at UND, using controlled microparticle levitation and steering techniques. From the shock tube tests, we will refine the procedure of introducing ice crystals in the flow, especially controlling the timing and placement of the crystals when they experience the shocks. In the longer term, we will apply these results to advance capabilities of controlling and observing ice crystal aggregates within the BAM6QT (year 3 of this project) and HYPULSE (continuing research) at Purdue.

Rough yearly costs of the project. The total comes to $200,000/yr. with at least 50% to UND. Materials ($37,000). Publications (2/yr., $5,000). Travel ($33,000): 1 conference/yr. for all personnel; D.C. program meetings; UND to Purdue 1/yr. for Dr. Chelmo and student for 2-4 weeks. Personnel ($125,000): 1 full-time UND student; 1 half-time Purdue student; faculty salary.

Bibliography

(1) Cziczo, D. J.; Froyd, K. D. Sampling the Composition of Cirrus Ice Residuals. *Atmos. Res.* **2014**, *142*, 15–31. https://doi.org/10.1016/j.atmosres.2013.06.012.

(2) Schmidt, J. M.; Flatau, P. J.; Harasti, P. R.; Yates, R. D.; Delene, D. J.; Gapp, N. J.; Kohri, W. J.; Vetter, J. R.; Nachamkin, J. E.; Parent, M. G.; et al. Radar Detection of Individual Raindrops. *Bull. Am. Meteorol. Soc.* **2019**, *100* (12), 2433–2450. https://doi.org/10.1175/BAMS-D-18-0130.1.

(3) Mehta, N. A.; Levin, D. A. Molecular-Dynamics-Derived Gas-Surface Models for Use in Direct-Simulation Monte Carlo. *J. Thermo* **2016**, *31* (4). https://doi.org/10.2514/1.T4934.

(4) Chen, S. Y.; Boyd, I. D. Boundary-Layer Thermochemical Analysis During Passive and Active Oxidation of Silicon Carbide. *J. Thermophys. Heat Transf.* **2019**, *34* (3). https://doi.org/10.2514/1.T5864.

(5) Mehta, N. A.; Levin, D. A. Multiscale Modeling of Damaged Surface Topology in a Hypersonic Boundary. *J. Chem. Phys* **2019**, *151*, 124710. https://doi.org/10.1063/1.5117834.

(6) Mackey, L. E.; Boyd, I. D.; Leger, T.; Gosse, R.; Vogiatzis, K. Effect of Hypersonic Flow Physics Modeling on Electro-Optical Sensor Assessment. In *AIAA Aviation*; 2017. https://doi.org/10.2514/6.2017-3836.

(7) Lin, T. C.; Thyson, N. A. Ice-Crystal/Shock-Layer Interaction in Hypersonic Flight. *AIAA J.* **1977**, *15* (10), 1511–1514. https://doi.org/10.2514/3.7446.

(8) Browne, O. M. F.; Al Hasnine, S. M. A.; Brehm, C. Numerical Method for Particulate-Induced High-Speed Boundary-Layer Transition Simulations. *AIAA J.* **2021**, *59* (4), 1196–1213. https://doi.org/10.2514/1.J059544.

(9) Al Hasnine, S. M. A.; Russo, V.; Browne ‡, O. M. F.; Tumin, A.; Brehm, C. Disturbance Flow Field Analysis of Particulate Interaction with High Speed Boundary Layers. In *AIAA 2020-3046*.

(10) Stith, J. L.; Avallone, L. M.; Bansemer, A.; Basarab, B.; Dorsi, S. W.; Fuchs, B.; Lawson, R. P.; Rogers, D. C.; Rutledge, S.; Toohey, D. W. Ice Particles in the Upper Anvil Regions of Midlatitude Continental Thunderstorms: The Case for Frozen-Drop Aggregates. *Atmos. Chem. Phys.* **2014**, *14* (4), 1973–1985. https://doi.org/10.5194/acp-14-1973-2014.

(11) Gao, D.; Ding, W.; Nieto-Vesperinas, M.; Ding, X.; Rahman, M.; Zhang, T.; Lim, C.; Qiu, C.-W. Optical Manipulation from the Microscale to the Nanoscale: Fundamentals, Advances and Prospects. *Light Sci. Appl.* **2017**, *6*. https://doi.org/10.1038/lsa.2017.39.

(12) Metcalf, A. R.; Narayan, S.; Dutcher, C. S. A Review of Microfluidic Concepts and Applications for Atmospheric Aerosol Science. *Aerosol Sci. Technol.* **2018**, *52* (3), 310–329. https://doi.org/10.1080/02786826.2017.1408952.

(13) Boyer, H. C.; Gorkowski, K.; Sullivan, R. C. In Situ PH Measurements of Individual Levitated Microdroplets Using Aerosol Optical Tweezers. *Anal. Chem* **2020**, *92*, 1089–

1096. https://doi.org/10.1021/acs.analchem.9b04152.

(14) 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.

(15) Louden, P. B.; Daniel Gezelter, J. Why Is Ice Slippery? Simulations of Shear Viscosity of the Quasi- Liquid Layer on Ice. *J. Phys. Chem. Lett* **2018**, *9*, 46. https://doi.org/10.1021/acs.jpclett.8b01339.

(16) Llombart, P.; Noya, E. G.; Sibley, D. N.; Archer, A. J.; Macdowell, L. G. Rounded Layering Transitions on the Surface of Ice. *Phys. Rev. Lett.* **2020**, *124*. https://doi.org/10.1103/PhysRevLett.124.065702.

(17) Sibley, D. N.; Llombart, P.; Noya, E. G.; Archer, A. J.; Macdowell, L. G. How Ice Grows from Premelting Films and Water Droplets. *Nat. Commun.* **2021**. https://doi.org/10.1038/s41467-020-20318-6.

(18) Slater, B.; Michaelides, A. Surface Premelting of Water Ice. *Nat. Rev. Chem.* **2019**, *3* (3), 172–188. https://doi.org/10.1038/s41570-019-0080-8.

(19) Murata, K.-I.; Asakawa, H.; Nagashima, K.; Furukawa, Y.; Sazaki, G. Thermodynamic Origin of Surface Melting on Ice Crystals. *Proc Natl Acad Sci U S A* **2016**, *113* (44), 6741–6748. https://doi.org/10.2307/26472328.

(20) Berridge, B. D. C.; Schneider, S. P.; Collicott, S. H.; Rufer, S. J. Thesis / Dissertation Acceptance This Is to Certify That the Thesis / Dissertation Prepared For the Degree of Doctor of Philosophy Is Approved by the Final Examining Committee : Approved by Major Professor (s): Steven P . Schneider Approved by : Wayne C, 2015.

(21) Casper, K. M.; Beresh, S. J.; Henfling, J. F.; Spillers, R. W.; Pruett, B.; Schneider, S. P. Hypersonic Wind-Tunnel Measurements of Boundary-Layer Pressure Fluctuations. *39th AIAA Fluid Dyn. Conf.* **2009**, 1–20. https://doi.org/10.2514/6.2009-4054.

(22) Richards, D. S.; Trobaugh, K. L.; Hajek-Herrera, J.; Davis, R. D. Dual-Balance Electrodynamic Trap as a Microanalytical Tool for Identifying Gel Transitions and Viscous Properties of Levitated Aerosol Particles. *Anal. Chem.* **2020**, *92* (4), 3086–3094. https://doi.org/10.1021/acs.analchem.9b04487.

(23) Richards, D. S.; Trobaugh, K. L.; Hajek-Herrera, J.; Price, C. L.; Sheldon, C. S.; Davies, J. F.; Davis, R. D. *Ion-Molecule Interactions Enable Unexpected Phase Transitions in Organic-Inorganic Aerosol*; 2020; Vol. 6.

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): Joseph Jewell, Assistant Professor, School of Aeronautics and Astronautics, College of Engineering, Purdue University, West Lafayette, IN

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 the Applicant/Principal Investigator **Hallie Boyer Chelmo** and the Collaborator/co-Principal Investigator **Joseph Jewell**. Dr. Chelmo will supervise a graduate student performing ice crystal monomer joining experiments at the University of North Dakota and the Collaborator will allow time for one of his students to perform the shock tube experiments at Purdue University. They will meet once every two weeks virtually and once per year in person by Dr. Chelmo and her student traveling to Purdue for an extended period, i.e. two to four weeks. The team will also attend one conference per year. Funds will be requested to support this research, including the faculty salaries, one graduate student at UND, travel for Dr. Chelmo and her student to Purdue at least once per year, all other travel, materials, and publication costs. Dr. Chelmo will therefore lead the project and will use more than 50% of the award for these costs. Dr. Chelmo is a full-time, tenure-track faculty member who has never served as PI or co-PI on a prior DoD-funded award.

Dr. Jewell is a full-time, tenure-track faculty member who currently serves as PI on an ONR sponsored project. He is an expert in hypersonic aerothermodynamics, with special focus on spacecraft re-entry aerothermodynamics and hypersonic wind tunnel design and operation. He has 16 years of experience in experimental hypersonics (Oxford, CalTech, AFRL, and Purdue), flight test analysis, facility modeling and simulation. He is currently an assistant professor at Purdue University and manages the Boeing/AFOSR Mach 6 Quiet Tunnel (BAMQT6), a Ludweig tube with significant noise reduction compared to conventional wind tunnels. He is an AIAA Associate Fellow and a Rhodes Scholar. Dr. Jewell is well-suited as a mentor for Dr. Chelmo based on his complementary expertise in high-speed flows and his long history of success with the DoD's funding calls to spur basic research in DEPSCOR eligible states. He will support Dr. Chelmo with navigating the DoD's grant application, management, and reporting procedures. He will require funding in the form of salary support for this work.

Project Science Collaborators: David Delene, Research Professor, Atmospheric Sciences, John D. Odegard School of Aerospace Sciences, University of North Dakota, Grand Forks, ND.

Jerome Schmidt, Meteorologist, Naval Research Laboratory, Monterey, CA.

The first project collaborator, **Dr. David Delene**, is also at UND and 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, which captured the ice crystal aggregate data. He will require funding in the form of salary for analyzing the thousands of images taken during the campaign. The second project collaborator, **Jerome Schmidt** was the project PI for the CapeEx19 field campaign. His funding needs are minor; therefore the money will be spent in North Dakota, with an exception if we cover travel costs for him to visit.

Basic Research Statement

It was recently recognized using a newly developed Particle Habit Imaging and Polar Scattering (PHIPS) probe that ice crystal monomers and larger ice aggregates are present in cirrus clouds, yet they are an under-explored category of atmospheric particles. 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. There is high potential for future research in developing new methods of testing the mechanisms of their joining together (aggregation) and how these mechanisms react in a shock wave, which will improve assessment on hypersonic vehicle impacts and flight path calculation models. Understanding these interactions (the impact of chain-like ice aggregates observed in tropical cirrus clouds) 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. Current literature focuses on observations of where these aggregates are found using airborne field campaigns. The proposed research addresses a shortage of reliable laboratory experiments to consistently predict whether these aggregates act as one single massive particle or a cluster of monomers that break apart in high-speed flow. With improved knowledge and understanding of the ice aggregates, there will be an improvement to the modeling of the flight path through cirrus clouds, which would allow more accurate targeting and assessment of the potential target area. Increasing fundamental knowledge of ice crystals' formation and how well they withstand high speed flows meets the DoD's criteria for Basic Research.

We hypothesize that the quasi-liquid layer (a disordered, pre-melted liquid layer) dictates how well the ice crystals stick together, necessitating a contactless technique for probing the individual behavior of ice crystals. The electrodynamic balance approach proposed here levitates the ice crystals while simultaneously retrieving their properties using Raman vibrational spectroscopy and brightfield and far-field imaging and enables the joining together under controlled conditions, while preserving the integrity of the ice-vapor interface. Fundamentally, 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 and ice crystal aggregate formation in tropical cirrus clouds. Understanding the individual crystals with this method and developing new approaches to integrating this method with shock tube and wind tunnel tests will elucidate whether the observed ice crystals and their aggregates impact as a large particle or break apart. Characterization of their sticking potential under controlled atmospherically relevant conditions 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. The precision and control of the single particle technique, combined with the simulation of high-speed flows in the shock tube and wind tunnel, presents a unique approach to improving predictions of where the ice aggregates are formed and the changes they may experience when traversing hypersonic boundary layers. It is imperative to run these tests to better understand the timing of formation and impacts, which is completely unknown. The information learned from this research will set the stage for future basic research in numerous related topics, including simulations of ice crystals and agglomerates passing through the shock layer, which require knowledge of the makeup and structural properties of the ice particles themselves; wind tunnel experiments using Purdue's HYPULSE; and large-scale model improvements for flight path calculations.