Experimental Design and Construction of the First Rotor Induced Collision Cell (RICC) for Studying High Velocity Molecular Impacts

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Experimental Design and Construction of the First Rotor Induced Collision Cell (RICC) for

Studying High Velocity Molecular Impacts

Abraham Lehi De la Cruz Hernandez

A dissertation submitted to the faculty of
Brigham Young University
in partial fulfillment of the requirements for the degree of

Doctor of Philosophy

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ABSTRACT

Experimental Design and Construction of the First Rotor Induced Collision Cell (RICC) for Studying High Velocity Molecular Impacts

Abraham Lehi De la Cruz Hernandez
Department of Chemistry and Biochemistry, BYU
Doctor of Philosophy

The identification and characterization of molecular biomarkers using mass spectrometry on an orbiting or fly-by spacecraft is one of the preferred analytical techniques in the search for life beyond the Earth. However, analysis is complicated by unwanted molecular dissociation occurring when sampled native molecules impact the instrument at high velocity. The mechanisms of chemical changes produced in high velocity impacts have been studied experimentally in some cases; however, there are significant experimental limitations to these techniques. Here I present the design, construction, and testing of a new experimental technique to produce high velocity molecular and microparticle collisions under a controlled lab setting using a high-speed spinning rotor. Chapter 1 of this manuscript gives a scientific review of the astrobiological importance of this project for future and current space missions as well as describing previous techniques used to produced hypervelocity impacts and their limitations. Chapter 2 presents the design, construction, calibration, and preliminary experiments of the new technique involving the high-speed rotor. Chapter 3 describes the fabrication of a molecular beam system from the ground up to be coupled with the high-speed rotor. Chapter 4, describes future project directions and presents future experiments using the rotor as a stand-alone instrument. Lastly, the appendix contains the standard operation procedures and design notes regarding the operation of these two instruments.

Keywords: astrobiology, space sampling, high velocity impacts, high speed rotor, molecular beam
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CHAPTER 1: INTRODUCTION

1.1 Justification: Astrobiological Importance

The search for life outside of our planet is one of the most monumental endeavors mankind has ever undertaken. The existence of habitable places in our solar system has led to an increased interest in the search for chemical signatures of life using analytical instruments on fly-by, orbiting, and even landed spacecraft. A preferred technique to make these measurements is via mass spectrometry due to its high sensitivity, selectivity, and fast analysis.\textsuperscript{1} However, the interpretation of fly-by and from-orbit measurements may become obscured by undesired chemical fragmentation caused by high velocity collisions as the molecules enter the instrument for analysis. This factor raises important questions for space exploration missions. Can we determine if previously collected data is compromised by high velocity impact fragmentation? Are there improvements to be made in current and future instrumentation that can reduce these undesired fragmentation effects? What is the appropriate encounter velocity at which molecules can be effectively sampled and accurately detected? Efforts have been made to understand high velocity impact fragmentation patterns in controlled lab settings to resolve the potential presence of biomarkers for previously reported data and for future exploration missions. The dissertation project presented in this manuscript involved the development of a high-speed rotor capable of producing high velocity impacts under high vacuum conditions. The introduction of this manuscript starts with a synopsis of the astrobiological environments and evidence of possible biomarkers in the solar system. Then, an overview and limitations of the fly-by sampling systems used in spacecraft mass spectrometers is presented. Additionally, a review of the limitations of previous instrumental designs built to study hypervelocity collisions is discussed. Finally, a brief
description of the theoretical aspects of the proposed instrument, a rotor coupled with a molecular beam, is explained.

1.2 Importance of Space Sampling in the search for Signatures of Life

A place in the solar system where life could potentially exist, and an approach to confirm, measure, and study such life using analytical instruments is one of the key motivations for this project. From our understanding of the universal laws that govern life on earth, life requires four essential components: one, chemical energy to drive metabolic reactions; two, a liquid solvent to mediate these reactions; three, essential elements to produce molecules and to catalyze organic reactions needed for life, and four time.\textsuperscript{2-5} Thus, the presence of water, chemical energy, and essential elements create habitable conditions to support life.\textsuperscript{6-8} Ongoing interpretations from telescope observations and fly-by probe measurements in the Jovian and Saturnian systems have led scientists to suggest the presence of habitable moons around both Jupiter and Saturn.

Recent findings from the Galileo\textsuperscript{9-13} and Cassini\textsuperscript{8,14-24} missions identified the presence of underground liquid oceans, surface plume activity (geysers), and potential for subsurface chemical reactions at Europa (Jupiter’s icy moon) and Enceladus\textsuperscript{25} (Saturn’s 6th largest moon), which leads scientists to consider the possibility of living microorganisms comparable to Earth’s extremophile bacteria (bacteria that survive under harsh conditions) found in hydrothermal vents in the depths of Earth’s seas.\textsuperscript{5,26} The ecosystem surrounding the formation and hydrothermal activity of the plumes sampled by Galileo and Cassini present a promising environment where life may dwell.\textsuperscript{27,28} For instance, Cassini detected the presence of a subsurface salty water sea enriched with minerals similar to earth’s oceans that is in contact with thermal energy emanating from Enceladus’ rocky core.\textsuperscript{29-32} Additionally, measurements from Cassini caused scientists to
speculate that the sampled plume content, high in molecular hydrogen produced by potential oxidative processes, is proof of hydrothermal and chemical activity at Enceladus’ ocean core boundary.\textsuperscript{33-40} All these factors provide evidence for scientists to target these moons in their focus to sample biomarkers that would support the presence of extraterrestrial life.

Nevertheless, even if a habitable place presents all the conditions needed to support life, it can still contain no life. Based on earth’s biological processes, life has always left behind chemical signatures as patterns of compounds such as amino acid distributions, fatty acid chain lengths, isotopic patterns, and enantiomeric excess, not all of which may be detectable using spacecraft apparatus. In addition, biomarkers found in a celestial body may not be enough evidence to prove the presence of extraterrestrial life because they may be the product of abiotic chemical reactions occurring in space.\textsuperscript{41} For instance, amino acids, a biomarker of earth’s living organisms, have been found in interstellar abiotic environments,\textsuperscript{42} places like asteroids and comets. The enantiomeric predisposition in asteroids for the D-enantiomer versus the L-enantiomer—which is used exclusively by living organisms on earth—suggests that the excess found in asteroids and meteorites doesn’t come from living biological processes, but from other non-biological chemical mechanisms.\textsuperscript{43-45} In addition, observations from the amino acid distribution coming from the Murchison meteorite show significant differences with respect to Earth’s ocean sediments (see Figure 1). Therefore, even the detection of important biosignatures of life may not be enough proof to demonstrate the presence of extraterrestrial life unless it can be shown that they come from biotic sources.
Figure 1. Relative amino acid distribution comparison between the Murchison meteorite (abiotic) and the Earth’s ocean floor sediment (biotic). Amino acids found in the Murchison meteorite: glycine, serine, and asparagine, are depicted. The rich distribution of various amino acids naturally found in the earth’s ocean floor sediment compared to the Murchison meteorite is one of the biological patterns scientists look for in celestial bodies to determine the presence of life. Reproduced from: Shock, E. L.; Schulte, M. D. Summary and implications of reported amino acid concentrations in the Murchison meteorite. Geochim. Cosmochim. Acta 1990, 54, 3159-3173. [Reference 46]. Copyright 1990 Elsevier Used with permission.

The constant dynamic plume activity present at Enceladus, propelling organic material from the subsurface sea water ocean into outer space, presents the perfect scenario to sample potential biomarkers using fly-by spacecraft containing analytical instruments. Spacecraft could be equipped with mass spectrometers to sample exospheric gas-phase molecules in the search for forms of life in space. Experiments suggest that if life were to be detected via mass
spectrometry, the presence of characteristic electron ionization (EI) fragmentation patterns for proteins, carbohydrates, and fatty acids needs to be ratified. For instance, if a protein biomarker is detected via mass spectrometry, it would leave a fragmentation pattern of phenol, nitrogen heterocycle, and cyclic dipeptide compounds; as for carbohydrates, the presence of oxygen heterocycles such as furans can be interpreted as a sign of extraterrestrial life.51, 53-59

However, there are other biomarkers that cannot be measured or identified via mass spectrometry alone. For example, chiral molecules cannot be sampled via mass spectrometry, as the fragmentation patterns will be the same for both chiral and achiral molecules unless a separation technique such as gas chromatography or capillary electrophoresis is coupled to the instrument. However, these techniques haven’t been incorporated into a space exploration probe yet. In addition, measuring isotopes wasn’t a possibility for past missions. The mass resolution of previous instruments was limited to 1-unit mass resolution, making it difficult to resolve isobaric interferences: for instance, distinguishing the CO and the N₂ isobars which both are identified at mass 28 amu. Despite this, new techniques are being developed to include chemical separation into mass spectrometry devices for space exploration.

In addition, assuming that plume content reaches the spacecraft’s path during fly-by sampling, the presence of cosmic radiation, solar wind, and the planet's electromagnetic field, which may cause ionization, radical formation, and bond-breaking of sampled species, could have an unwanted degradative effect on potential biomarker molecules.60-61

A significant part of ice grains detected in Enceladus’ plume material is believed to be frozen ocean spray.62 If frozen ocean spray is to be sampled using flyby instruments, the dominant presence of salts contained in Enceladus’ ocean may suppress the detection of less
abundant dissolved species, thus modifying and reducing the intensity of their mass spectral appearance.\textsuperscript{54} For example, soluble biomarkers like amino acids, which are expected to reside in Enceladus’ ocean ice grains, will dissolve in Enceladus’s salty ocean, which compromises their limit of detection. On the other hand, it is reasonable to hypothesize that long-chain fatty acids and low polarity biomarkers, due to their low solubility in water, will not dissolve in the ocean, but form a phase-separated organic layer on top of the water surface, which may provide the ideal environmental conditions to promote the aerosol formation of these constituents and subsequent serendipitous clathrate formation. Kieffer and coworkers\textsuperscript{63} have theorized that such biomarkers can survive the degradative effects of these radiative forces is through the formation of clathrates. Due to the hydrated, high-pressure environment in which these biomarkers are found, there is a high possibility for clathrate formation. A clathrate is an analyte molecule of low polarity that is encaged inside a crystal of water molecules. It is formed when thermal energy combines with high pressure forming a water “pocket” structure that serves as a carrier and shielding from radiation.\textsuperscript{63-68} As the encaged molecules enter space, they turn into ice grains due to the extreme low temperature and pressure becoming a clathrate see Figure 2. This has led scientists to believe that a clathrate could protect molecular evidence of life from space radiation degradative forces during fly-by probe observations.
Figure 2. Schematic of plume formation and suggested ice coated organic particle trapping mechanism. On the left, Layout of Enceladus’ rocky core similar to Earth’s, a salty water ocean rich in minerals, and ice crust proposed dynamic ecosystem at the south pole. Yellow line shows the depth above which clathrates hosting plume material are not stable. Clathrate formation is susceptible to occur at any point of the ocean where pressure and temperature conditions are met. On the right, proposed mechanism of clathrate formation and its deliverance from Enceladus’ subsurface salty ocean into space. It shows the way organics could be encaged by water molecules forming ice coated organic particles that are then carried out through a plume to be expelled into space. Reproduced from: Kieffer, S. W.; Lu, X.; Bethke, C. M.; Spencer, J. R.; Marshak, S.; Navrotsky, A. A clathrate reservoir hypothesis for Enceladus' south polar plume. Science 2006, 314, 1764-1766. & Bouquet, A.; Mousis, O.; Waite, J. H.; Picaud, S. Possible evidence for a methane source in Enceladus' ocean. Geophys. Res. Lett. 2015, 42, 1334-1339. [Reference 63 & 69]. Copyright 2006 Science. Used with permission.
1.3 Cassini Challenges during Fly-by Space Sampling

While science is still working on addressing these and other concerns, the potential of sampling and accurately identifying biomarkers of life is a major target of space exploration. There are current and ongoing efforts from NASA to send fly-by spacecraft equipped with analytical instruments to sample these water plumes in the search for biosignatures to Europa as part of the Europa Clipper Mission\textsuperscript{48,70} to be launched in 2024 and to Enceladus as presented in the Enceladus Life Finder (ELF) mission proposal.\textsuperscript{71} Due to its high sensitivity, the preferred method to detect and characterize these signatures of life for fly-by spacecraft missions is via mass spectrometry. Mass spectrometry instruments have been found in relevant space exploration spacecraft missions like Galileo Probe Mass Spectrometer\textsuperscript{72} (GPMS) in Galileo (1989-2003), the Ion Neutral Mass Spectrometer\textsuperscript{73} (INMS) in Cassini (1997-2017), the Rosetta Orbiter Spectrometer for Ion and Neutral Analysis\textsuperscript{74} (ROSINA) in ROSETTA (2004-2016), the Neutral Gas and Ion Mass Spectrometer\textsuperscript{75} (NGIMS) in Maven (2014-Present), and similar mass spectrometers are to be used for future space exploration missions like JUpiter ICy moons Explorer (JUICE)\textsuperscript{76} and Europa Clipper mission (49) in which detection of signs of life is pursued.

The Cassini-Huygens spacecraft, the most recent probe sent to Saturn, utilized the INMS during its flyby observations through Saturn and its moons. This instrument was designed to sample space material using two different inlet sources.\textsuperscript{77} One is the open-source which is operated by ionizing the sampled material immediately as it enters the instrument. The second is the closed-source which involves the use of a spherical antechamber device prior to ionization to thermalize sampled analytes by collision with the walls (see Figure 3). The open source provided
low sensitivity, whereas the closed source allowed for a ram pressure enhancement, molecular thermalization, improved signal, and higher sensitivity.\textsuperscript{78-82}

Figure 3. Cassini’s closed source antechamber design. On the left the angle of attack of incoming molecules is shown. Molecules have a higher density inside the antechamber due to RAM pressure enhancement. The line inside the antechamber represents the analyte undergoing a series of molecular collisions to become thermalized. The analyte then enters and diffuses across the transfer tube for ionization and mass analysis. Reproduced from: Teolis, B. D.; Perry, M. E.; Magee, B. A.; Westlake, J.; Waite, J. H. Detection and measurement of ice grains and gas distribution in the Enceladus plume by Cassini’s Ion Neutral Mass Spectrometer. Journal of Geophysical Research: Space Physics 2010, 115. [Reference 83]. Copyright 2010 Wiley. Used with permission.

Impact thermalization occurs as the translational kinetic energy of the sampled molecule is transferred into the walls of the antechamber’s surface in the form of thermal energy.\textsuperscript{79, 84-87} A portion of the remaining (recoil) energy goes into the vibrational energy states of the molecule.
causing it to become vibrationally excited. Thermalization takes place as this process is repeated, thus dissipating the impact energy until the molecule is relaxed. The time interval from one collision to the next inside the antechamber has to take place within the time scale of the molecule’s excitation lifetime to avoid fragmentation. Conversely, if the thermalization impacts in the antechamber occur in time scales shorter than the molecules’ excitation lifetime, it can produce undesired chemical changes such as fragmentation, ionization/neutralization, radical formation, and racemization reactions; hence, obscuring the identification of sampled chemical species in space.

With the intent of addressing this instrumental fly-by drawback, atomic-scale fragmentation studies have been performed using molecular dynamics and transition state theory. Cassini’s Cosmic Dust Analyzer (CDA) and INMS mass spectral observations of sampled plumes at Enceladus showed the rise of some prominent monotonic peaks as the spacecraft encountered the plume ice grains. A subsequent decrease in the signal of such peaks and other significant variations of unfamiliar spectral peak distributions also appeared as the probe moved away from the plume. The peak pattern suggests that the low-mass ambiguous peaks come from fragments of large organic-rich parent molecules (<200 amu). Although sampled ice grains could have caused noticeable instrumental background noise, the chance of individual molecules fragmenting and chemically recombining inside the antechamber is still plausible. This idea is supported by the presence of unfamiliar low mass fragments in the spectral data. This suggests that molecular fragmentation or chemical recombination reactions occurred to the sampled molecules as opposed to interpreting that a myriad of intact molecules sampled from the ice water grains produced such fragments. This line of evidence explains the presence of ambiguous smaller organic fragments pertaining to C_2 (>30 Daltons), C_3 (>44 Daltons), C_4
 (>56 Daltons), and C₆ (>78 Daltons) organics that were detected by the INMS and could be misinterpreted as indigenous neutral gas constituents from the plume (see Figure 4).

Figure 4. E-5 fly-by data collected from the CDA and the IMNS on Cassini. On the left CDA mass spectral results. Interference peaks of inorganic species, sodium and rubidium species, are color-shaded and marked by green triangles. Odd organic mass lines appear at 15 amu, 27 amu, 29 amu, 39 amu, 41 amu, and 43 amu indicating an atypical hydrocarbon fragmentation fingerprint (CH₃⁺, C₂H₅⁺, C₂H₅⁺, C₃H₃⁺, C₃H₅⁺+, and C₃H₇⁺). Although other interpretations for individual mass lines are possible (for example, HCN⁺ (27 amu), COH⁺ (29 amu), and CH₃CO⁺ (43 amu). On the right is the INMS mass spectra. The mass lines are color-matched to the most fitting compound (for example, H₂O (18 amu), CO₂ (44 amu), and C₆H₆ (78 amu). It also shows the presence of uncommon undefined fragments (C₂, C₃, and C₄ organics at the mass lines listed above) that are believed to be the fragmentation product of an organic molecule after undergoing a high velocity impact. Reproduced from: Postberg, F.; Khawaja, N.; Abel, B.; Choblet, G.; Glein, C. R.; Gudipati, M. S.; Henderson, B. L.; Hsu, H.; Kempf, S.;
The enigmatic fly-by results from Cassini have led to investigating the effects of hypervelocity during space sampling to predict the fragmentation or survival of sampled molecules. From a purely thermodynamic point of view, after a molecule becomes activated – in other words when it collides with a spacecraft at high velocity – there is a randomization of internal energy over all the vibrational modes of the molecule prior to any fragmentation. Experiments using Surface Induced Dissociation (SID) for ions show that there is a 14-20% conversion from translational energy (energy at the moment of the impact) to vibrational energy (internal energy of the molecule after the impact). On the other hand, the energy conversion is still unknown for neutral molecules because there are no instruments capable of producing high velocity collisions for neutrals in a controlled lab setting. The increase in vibrational energy may lead to rearrangements or fragmentation within the molecule. This rearrangement demands molecular changes to occur in an energetically favorable manner, for instance, the energy needed for bond rupture on one side of the molecule is compensated by the energy received from the collision. In addition to the velocity of the impact (translational energy), there are other important elements that affect the fragmentation process and the survival rate of impacted species such as: angle of impact, because it can affect the interaction of the sample molecule.
with the surface; molecular structure, since vibrational energy may be distributed more
efficiently in some molecules compared to others; impact surface material, for the reason that
materials interact chemically with the projectile; and the presence of ice shells surrounding the
impacted material, which can form radicals leading to molecular recombination and loss of the
indigenous characteristic of the sampled molecule. Therefore, all these theoretical elements need
to be included in a model in order to accurately predict the impact outcome of molecules
sampled at hypervelocity.

1.4 Limitations of Current Theoretical and Experimental Approaches to Study Hypervelocity
Impacts

High-velocity impact fragmentation of molecules was first modeled by Jamarillo-Botero
et al. using reactive molecular dynamics methods (RMD) utilizing Cassini's INMS sampling
conditions.\textsuperscript{90} He defined a hypervelocity impact as a collision occurring at speeds over 3 km/s.
He suggested that molecular fragmentation depends not only on collisional speed but also on the
molar mass of the impacted molecule, and that molecular fragmentation will likely occur at
speeds 5 km/s and above for molecules weighing less than 100 amu.\textsuperscript{89} Most recently, he
theoretically calculated the effects of the angle of impact, collisional excitation lifetime, and
fragmentation velocity for important biomarkers such as amino acids, fatty acids, and other
cyclic organics.\textsuperscript{97} Additionally, Klenner and coworkers\textsuperscript{54} have used molecular models to
calculate the appropriate spacecraft speed at which biomarkers can be safely collected.\textsuperscript{54} His
work suggests that the maximum sensitivity occurs at 4-10 km/s for amino acids, while for fatty
acids the optimal sampling speed ranges from 3-6 km/s. These results may also indicate that
amino acids and polypeptides that remain intact after a hypervelocity impact can be used to
identify the original constituents of space sampled particles by piecing them back together in a
way that reassembles their indigenous form. The models employed for these studies suggest a recoil lifetime after impact of <1 ps for neutral molecules, meaning a sampled molecule would need less than one picosecond segment in between collisions with the walls of the instrument to survive assuming it is charged. Although theoretical work has been done to address this subject, there are no experimental studies to back-up the theoretical work.

A different approach to address this research question is by designing instrumentation that can reduce the negative effects of fragmentation during fly-by sampling. Turner and coworkers proposed a new inlet design to reduce chemical fragmentation using the hexane molecule as a model. The inlet uses a plate with small elongated microchannels to aid the thermalization process. The design reduces the time it takes the molecule to thermalize. He proposed that his inlet design would increase the dissociation velocity of hexane by a factor of 1.25, raising the allowable velocity of the spacecraft for a given mission. In addition, Turner determined the fragmentation pattern for hexane. He predicted the formation of two different sets of radicals, the \( \text{C}_2/\text{C}_4 \) and \( \text{C}_3/\text{C}_3 \) radicals (see Figure 5). Although Turner’s microchannel plate design would reduce the diameter of the instrument’s entrance port compared to Cassini's antechamber, it would increase the spacecraft sampling velocity at which molecules may survive a hypervelocity impact. Despite Turner’s efforts to reduce the effects of molecular fragmentation during high velocity fly-by sampling, new laboratory techniques are still needed to generate hypervelocity molecular impacts which will help test instruments such as Turner’s inlet design.
Figure 5. Hexane’s proposed fragmentation pathway and its survival fraction after a hypervelocity impact. On the left (top) hexane 3-D molecular structure. At the bottom the formation of two sets of radicals C₃H₇/C₃H₇ and C₂H₅/C₄H₉ due to unimolecular dissociation of the parent molecule (hexane). On the right a comparison of the molecular survivability (no fragmentation occurs) between Cassini’s antechamber (5 cm red colored) and Turner’s design (10 µm blue colored) is shown. Reproduced from: Turner, B. M.; Anupriya; Osburn-Staker, S.; De la Cruz, A.; Crowther, P.; Sweet, L. R.; Sevy, E. T.; Austin, D. E. A microchannel thermalization inlet design to reduce molecular fragmentation in orbital and flyby closed-source mass spectrometers. Planetary and space science 2019, 172, 1-7. [Reference 78]. Copyright 2019 Elsevier. Used with permission.

Prior to addressing how space hypervelocity impacts may obscure the detection of earth-like biomarkers during space sampling, experiments had already been developed to study the effects of hypervelocity impacts on space gear and instrumentation for safety purposes of space exploration missions. The first high velocity impact studies emerged in the 1950s. They were focused on understanding the endurance of astronomical vehicles and equipment after colliding
with interstellar particles at high speed. These experiments also addressed the effects of cosmic dust on analytical instrumentation used in space probes as well as the effects of micron-size particle impacts on space protective gear. Shelton developed the first laboratory high velocity microparticle impact experiment using electrostatic acceleration where a potential drop was delivered to accelerate individual charged particles. Later on, Friichtenicht, with a modified version of the Van De Graaf accelerator, increased the potential applied to a charged particle, allowing it to reach faster acceleration. This technique eventually evolved into electrostatic dust accelerators and electric-field-induced acceleration for spores, ice, and other micron-size particle analytes of astrobiological importance. Other techniques and forms of acceleration to address space safety have also been developed involving ballistics and propellant gasses, magnetic fields using flier plates, laser shock fields, high intensity molecular beams, plasma acceleration, and linear accelerated motors. However, most of these techniques only applied to ions and charged molecules and not neutrals, these last being of great importance for MS closed-source instruments.

There are important chemical differences between ions and neutral molecules that would influence the use of neutrals for hypervelocity experimental studies. For instance, ions tend to be chemically unstable compared to neutrals. Ionization tends to weaken the bonds inside the molecule. Weakening a bond means longer average bond lengths, causing a higher tendency for bond dissociation. This could present a problem because the collision energy needed for an ion compared to a neutral may not be an accurate representation of its fragmentation after a hypervelocity impact. More importantly, closed-source instruments are meant to sample neutrals. Therefore, the high velocity impact experiments on ions will not be able to fully describe the
type of neutral impact chemistry that occurs during space sampling using the analytical source (closed-source) presented in this manuscript.

The existing laboratory techniques used for space safety are limited and insufficient to study this type of neutral impact chemistry. For example, it is challenging to produce a measurable amount of chemical post-collision products using ballistic experiments because of the destructive nature of the technique and contamination produced by the propellant gas at high pressure. Additionally, the use of electric and magnetic fields, laser shock, and plasma acceleration present a drawback for studying hypervelocity impacts of neutrals, because the analyte molecules become charged or excited prior to impact, hence weakening the stability of the analyte. Molecular beams and linear acceleration can be used to accelerate neutral molecules; however, they do not provide enough collisional energy –energy produced from bodies colliding with each other– to produce any kind of dissociation reaction. Therefore, these techniques are insufficient to study the impact chemistry in a controlled lab setting for neutral or poorly ionized microparticles. Therefore, the main hindrance to these acceleration techniques is that they rely on charge acceleration or they don’t generate sufficient velocity.

Another important element for the development of a controlled laboratory technique for neutral molecules is the size of the particle. Due to its low mass detection range (100 m/z), INMS was primarily designed to sample gasses of molecular size, whereas Cassini CDA was intended to detect microparticles in the form of cosmic dust and ice grains. While orbiting Enceladus, Cassini sampled material in the form of both molecules (atomic size) and ice grain microparticles (<100-micron size). To understand how hypervelocity may have affected the accurate detection of chemical species sampled by Cassini, microparticle acceleration
experiments using electrospray ionization and light gas guns have been performed to decipher
the CDA fly-by data.

At the University of Kent, Burchell and coworkers conducted experiments firing up to
7.4 km/s water-ice grains microparticles doped with organics (4.5 mm diameter, 6.0 mm length)
using a hollowed-out sabot light gas gun.\textsuperscript{114} A similar experiment using organic ice-particle
simulants (4-10 microns) was also performed. It fired iced organic material using a light gas gun
reaching speeds up to 3 km/s.\textsuperscript{105} However, these instruments were not capable of identifying any
chemical changes in-situ because of their inability to be operated under high vacuum conditions.
In addition, at the University of Colorado Boulder, Fe and Al micron size particles were
impacted into a cryocooled water-ice surface at 3-50 km/s.\textsuperscript{115} The resulting impact produced ions
that were then analyzed using Time of Flight Mass Spectrometry (TOF-MS). A limitation to this
method is that the energy absorbed by a micron-sized metallic ice grain does not equally
compare to the analytes found in space plume ice grains. Current work done at University of
California San Diego using the Aerosol Impact Spectrometer (AIS) is capable of trapping 0.1-2-
micron single ice grain particles.\textsuperscript{100} The instrument allows for size and charge characterization by
accelerating grains up to speeds of >2 km/s using linear electrostatic acceleration stages (see
Figure 6). The post-impact ions are then characterized using TOF-MS. This system is a
promising technique for poorly ionized microparticles, and it is in the process of reaching
velocities up to 5 km/s.
Figure 6. Schematic and perspective of the Aerosol Impact Spectrometer. Components starting from the charged aerosol source: Enclosed electrospray ionization (ESI) Desolvation tube; Aerodynamic Lens (ADL) Differential pumping, ion optics and image charge detector ICD-QD1, Quadrupole Deflector (QD), ion optics and ICD-QD2; Injection optics for nanoparticle Electrostatic Trap (NET), including ICD 1; NET, including ICD 2; Injection optics; and 9-stage Linear Accelerator (LINAC); Final ICD (ICD3) and collision target. Indications of image charge detection tubes are indicated with a black arrow. Reproduced from: Adamson, B. D.; Miller, M. E. C.; Continetti, R. E. The aerosol impact spectrometer: a versatile platform for studying the velocity dependence of nanoparticle-surface impact phenomena. EPJ Tech. Instrum. 2017, 4, 2. [Reference 100]. Copyright 2017 Springer. Used with permission.

In addition, the Jet Propulsion Lab (JPL) is currently developing a similar system to impact ice grain onto a metal plate at 3-5 km/s and orthogonally analyze the ions using TOF.116
However, this technique relies on electrostatically charging the ice grain and is not suited for neutral species; hence the need to develop instruments that can precisely study hypervelocity impacts of neutral species is still a challenging obstacle for instrument developers.

Not only is it a challenge designing neutral particle hypervelocity impact experiments with the appropriate experimental controls similar to space conditions, but also interpreting the high velocity collision results of the experiment. For instance, for charged microparticles, Smith and Adams, investigated the plasma production resulting from a microparticle hypervelocity impact and the possible molecular recombination process caused by the plasma.\textsuperscript{117} Burchell and coworkers found that the microparticle’s percent charge prior to surface collision affected the degree of impact ionization.\textsuperscript{118} In addition, Sysoyev and coworkers, showed that high velocity charged particles undergo electron bombardment, turning it into a neutral, prior to impact, resulting in surface ionization.\textsuperscript{119} As a result of these experiments, high velocity impacts of microparticles both charged and neutral are expected to have different impact products, perhaps even for microparticles of the same composition and same velocity.

Even if the high velocity impact of charged microparticles differs essentially from that of neutral microparticles, high velocity impact experiments of neutral molecules present a similar drawback compared to high velocity microparticle impact experiments, since individual molecules must remain in their neutral form prior to surface impact; no controlled laboratory experiment has been able to achieve this yet. Additionally, a sort of impact products difficult to characterize may also be expected. Despite this, neutral molecular high velocity impact experiments are still of astrobiological important as they can help resolve questions regarding the detection of material sampled by Cassini’s INMS antechamber, a process that hasn’t been addressed in any previous experiments. While most of the dissertation will focus on presenting
and developing a technique for molecular hypervelocity impacts using a high-speed rotor, the potential for including a charged microparticle accelerated device into the experiment will also be addressed briefly in Chapter 4.

1.5 High speed Rotors, a suitable technique to study hypervelocity impacts

High speed rotors present an alternative technique to produce hypervelocity impacts for neutral species compared to the previous methods because, according to literature, they are capable of producing high speed rotation >2 km/s.\textsuperscript{112} As a laboratory technique, a high-speed rotor is unique and versatile because it can be easily adapted to other acceleration techniques to study not only molecular but also microparticle hypervelocity impacts. For example, a spinning rotor can be coupled to both a molecular beam and an electrospray microparticle accelerator.

Rotors are mechanical devices that use an electric or magnetic field to produce high speed spinning. The first rotor design found in literature was developed by Lodge in 1897. He paired two hard, steel discs (1 ft. thickness, 1-yard diameter, 711 kg of weight) clamped on a vertical axis an inch apart from each other, reaching a speed close to 3000 rpm (~150 m/s).\textsuperscript{120} The rotation mechanism consisted of a set of balancing screws and ball bearings rested on a collar that supported the shaft see Figure 7. The shaft consisted of a hard steel pivot placed on another steel surface located inside an oil chamber. The shaft was too weak to carry the weight and had the tendency to bend. The shaft was mechanically powered by a motor and operated at atmospheric pressure. He used his rotor to bifurcate a beam of light, sending each half in opposite directions, in order to measure the “ether” through which it was speculated light propagated. He also used it to create a magnetic field by applying a current through wires attached to the discs, creating a dynamo.
Figure 7. 2-D visualization of the oblate spheroid spinning device. It shows the placement of the balancing screws, shaft, and the wire holes. Reproduced from: Lodge, O. J. VI. Experiments on the absence of mechanical connexion between ether and matter. Philosophical Transactions of the Royal Society of London. Series A, Containing Papers of a Mathematical or Physical Character 1897, 149-166. [Reference 120]. Copyright 1897 RSC. Used with permission

Subsequently, in the 1940’s, Marshall, Bull, and Moon were the first to develop a rotor for molecular science. The rotor spun around its vertical axis, traveling at speeds of ~1 km/s.\textsuperscript{112} It was used to guide particles onto an orifice by collision with the rotor wings to produce a collimated high-speed beam of particles.

They were the first ones to develop the theory and experimentally test high speed rotation devices. Moon’s theoretical work focused on determining the dimensions of an optimum rotor shape. Moon contrasted two main rotor designs: a rotating disc and a rotating tapered rod. He compared the ideal rotor shape to a Gaussian curve extending to an infinite radius or zero thickness for both a disc and a tapered rod. A tapered rod achieved higher rotational speed theoretically due to the absence of circumferential stresses resulting in a better rotor design. In
addition, he developed the mathematics needed to calculate the optimal variation of cross-sectional area (thickness/width) as a function of radius for a tapered rod rotor. Their model is based on keeping the tensile force uniform throughout the length of the rotor by varying the wing cross sectional area as a function of distance from the rotational axis. This assumes that the stress is such that it is the same at all parts of the rotor wing, therefore, maximum rotational speed depends only on the shape and material of the rotor. The optimum rotor cross section across the blade area can be calculated by the following equation:

$$A \rho \omega^2 r dr = -\frac{d}{dr} (SA)dr$$  \hspace{1cm} (1)

Where A is the cross-sectional area, as a function of the distance, r, from the rotational axis; r is the radius, \(\rho\) is the density of the material, \(\omega\) is the angular velocity, and S is the maximum stress the material can withstand before breaking or permanent deformation.

Assuming that not only stress but also density is uniform and constant, the maximum velocity at which the rotor would break will be defined by the log of the ratio of cross-sectional areas as shown in this equation:

$$V_m = \sqrt{\frac{2S}{\rho} \ln \frac{A_0}{A_m}}$$  \hspace{1cm} (2)

Where \(V_m\) is the velocity at the point of truncation (desired tip distance); \(A_0\) is the cross-sectional area at the origin of the rotational axis, and \(A_m\) is the cross-sectional area at the tip.

Another important consideration when calculating the rotor's optimal shape (thickness/width) is the machinability of the material; for instance, if the rotor blade becomes too thin, it’s hard for the machinist to make a cut on a highly dense material leading to accidentally
breaking the blade. As a result, Moon sketched and experimentally tested several shapes of tapered rod rotors to prove his theoretical work. His rotor designs became the first high rotational spinning devices published in literature (see figure 8).\textsuperscript{112}

Figure 8. Rotor shapes: (a) Cone, (b) sphere-and-cones, (c) sphere-and-double-cones; Rotor elements: G, gully; B, hard-steel ball. Reproduced from: Marshall, D. G.; Moon, P. B.; Robinson, J.; Stringer, J. T. A Technique for High Rotational Speeds. J. Sci. Instrum. 1948, 25, 348. [Reference 112]. Copyright 1948 IOP. Used with permission

Moon also reported some important complications during his rotor performance tests. For example, during testing, Moon observed that the rotor’s orbital motion created a motion frequency different from its natural frequency of rotation. This led to nutation, a wobble effect in the rotational axis of an object in motion. Nutation presented an impediment to developing a
fixed rotational axis rotor. In addition, nutation created violent disturbances in the rotation that ended in catastrophic failures during tests at high speed. Moreover, the frictional forces caused an increase in the heat of the system, achieving a temperature of almost 150 degrees Celsius. In order to reduce rotor heating, Moon made improvements to his design by changing the size and profile of the magnetic pole, leveling the parts in the rotational system, and polishing the bearing balls. However, his improvements had little to no effects in reducing the heating of the rotor.

From the rotor designs presented by Moon, the design with the best performance consisted of a sphere of double cones made of a nickel-chromium-molybdenum steel alloy; its shape consisted of two conical shaped wings that extended on the sides of a tapered, hard sphere ball (see figure 8b). The rotation was driven by a rotating magnetic field under rough vacuum conditions of $1 \times 10^{-3}$ Torr inside a glass chamber (see figure 9).
Soon after, in 1951, Moon’s newly developed rotor found an application in the generation of pulsed molecular beams. Bull and Marshall employed Moon’s design with a little modification to the blades to produce a beam of potassium atoms. They used a steel tapered rod at both ends, each arm terminated in a small cup which was filled with potassium to create pulses of potassium atoms see Figure 10. The experiment was used to calculate the lifetime of
potassium ions as they travel through a tungsten filament.\textsuperscript{121, 122} The rotor was accelerated to 90,000 rpm using the same rotating magnetic field employed by Moon.

Figure 10. Apparatus used to create a pulsed beam of potassium atoms using a high speed rotor laboratory technique. Reproduced from: Moon, P. B. Some scientific applications of high speed-rotation. British Journal of Applied Physics 1953, 4, 97. [Reference 123]. Copyright 1953 IOP. Used with permission

Additionally, in 1953, Moon published an article expanding his theoretical work and further explaining the principles of rotor design and their application.\textsuperscript{123} Using the Gaussian error function, he predicted the limited tip speed at which a rotor would break based on the tensile strength and density of the material and the blade's cross-sectional area. He also addressed issues related to fixed axis of rotation, motor power, and rotational stability. Furthermore, he introduced two new rotor shape designs: a mushroom shaped and a single-winged rotor (see Figure 11).
Later on, in 1954, Bull and Moon performed the first crossed beam experiment using a one wing high speed rotor-assisted pulsed molecular beam to study the formation reaction of CsCl from impacting CCl₄ with Cs gas reaching 0.6 km/s speed. The used an aluminum alloy rotor with an effective tip area of 0.25 cm² to sweep away a greater number of molecules on each cycle. They performed the experiment inside a Pyrex glass chamber and were able to achieve 90,000 rpm of rotational speed. A schematic of their experimental design can be seen in Figure 12.
Figure 12. Experimental rotor set up showing the Pyrex glass chamber and the other experimental elements. Rotor is on the left, S entrance slit, R stream of cesium, C cold trap, F tungsten filament, K cylinder was negatively charged with respect to the tungsten filament, which acted as a surface ionization detector. Reproduced from: Bull, T. H.; Moon, P. B. A mechanical method for the activation of fast reactions. Discuss. Faraday Soc. 1954, 17, 54-57. [Reference 121]. Copyright 1954 RSC. Used with permission

Two decades later, in the late 1970’s, Moon, Rettner, and Simons generated a pulsed supersonic beam for molecules as big as W(CO₆) using a carbon fiber composite material rotor with a conical tapered shape.¹²⁴ The carbon fiber composite shaft was cemented with epoxy to a central stainless boss. The rotor was magnetically levitated and spun up to >2 km/s. The drive system was provided by a rotating magnet field generated by 4 water-cooled coils surrounding
the rotor and operated at 7.5 $10^{-5}$ Torr see Figure 13. The phase and the frequency of the rotor was measured by a He/Ne laser.

Figure 13. (a) Rotor vessel assembly; A, rotor; B, levitation lamp; C, cooling coil; D, damping magnet; E, lift magnet; F, levitation photocell; G, frequency-monitored photocell; H, He/Ne laser beam. (b) Apparatus for beam characterization; A, rotor; B, collimating aperture, C, quadrupole mass spectrometry for gas analysis; D, gate valve; E, quadrupole mass spectrometer

Two years later, Barker, Moon, and Ralls published a work on carbon fiber reinforced plastics as materials for high speed rotors and proposed a new rotor design, a steel ball with thin blades see figure 14. They used computer calculations to determine the breaking speed and performance of their design at high rotational speeds. In addition, they compared their design to other rotor designs previously introduced by Moon (see Figure 15).

![Figure 14](image)

Figure 14. 2-D final design as cemented into the steel ball for levitation and driving. Reproduced from: Barker, A. J.; Moon, P. B.; Ralls, M. P. Carbon fibre-reinforced plastics as a material for high-speed rotors. Composites 1979, 10, 149-151. [Reference 125]. Copyright 1979 Elsevier. Used with permission
Figure 15. Examples of rotor shapes: (a) Gaussian, (b) double cones, (c) cylinder and truncated cones. Reproduced from: Barker, A. J.; Moon, P. B.; Ralls, M. P. Carbon fibre-reinforced plastics as a material for high-speed rotors. Composites 1979, 10, 149-151. [Reference 125]. Copyright 1979 Elsevier. Used with permission

In addition to the theoretical work, Barker et. al also performed physical experimental tests on his designs as well as Moon’s. The rotors were built with carbon fiber reinforced plastics and spun to 75% of their theoretical maximum speed. The maximum rotor speed reported from their tests was ~2 km/s and was maintained for several hours.

In the same decade, Moon and Ralls introduced another application to high-speed rotation and developed a twin-rotor system, which involved the synchronization of two high speed rotors.\textsuperscript{126} They discovered that when the rotors are operated simultaneously, an impacting region is formed in between the two rotors where molecules can collide, similar to a cross beam...
experiment. The twin rotors were made of carbon fiber reinforced materials and synchronized
digitally using a phase lock system and were able to rotate at a speed of 2.2 km/s.

Lastly, in Moon’s final works, he theorized a solution to the overheating problem present
in his previous rotors. He suggested the use of thinner shafts and even reverting the shaft back to
a disc shape. Additionally, Moon theorized that future new materials, high in tensile strength and
low weight, could allow for a reduction of heat during high-speed rotation. Lastly, he also
proposed new shapes for rotor blades including a hollowed rotor and curved rotor tips similar to
the blades used in turbomolecular pumps.

After Moon’s work, rotors were used solely to produce high speed atomic and molecular
beams mainly for spectroscopy and chemiluminescence applications. For example, Simmons
et. al, building on Moon's rotor work, created a carbon fiber composite rotor to generate a
superthermal molecular beam. They manipulated the speed of the rotor from 0 to 2.5 km/s at a
pressure of 7.5 x 10⁻⁵ Torr. They used his experimental setup to differentiate metastable atoms.
The ability to pulsate and control the speed of the molecular beam allowed them to study other
spectroscopic parameters later on.

A new awakening in rotor technology occurred in 1999 when Herschbach took Moon’s
rotor prototype of a hollowed one-wing and mounted a gas-fed line system at the tip of Moon’s
rotor to create a supersonic molecular beam spray. His rotor was mechanically driven and was
able to accelerate O₂ molecules up to ~1 km/s. The rotor was made of aluminum alloy T6,
9.9 cm in length, and 29 g in weight. The shape consisted of cylindrical stepped segments
approximating the shape of a Gaussian error curve across the blade (see Figure 16). The rotor’s
shaft was made of stainless steel to minimize heat conductance. The shaft was grasped to a
vacuum motor equipped with ceramic bearings that were lubricated with non-volatile vacuum grease. The AC motor was bolted to a water-cooled copper plate and held approximately to 18 °C. In addition, the motor was also bolted to a stainless-steel block to dampen any internal vibrations during spinning. An adjustable screw was used to enable better rotor’s rotational balance located at the end of the rotor (see Figure 16). The entire motor driven unit was mounted on neoprene spacers to dampen further vibrations (see Figure 17). The rotor was able to reach 42,000 rpm and a peripheral velocity of 435 m/s.

![Figure 16. One-wing rotor, the exit aperture contains a pinhole nozzle to produce a supersonic beam, the segmented regions are shown as well as the inlet gas source and the location of the balancing screw. Reproduced from: Gupta, M.; Herschbach, D. Slowing and speeding molecular beams by means of a rapidly rotating source. The Journal of Physical Chemistry A 2001, 105, 1626-1637. [Reference 129]. Copyright 2001 ACS. Used with permission](image-url)
Figure 17. Plane view of the rotor, gas feed, and drive mechanism. Components: A, rotor; B, needle gas inlet; C, motor; D, mounting block; E, cooling plate; F, accelerometer; G, neoprene. Reproduced from: Gupta, M.; Herschbach, D. Slowing and speeding molecular beams by means of a rapidly rotating source. The Journal of Physical Chemistry A 2001, 105, 1626-1637. [Reference 129]. Copyright 2001 ACS. Used with permission

Since then, Herschbach's one-wing spray rotor prototype has served as a pattern for many more molecular beam experiments. For example, Makarov, using an almost identical rotor design, generated an atomic beam to study molecular radicals. Even though the rotor designs that came after Herschbach’s prototype differed in matters of size, length, and material composition, there are some commonalities between them. For instance, they both use an inductive AC driving motor, rotate at speeds close to 0.6 km/s, use the same spray gas delivery system, and are speed-controlled, meaning they can be accelerated or decelerated according to the needs of the experiment.
On the other hand, some scientists persisted in working with the original two-wing rotor design presented by Moon. For instance, Narevicius et al. generated a 0.5 km/s two-wing rotor-assisted supersonic beam. Their rotor was made out of titanium with a 50.5 cm radius, see Figure 18. The significant increase in the rotor's diameter allowed for lower rotational rates. The rotor spinning mechanism was coupled with a ferrofluid feedthrough that operated up to 10,000 rpm. The entire system was run under vacuum at a pressure of \(10^{-8}\) Torr. Thus, the theory, equations, and designs introduced by Moon and successors of his works laid the foundation and basis for high speed rotation devices.

Figure 18. Schematic of the rotor apparatus. Positions of turbomolecular pumps are indicated as P1-P4; the atom trajectory is shown as a dotted line. The dashed lines stand for the propagation path of the direct beam. Reproduced from: Narevicius, E.; Libson, A.; Riedel, M. F.; Parthey, C. G.; Chavez, I.; Even, U.; Raizen, M. G. Coherent slowing of a supersonic beam with
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A summary of the key characteristics of the most important high-speed rotation devices previously described is presented in the following Table 1.
Table 1. The chronological development of high-speed rotors for scientific applications. A description of their design, experimental performance, and unique characteristics is also included. Note that additional rotors have been used in the recent years, however they are patterned after Gupta & Herschbach’s design; therefore, they are not mentioned in this table of unique rotor designs.

<table>
<thead>
<tr>
<th>Builder</th>
<th>Year</th>
<th>Shape and dimensions</th>
<th>Material</th>
<th>Vacuum Pressure</th>
<th>Drive Rotational System</th>
<th>Application</th>
<th>Speed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oliver Lodge (120)</td>
<td>1897</td>
<td>Two paired hard steel discs clamped on a vertical axis (1 ft. thickness, 1-yard diameter, 711 kg of weight)</td>
<td>Steel</td>
<td>Atmospheric</td>
<td>Motor powered</td>
<td>Bifurcating a beam of light and creating a magnetic field</td>
<td>~0.1 km/s 3,000 rpm</td>
</tr>
<tr>
<td>D. G. Marshall, P. B. Moon, F. R. S., J. E. Robinson, and J. T. Stringer (112)</td>
<td>1948</td>
<td>They tested a cone, a sphere and cone, and a sphere of double cones (4 in diameter, weight not found)</td>
<td>Nickel-chromium-molybdenum steel</td>
<td>$1 \times 10^{-3}$ Torr</td>
<td>Iron-cored lifting magnets to drive the rotor with driving coils and magnetic oil suspension</td>
<td>Proof of concept for future molecular beam applications</td>
<td>$&gt;1$ km/s 90,000 rpm</td>
</tr>
<tr>
<td>T. H. Bull, P. B. Moom (121)</td>
<td>1954</td>
<td>One wing High speed rotor with an effective tip area of 0.25 cm²</td>
<td>Aluminum alloy</td>
<td>Not found</td>
<td>Magnetically levitated motor driven</td>
<td>Pulsed molecular beam</td>
<td>0.6 km/s</td>
</tr>
<tr>
<td>P. B. Moon, C. T. Rettner, and J. P. Simons (124)</td>
<td>1977</td>
<td>Conical tapered shape where the shaft was cemented with epoxy to a central stainless boss</td>
<td>Carbon fibre composite materials</td>
<td>$7.5 \times 10^{-5}$ Torr</td>
<td>Rotating magnet field generated by 4 water-cooled coils surrounding the rotor</td>
<td>Pulsed supersonic beam</td>
<td>$~2$ km/s</td>
</tr>
<tr>
<td>A. J. Barker', P. B. Moon, and M. P. Ralls (125)</td>
<td>1979</td>
<td>Shapes: Gaussian, double cones, cylinder truncated cones, and steel ball with thin blades (15 mm diameter)</td>
<td>Carbon fiber reinforced plastics</td>
<td>Not found</td>
<td>Magnetically levitated motor driven</td>
<td>Testing of new rotor designs</td>
<td>$~2$ km/s</td>
</tr>
<tr>
<td>P. B. Moon, M. P. Ralls (126)</td>
<td>1989</td>
<td>Twin rotors</td>
<td>Carbon fibre reinforced materials</td>
<td>Not found</td>
<td>Magnetically levitated motor driven</td>
<td>Synchronization of two high speed rotors</td>
<td>$~2.2$ km/s</td>
</tr>
<tr>
<td>M. Gupta, D. Herschbach (130)</td>
<td>1999</td>
<td>Stepped cylindrical segments approximating the shape of a gaussian error curve (9.9 cm diameter, weight 29 g)</td>
<td>Aluminum alloy T6</td>
<td>100 Torr</td>
<td>AC motor</td>
<td>Gas-fed spray rotating device</td>
<td>0.6 km/s 42,000 RPM</td>
</tr>
<tr>
<td>E. Narevicius et. al. (132)</td>
<td>2007</td>
<td>Two-winged rotor (50.5 cm radius)</td>
<td>Titanium</td>
<td>$1 \times 10^{-8}$ Torr</td>
<td>Motor coupled with a ferrofluid feedthrough</td>
<td>Rotor-assisted supersonic beam</td>
<td>0.5 km/s 10,000 rpm</td>
</tr>
</tbody>
</table>
Despite the different rotor designs and applications previously mentioned, nobody has utilized high speed rotation devices to create hypervelocity impacts experiments yet. Designing a rotor for hypervelocity impacts requires important adaptations. For instance, a flat impacting surface at the end of the rotor’s wings is needed, which wasn’t found on nor built for any previous rotor designs. Based on Moon’s rotor theory, the shape of a rotational device has a direct impact on the maximum rotational speed attainable. In addition, the change in shape could add unwanted imbalances to the rotational stability and as a result cause overheating of the system’s electronics. Therefore, small changes in the rotor's shape can greatly affect its performance.

The work presented in this manuscript discusses the development of a laboratory instrument that can produce high velocity impact collisions. We decided to modify previous rotor designs to build a high-speed rotor that could be used for high velocity impacts for both molecules and microparticles alike. We adapted a Moon’s Gaussian shaped rotor to include flat paddles at the ends of the wings. This required us to apply Moon’s rotational speed equations in our design to maximize the cross-sectional area across the rotor’s blade. In addition, we included extra rotor blades to our design, which eliminated the rotor’s intermediate axis of rotation and as a result increased rotational stability. Chapter 2 will be devoted to the design, construction, and characterization of a magnetically levitated high-speed rotor running at <1 km/s speed as well as to the results from the characterization of the rotor’s material elongation while spinning. Lastly, other experimental applications for the rotor, such as molecular adhesion at high speed, will also be discussed later in chapter 5.
Even though the rotor presents a new approach to produce high velocity impacts, it still doesn’t produce enough collisional energy (<1 km/s impact speed) to observe any qualitative chemical changes as a stand-alone instrument. This opened the possibility to incorporate other neutral acceleration techniques into the design. The addition of a molecular acceleration to the rotor technique could provide the additional collisional energy (at least 3 km/s speed) needed to generate chemical changes upon collision onto a target. In addition, a molecular beam will allow for molecular size particle hypervelocity impact experiments. Molecular beams have been employed to transport gases to supersonic speeds by utilizing a pressure differential to accelerate a gas through an orifice (>2 km/s). They can accelerate molecules while maintaining a collisionless environment while still obtaining useful densities. In addition, molecular and atomic beams provide an excellent means for many other detailed studies; for example: the interaction of neutral molecules with surfaces, with other particles, and with fields. They have been used in experiments that seek to study the interactions of: atom-atom, atom-ion, atom-electron, atom-photon scattering; magnetic resonance methods, photoelectron and fluorescence spectroscopy; and interactions of free metals atoms with photons or charged particles, and electrons or protons. For these reasons molecular beams present an ideal technique to accelerate neutral molecules and increase collisional energy, which is desired for our experiment.

Another advantage to using a molecular beam is that it can be seeded. Seeding supersonic beams produce a velocity distribution under high vacuum conditions, where if the carrier gas is lighter than the seeding molecule, the latter is accelerated to high energies. While seeding may not change the speed of the beam, the capability of introducing different types of neutrals inside a carrier gas makes this instrument a robust technique for our purposes, which are to test several molecules of astrobiological importance. In addition, the molecular beam’s ability to produce
high speed molecular collisions under vacuum will allow for quantitative and qualitative analysis post-collision. Hence, we decided to build a custom molecular beam to complement the previously outlined high speed rotor device. The molecular beam will theoretically increase the impact collisional energy of the experiment to ~3 km/s by firing molecules at 2 km/s to the tips of a 1 km/s spinning rotor. The design and assembling of the molecular beam as well as preliminary results will be presented in chapter 3. Chapter 4 will present and explain the future assembly of these two instruments (rotor and molecular beam) as well as future experiments involving the rotor as a stand-alone instrument; for instance, utilizing the rotor to measure molecular adhesion onto a surface at high rotational speeds. Standard operational procedures and detailed assembly instructions for both the rotor and molecular beam will be presented in Appendix A and B.

I am proud to present the pioneering work, construction, and experimental performance of the first Rotor Induced Collision Cell (RICC).
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CHAPTER 2: ROTOR DESIGN, CONSTRUCTION, AND PRELIMINARY TESTS

Note: this chapter is largely taken from a manuscript being prepared for submission to the *International Journal of Impact Engineering* titled High-speed rotor for microparticle impact studies by Abraham De la Cruz\(^a\), Timon Achtnich \(^b\), Emile Decosterd\(^b\), Eric T. Sevy\(^a\), Matthew C. Asplund\(^a\), Daniel E. Austin\(^a\)*

2.1 Introduction

Hypervelocity impacts of microparticles have been studied for over a century in the context of micrometeoroid processes in space. Recent interest includes impacts of microparticles with significant water and organic composition, driven in part by the discovery of plumes erupting from subsurface oceans on Enceladus and potentially from Europa and the potential habitability of such oceans. Chemical information derived from in situ analysis of impacting micrograins is complicated by the lack of robust methods of reproducing such impacts in the laboratory. For example, Techniques for producing organic and water ice micrograins are being developed but are not yet a mature technology.\(^1\) Techniques for accelerating microparticles typically rely on the particles being electrically charged; however, the charge itself alters the chemistry of the impact due to rapid electron transfer immediately prior to impact.\(^2\) In addition, charge-based acceleration suffers from a trade-off between particle size and particle velocity—larger grains (i.e., in the micron size range and larger) cannot be accelerated to sufficiently high speeds to stimulate the velocity of a typical flyby or orbiting spacecraft. Acceleration Techniques using light gases for ballistics and propulsion, such as light-gas gun, can accelerate much higher projectiles, but are not ideal for chemical quantification of post collision products due to chemical interference from propellants.\(^3,4\)

In this paper we describe an alternative approach based on a mechanical rotor spinning at high speed. Rotors have been developed previously and found application to other areas of
interest. As far as 1897, Lodge et al.\textsuperscript{5} paired and spun two steel discs were screwed together on a vertical axis, the screws fixed the rotational axis acting as a shaft. The discs reached a speed close to 3000 rpm (revolutions per minute) or 144 m/s. Subsequently, in the late 1940’s Marshall, Moon, and coworkers\textsuperscript{6} developed a two-winged rotor. It consisted of a nickel-chromium-molybdenum steel tapered rod rotating around its axis using a magnetic driving field. The shape of the rotating rod consisted of two conical shaped wings that extended on the sides of a tapered, hard sphere ball. Therefore, no shaft was used to fixed the rotor’s axis of rotation. The rotational device reached a tip speed of >1 km/s.\textsuperscript{6} Later on, Bull and Moon\textsuperscript{7} generated a beam of molecules using the same rotor prototype to perform one of the first molecular crossed-beam experiments to study the formation of CsCl from CCl\textsubscript{4} and CS.\textsuperscript{7} After that, Bull and Marshall\textsuperscript{8} employed a similar rotor device created beam pulses of potassium atoms and calculated the lifetime of potassium ions on a tungsten filament.\textsuperscript{8} Following work performed by Moon and colleagues\textsuperscript{9} applied the rotor to generate molecular beams of heavier gases.\textsuperscript{9} A few years later, Rettner & Simons\textsuperscript{10,11} used a different rotor material, carbon-fibre composite (c.f.c), but same rotational mechanism to achieve 1.5 km/s speed. Barker et al.\textsuperscript{15} using the same material and a similar magnetically driving mechanism tested rotors of different blade shapes and was able to reach 2 km/s speed.\textsuperscript{15} During the late 1980’s, Moon & Ralls\textsuperscript{12} found another application for rotor mechanical acceleration and developed a twin-rotor system, which involved the synchronization of two ultra-high spinning rotors reaching speeds of ~2 km/s.\textsuperscript{12} From this time forth, the rotor design developed by Marshall and Moon was used mainly as a technique to accelerate metastable atoms to produce atomic beams at high-speed for spectroscopy and chemiluminescence applications.\textsuperscript{13} However, despite all of its different scientific applications, the Marshall and Moon two-wing rotor design is insufficient for microparticle impact experiments because
mechanistically it doesn’t use a shaft to fix its rotational axis, which rotational steadiness is important for aiming microparticles with precision onto the tips of the rotor blades.

Common challenges of the Marshall & Moon rotor design included overheating of the rotor shaft\textsuperscript{14} and difficulties in rotational stability.\textsuperscript{6,15} To overcome challenges regarding the heating of the rotor electronics, Moon\textsuperscript{14} suggested the use of thinner blades and shafts and even reverting the shape of the shaft to a disc. He also the use of future undiscovered materials, high in tensile strength and low density could allow for a reduction of heat during high-speed rotation. Lastly, Moon also proposed that increased rotational stability from previous rotor designs would require high precision machining of the rotor’s shape.\textsuperscript{15} Another challenge for impacts onto rotors is that the impact angle varies with the curvature of the rotor surface, and the impact speed varies along the length of the rotor arm.

A decade later, Gupta et al.\textsuperscript{16,17} took Moon’s prototype of a hollowed one wing rotor to mount a gas-fed line system to create a supersonic beam spray, which was used to accelerate O\textsubscript{2} molecules to <1 km/s.\textsuperscript{16,17} The rotor was made out of aluminum with a cylindrical segmented shape across the blade. It was mechanically driven, contained a steel shaft grasped into a motor and rotated under a fixed axis. This same prototype served as a pattern for many more molecular beam applications. For example, Makarov and coworkers\textsuperscript{18} using an identical rotor design generated an atomic beam to study molecular radicals.\textsuperscript{18} However, meanwhile other groups persisted in working with and further developing the original two-wing rotor design presented by Marshall & Moon. For instance, Narevicius et al.\textsuperscript{19} using a titanium rotor, produced a 0.5 km/s helium beam to study coherent slowing of a supersonic expansion.\textsuperscript{19} Even though Gupta’s one-wing rotor design holds a fix axis of rotation, it is limited because of its low peripheral speed and blade shape for high-speed microparticle impact experiments. Prior work
focused on molecular impacts onto rotor surfaces, but none of these studies (Marshall & Moon and Gupta) has looked at the possibility of impacting microparticles.

To better constrain parameters of impacts with microparticles, we desired to have flat surfaces (paddles) at the tip of each rotor wing. Previous rotors have not had this feature; hence the impact angle varied widely.

We present here the design and testing of a novel rotor for microparticle impacts. The design is generally based on Marshall’s et al. equation for wing cross sections, with the addition of flat surfaces on each wing tip. The rotor includes four wings and two shafts and is spun using two magnetically levitated bearings and motors. A titanium rotor based on this design was built and tested. In addition, elongation of the rotor arms as a function of speed was measured using a laser light optical technique.

2.1.1 Theoretical Overview

Marshall et al. presented a mathematical model for the optimal rotor shape to achieve the highest possible rotational speed. Their model is based on keeping the tensile force uniform throughout the length of the rotor by varying the wing cross sectional area, $A$, as a function of distance, $r$, from the rotational axis:

$$A \rho \omega^2 r dr = - \frac{d}{dr} (SA) dr \quad (1)$$

Where $\rho$ is density of the material, $\omega$ is the angular velocity, and $S$ is the maximum stress the material can withstand before breaking or permanent deformation. The resulting optimal
shape follows a Gaussian curve. Assuming truncation of the wings at some distance, \( m \), from the rotational axis, the maximum theoretical velocity of the wing tip is:

\[
v_m = \sqrt{\frac{2S}{\rho \ln \frac{A_0}{A_m}}} \tag{2}
\]

Where \( v_m \) is the velocity at the point of truncation (the wing tip), \( A_0 \) is the cross-sectional area at the origin (at the rotational axis), and \( A_m \) is the cross-sectional area at the tip. The equation also holds if \( A_0 \) represents the cross-sectional area at any point between the tip and the rotational axis, thus avoiding the geometric complications of determining cross sections near the axis. Note that the above model ignores shear stresses in the rotor material. Also, the maximum velocity calculated as above does not consider the physical elongation or strain of the rotor wings due to the stress applied by rotation.

These equations suggest that the material’s tensile strength, its density, and the rotor shape are the only factors affecting the maximum achievable tip speed. As a smaller tip can reach a higher velocity, there is a trade-off between overall rotor size or mass, tip size, and tip speed. Not surprisingly, a high-tensile strength, low-density material allows the highest rotational speeds for a given rotor shape, size, or weight.

2.2 Experimental

In contrast to prior rotor designs, all of which employed cylindrical or conical wings, for our application we desired to have flat impact surfaces (“paddles”), and to keep them aligned so that the surface was always perpendicular to the instantaneous velocity vector. This required aligning the rotor tips (and hence, the entire rotor) with the rotational axis. To accomplish this,
the rotor design includes a shaft for mounting. Initial experiments with a shaft-mounted two-wing rotor showed instability due to the intermediate axis theorem. Specifically, the desired rotational axis had the intermediate moment of inertia among the three Cartesian axes, resulting in a tendency for unstable rotation. Laboratory attempts to spin this two-wing rotor ended in a catastrophic failure, likely due to rotational instability. The subsequent design includes four wings, solving this intermediate axis problem. As an additional advantage, four wings instead of two results in twice as many impacting surfaces per rotation.

A lighter, three-winged rotor design was considered, but might have become instable at higher speeds if there were any anisotropy in the Young’s modulus of the rotor material. A lower modulus would result in more elongation and a higher moment of inertia at higher speeds. With four wings, even if the elongation differed between the x- and y-wings (assuming z is the rotational axis), the two x-wings would balance each other, and the two y-wings likewise, resulting in no net imbalance at higher speeds.

The paddle regions were defined to have a thickness of 1.4 mm, a height of 6 mm, and a length of 7.5 mm, providing a flat impact area of 45 mm². These values were chosen based on the spread of particles in prior microparticle impacts experiments and for mechanical stiffness during machining. Given these dimensions, the cross-sectional area of the remainder of the wing was calculated using the Marshall et al. equations. The rectangular cross section and aspect ratio of the paddle were maintained for the length of the wing. The final design of the rotor wings and complete rotor are shown in Figure 19. The theoretical maximum tip speed was calculated by increasing the rotor’s rotational speed until the tensile force equaled the yield strength of the material, resulting in ~1.7 km/s.
Figure 19. Rotor wing width and thickness profiles, complete design, and fabrication: a) rotor wing thickness profile (top right vertical view of the wing); b) rotor wing width profile (top right horizontal view of the wing); c) 3-D CAD model of the complete rotor prior showing the 4-wing design; and d) photograph of the completed rotor, mounted on only the bottom end of the shaft. The top mount was removed for visibility.

2.2.1 Modelling analysis of rotor performance

Rotor performance was modelled using finite element analysis. For an idealized rotor design (following the Marshall et al. equations), stress should be uniform throughout the rotor wing. Of course, uniformity of stress along the paddle would not be expected because the paddle
does not follow the idealized design. Similarly, shear forces and the wing junction at the shaft would also result in non-uniform stresses. The modelling results (Figure 20) agreed with these predictions. Higher stress was found in the transition area between the wing and the shaft and in the area between the paddle and the rest of the wing. In addition, 3-D dynamic simulations were conducted to identify mechanical resonances. Resonance occurred at 4 KHz which corresponds to 240,000 rpm, which is higher than the theoretical maximum velocity. The results from the material stress analysis showed a maximal stress of 580 MPa at a nominal speed of 150 krpm which is less than the 582 MPa stress limit (with 15 % margin).
2.2.2 Safety Considerations

Safety precautions to mitigate the risks of catastrophic failure of the high-speed rotor are essential. The impact force produced by a fragment of titanium rotor traveling at high speeds could be significant. For the present experiments a safety shielding was built around the rotor vacuum chamber using a combination of gravel and steel. The shielding was designed to safeguard the operators from the largest possible rotor fragment projectile traveling at 1.5 km/s using equations for projectile penetration. Due to the uncertain behavior of a rotating/spinning fragment, a conservative design factor was used.
2.2.3 Rotor Construction

The rotor was machined from a single piece of grade 5 titanium, which combines a high tensile strength over a wide temperature range with a moderately low density. The hollow ends of the rotor shaft were packed with magnets and the assembly was mounted between two magnetically levitated motors (CM-AMB#2300, Celeroton AG, Volketswill, Switzerland). A custom housing was built to maintain motor spacing and alignment. A magnetic bearing controller (CC-AMB-500, Celeroton) and software were used to control acceleration, monitor frequency, power, and bearing temperature. The rotor is oriented vertically and partially supported by a passive magnet to reduce axial bearing load while the stators are actively cooled. A Sorensen XTR 100-8.5 and BL Precision 1697B were used as power supplies. The rotor, including shaft and embedded magnets has a mass of 153 g.

A cooling system was implemented to dissipate the heat generated from the motors. A Julabo FC 1200T (Allentown, Pennsylvania, USA) was used during initial testing with a 1:1 glycol/water mix), whereas recirculating water was found to be sufficient and was used during subsequent experiments.

The rotor was operated under high vacuum to minimize drag forces and associated heating using a 17” custom vacuum chamber (MDC Vacuum, Hayward, California, USA). Initial rotor testing was performed at Celeroton at a pressure of $10^{-5}$ Torr, and additional rotor tests were done at Brigham Young University at $10^{-7}$ Torr. Two sets of pump systems were used to create high vacuum. The set used for initial testing included a diaphragm pump, (Divac 1.4HV3, Leybold, Cologne, Germany) and a 90 L/s turbo pump (90iX Turbovac, Leybold). Subsequent testing used a dry scroll pump (nXDS10i scroll pump, Edwards, Crawley, UK), and a 350 L/s
turbo pump (350i Turbvac, Leybold). All pressure readings were recorded using a Leybold TM9 thermovac gauge for monitoring rough vacuum and a Leybold PTR 90N Penningvac gauge for high-vacuum.

2.2.4 Testing

Assembling of the rotor and initial tests were done to verify the rotor’s rotational balance (both homopolar and heteropolar orbits of rotation in the x-y plane) and using the bearing controller to monitor bearing temperature at different rotational speeds. These tests were done at 35,000, 75,000, and 100,000 rpm (see Figure 21a, b and c).
Figure 21. Rotor’s experimental performance test at a) 35,000 b) 70,000 and c) 100,000 rpm. At left: the rotor’s orbiting performance in the x-y plane for both homopolar and heteropolar sides (the average center of the rotor shaft is depicted with a small “x”). At right: rotational speed measurements sampled at a frequency of 100 kHz for a) and b), and for 400 kHz for c)

2.2.5 Rotor Characterization Using a Laser

Subsequent experiments to characterize rotor performance, including a measurement of wing elongation were made by focusing a diode laser at the rotor wing tip and using a micropositioner to adjust the position of the laser spot (see Figure 22). These experiments used a
4.5-mW, 532-nm laser (CP532-Collimated Laser-Diode-Pumped DPSS Laser module, Round Beam, Thorlabs, Inc., Newton, New Jersey, USA). A silicon photodiode detector (SM1PD1A Type A, cathode grounded, Thorlabs) was used to detect laser light signal in connection with a DC reversed bias module. Other optical devices such as a neutral density filter, a 15-mm one-dimensional translation stage, lenses, and mirrors, were also purchased from Thorlabs. A 600 MHz oscilloscope (Teledyne LeCroy, Chestnut Ridge, New Jersey, USA) was used to process and digitize the detected signal. The data was analyzed using an algorithm written in python 3. A 350-point window moving average was performed to smooth out high-frequency noise in the signal. The signal was collected over a period of 100 microseconds.

The length of each wing was measured by aligning the laser with the edge of the rotor’s range of rotation and measuring the fraction of light blocked as each wing passed by that point. The micropositioner was used to scan the laser position from fully blocked to fully unblocked. The position at which 50% of the light was blocked gave the relative location of the tip for each wing, while measurements using the micropositioner, with increments of 5 micrometers, allowed accurate determination of this 50% position. This process was repeated at different rotor speeds ranging from 50 rpm (essentially at rest), from 35,000 rpm to 95,000 rpm in increments of 10,000 rpm, and at 100,000 rpm. This technique allowed measurement to within a few micrometers of 1) any non-uniformity in wing length from rotor fabrication or magnet mounting, 2) elongation (strain) of wings at different rotational speeds, and 3) anisotropy, if any, in the Young’s modulus between x- and y-wings. These measurements would also detect rotor imbalance, but with less sensitivity than measurements made during testing.
2.3 Results and Discussion

The test of rotational stability showed that the homopolar and heteropolar shaft positions are stable over a period of 30 minutes for 75 krpm and 100 krpm speeds respectively, although some wobble is present (see Figure 21). For the test at 75 krpm the deviation of the center of the rotor shaft from the center of the bearings remained within 25 microns, well within the design tolerance of 40 microns for both the homopolar and heteropolar ends. At the test pressure of 6 x 10^{-5} Torr, the motors dissipated 25 Watts (W) of power resulting in a temperature at the heteropolar and homopolar bearings of 35 and 63 degrees Celsius, respectively. For the test at 100,000 rpm the radial excursion was <30 microns. At a pressure of 6 x10^{-5} Torr this produced a
temperature in the heteropolar bearing of 44 degrees Celsius, while the homopolar bearing reached a temperature of 53 degrees Celsius. The temperature limit for the electronics is 60 Celsius. Hence, achieving higher speeds may require an improved cooling system. The temperature difference between the homopolar and heteropolar stators is due to differences in their functions. While both stators contain radial bearing windings, the heteropolar bearing also contains the motor winding, whereas the homopolar bearing carries the axial weight-bearing winding. The motor winding draws minimal power in steady state, which is the case for the testing vacuum environment. However, the axial bearing counteracts the weight of the rotor, thus drawing more power and producing more heat.

2.3.1 Laser Signal

In laser experiments to observe and measure the rotor during operation, as each rotor wing passed in front of the beam, a portion of the beam was blocked, yielding a signal as shown in Figure 23a. The resulting laser signal was converted into a percent transmittance. As seen in Figure 23b, the signal showed a recurring 4-peak pattern corresponding to slight differences in the physical lengths of each of the four rotor wings. We labeled each peak as A, B, C, and D, although there was no way to determine which specific wing was responsible for which peak in the pattern. The spectral data was processed by segmenting the signal in groups of four peaks starting with peak A as shown in figure 23b and then taking an average of the peak heights for each one of the letter peaks. This process was repeated as the laser light moved cross the rotor blade, scanning from 0 to 100% transmittance as shown in Figure 23a. Peak A showed a lower percent transmittance than the rest of the peaks while peak D showed the highest transmittance.
of the four peaks. Peak B and C transmittance values were relatively close together. Changes in this pattern at different rotor speeds likely indicate wobble of the rotor during operation.

Figure 23. Representative photodiode signal from laser characterization of the spinning rotor: a) the signal includes strong peaks representing obstruction of light by the tip of each rotor wing; b) the peak profile of light blocked by each rotor wing. At the top right corner is a visual representation of the peak labeling of the rotor wings that correspond to the peak signal.

As the rotational speed was increased during the characterization experiments, the centrifugal forces acting on the rotor arms also increased, causing slight elongation of the rotor wings. This elongation was measured as described earlier. Figure 24 shows the results of these experiments—the laser transmittance as a function of the micropositioner measurement. The relative position of the edge of each wing tip is accurately represented by the micropositioner reading at which 50% of the light is transmitted. Measured transmittance over the micropositioner range yielded a sigmoidal shape corresponding to the cross section of the focused laser. The sigmoidal curve fitting was performed using the experimental values in order to accurately determine the 50% location and hence the location of the edge of each wing tip.
Figure 24. Laser transmission measured over the range of displacements of the micropositioner. These data were used to calculate the physical position of the edge of the rotor wing tips—the relative position in each series was assigned to be the displacement value at which 50% of the laser light was blocked. Measurements of each wing (A, B, C, and D) and for two rotational speeds (50 rpm and 100,000 rpm) show variation among the wings at each given velocity, and also show a general shift between the two velocities corresponding to physical elongation (stretching) of the rotor wing material at the higher speed.

The total elongation for each rotor arm was 108.4, 115.8, 109.3, and 94.6 µm for peaks A, B, C, and D respectively. The calculated average elongation was 107.1 +/- 4.0 µm expressed as a standard deviation at 95% C.I. The differences between the four wings were not statistically
different—such a difference, if present, would have indicated an anisotropy in the elastic modulus of the rotor material between the x- and y- directions.

Young’s modulus can be calculated by combining the measured elongation with the tensile force along the length of each rotor wing. The wing elongation as a function of the square of the angular velocity (in rpm) is shown in Figure 25. The square of the angular velocity is proportional to the centripetal force acting on the rotor wings, ignoring the slight increase in centripetal force due to the elongation itself (as the material elongates, it sweeps a slightly larger radius and experiences a larger corresponding centripetal force). Thus, the results in Figure 25 represent a stress-strain curve for the rotor material (Grade V titanium alloy) and are expected to be linear to first order. A model was used to convert the slope of these data into Young’s modulus, considering the different cross section and different centripetal force at each point along the length of each rotor wing. The calculated value of Young’s modulus from this experiment is 116 +/- 10 GPa, in excellent agreement with the literature value of 114 GPa. The error bars in Figure 25 represent 95% confidence intervals in the measured elongation. Note that the first data point for peak D was somewhat off from the best fit line; however, this point did not fail the Grubbs test and was retained in the analysis.
Figure 25. Rotor wing strain or elongation as a function of the square of the rotational speed (with speeds ranging from 50 rpm to 100,000 rpm), shown for each of the four wings (A, B, C, and D). Data points correspond to the micropositioner measurement at which 50% of the laser light is blocked. Best-fit lines are shown for each wing. Displacement for all measurements is relative to the shortest wing at the lowest speed. Error bars represent the standard deviation at a 95% C.I. based on fitting the sigmoidal curves as in Figure 24.

2.4 Conclusion

In this report we have described a high-speed rotor intended for use in creating hypervelocity impacts of microparticles. In contrast to previous high-speed rotors, this design includes flat impact surfaces at the tips of each of the four wings, and those surfaces remain
normal to the velocity vector during operation. Preliminary performance of this rotor was
demonstrated by being able to spin stably under high vacuum at 100,000 rpm, with a
corresponding tip speed of 670 m/s. Higher speeds are theoretically possible based on the rotor
design and material, but may be limited by the efficiency of cooling the magnetically levitated
motor/bearing assemblies. The choice to use four 4 wings (rather than two as in prior rotor
designs) is a necessary consequence of the requirement to provide normal impact surfaces. With
only two wings, the addition of a mounting shaft along the rotational axis created an intermediate
rotational axis, leading to instability. During operation the rotor wings were observed to elongate
due to the centripetal force.

Whereas prior microparticle impact experiments have relied on electrostatic or
electrodynamic acceleration of charged microparticles, this rotor enables impact experiments
with either charged or uncharged species. Further experiments are planned in which aerodynamic
introductions of aerosols (such as with an aerodynamic lens) would introduce microparticles to
the rotor for impact. A limitation of using this rotor for impact studies is that impacts do not
occur at a fixed point in the laboratory frame of reference, making observation of the impact
process and analysis of the impact products more challenging.
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CHAPTER 3: MOLECULAR BEAM: AN INSTRUMENT TO COMPLEMENT THE HIGH-SPEED ROTOR

3.1 Justification

In addition to designing, building, and calibrating the high-speed rotor as previously discussed in chapter 2, I also worked on designing and constructing a molecular beam to work in conjunction with the rotor apparatus.

A justification for adding a molecular beam is that currently, as a standalone instrument, the high-speed rotor is not capable of producing enough collisional energy for studying hypervelocity microparticle impacts. The speed needed for chemical changes produced by a hypervelocity impact is above 3 km/s for space collisions. Molecular beams are capable of producing atomic velocity distributions of >2 km/s even if the molecular beam is seeded with another gas. For instance, Gorry and coworkers reached 2.2 km/s speed using a helium seeded oxygen atom beam. Similar seeded beam velocities have been achieved for bigger molecules while using helium as a seeded gas. Thus, assuming that momentum is conserved, the addition of the molecular beam velocity to the rotor’s rotational speed (~3 km/s) will allow sufficient collisional energy, in the space velocity molecular impact realm, for the experiment.

Another reason to include a molecular beam into our instrument design is that it can be used under controlled lab conditions that allow chemical detection. High vacuum allows for mass spectrometry detection which would be needed for high-sensitivity quantification during the experiment.

Most importantly, a molecular beam will allow the acceleration of neutral molecules. Because of its simple method of operation, permitting a gas to flow into high vacuum through an
aperture, molecular beams do not fundamentally use charging an analyte to induce acceleration. Neutrals, in contrast to ions, are of high astrobiological interest. Abundant neutral molecules are found in interstellar space, and their detection can lead to finding signs of life. For example, instruments like Cassini's INMS closed source were primarily designed to sample, thermalize, and mass analyze neutrals. Therefore, molecular beams present a suitable technique to study hypervelocity impact for neutral molecules.

However, molecular beams could be inconvenient to work with because of potential cluster formation. This could be a hindrance for studying high velocity impacts of individual molecules experimentally. When the beam is created, the low temperatures and collisions in the high-intensity region of the jet can cause dimer or even polymer formations especially for high-boiling samples, which could lead to bulk condensation; hence, the selection of a low boiling point analyte that would not only be of astrobiological interest but also have low tendency for clustering is desired. Therefore, clustering would present an important concern as it would obscure detectable chemical changes of individual molecules during the experiment.

Nevertheless, there are different ways a molecular beam experiment design may overcome clustering formation. One way to avoid dimer formation is by mixing the sampled gas with low concentrations of helium. The concentration of the helium carrier gas controls the degree of clustering. This makes it easier for the seeded gas to be expanded in a jet and can prevent/reduce dimer formation. Another alternative is to add heat to the system. The internal energy (temperature) of clusters and nanoparticles is a parameter that affects many of their properties as well as their processes. As a result, clustering can be prevented if the temperature is such to keep molecules in the gas phase and to impede individual molecules from interacting with each other to form dimers.
Other reasons for including a molecular beam in the experimental design are that it can be easily directed at a target, its intensity delivers concentrations that are detectable using mass spectrometry methods, and it can be built to meet spatial needs of the experiment. This last reason plays an important role in our molecular beam design due to restrictions in instrumental space inside the safety shielding.

After describing the reasons to support the addition of the molecular beam into the instrument design, the following section will present a brief historical review on the development and application of molecular beams. This will provide the background knowledge needed to construct a molecular beam that would satisfy the experimental needs of our proposed design.

3.2 Introduction

The first molecular beam experiments were carried out by Dunoyer in 1911. He developed the first directed neutral molecular beam experiments. He ran his experiments at such low pressures that the molecular collisions for most purposes were negligible. He used a glass tube divided into three separate compartments. Compartments 2 and 3 were kept under vacuum while in compartment 1 some sodium was introduced and heated until it vaporized. The gas traveled through the compartments forming a beam of particles. As a result, a deposit of sodium was produced and appeared in the third compartment. The deposit formed led to the idea that sodium molecules traveled in a straight line forming a beam through the evacuated chambers. This experiment using a pressure differential gave birth to the first molecular beam.

Soon after, in 1919, molecular beams started to be applied in planned scientific experiments. Stern was the first to start a series of experiments to use the newly developed
molecular beam technique to determine the thermal velocity of molecules. In his experiments, he approached the topic of gas dynamics using classic physics. Although the recently developed quantum theory proposed concepts contrary to classic physics, Stern kept studying these controversial topics using simple experiments involving classic methods. Stern's papers laid down many of the principles that govern molecular-beam techniques, which have been followed even to the present time. In the next decades, contemporary scientists expanded Stern’s work further and derived other important molecular beam experimental parameters. For example, Lubman and coworkers calculated the number of collisions per second a molecule makes with other molecules in an effusive molecular beam; Hurlbut measured and characterized the cosine distribution made by the flow of atoms created by a molecular beam, which is important in instrument design when aiming at a target; Gordon and Ponomarev studied the collisionless flow of gasses through a cylindrical tube in attempts to understand how tube size and shape affect flow. Later on, Lucas, using the theory of gas flow that had been developed throughout the previous years, published both experimental parameters and theoretical equations to build a molecular beam from the ground up. He described appropriate size of nozzles, nozzle length, vacuum pressure, etc. As a result of all the work done characterizing the behavior of supersonic jets, molecular beam instruments became a practical and straight-forward tool to perform experiments in controlled vacuum lab settings.

Since then, a myriad of extensive experiments involving molecular beams to study atomic properties, such as the behavior of individual species with electric and magnetic fields, have been done. More recently, a better understanding of the theory and the development of new instrument techniques has led to experiments involving the interactions between molecular beams with other beams, gasses, or lasers. Another important molecular beam application is the
studying of gas-surface interactions with the intent of examining the most fundamental level of chemical kinetics, intermolecular forces, and surface properties. For example, molecular beam epitaxy is a technique that uses molecular beams to study dynamics and kinetic surface reactions. It generates thin film deposition on surfaces, thus creating a clean coated surface. This technique has been applied to doping surfaces in the creation of new semiconductor materials. The different instrumental methods used by these techniques will not be discussed in this chapter because they target areas of chemistry that do not relate directly to the objective of this research.

Our research is interested in studying the interaction of a neutral molecular beam with a surface (rotor) that is traveling at high speed. The rest of the chapter will focus on describing the equations that govern the supersonic expansion of a jet as well as the parameters needed for building a custom neutral molecular beam. With this theoretical knowledge, a detailed description of the design, instrumentation used, and assembly of a custom molecular beam will be presented. Lastly, the performance results of the testing of the molecular beam will be discussed.

3.3 Design and Assembly

The addition of a molecular beam to the high-speed rotor introduced significant constraints to our experimental design. Due to safety concerns, a safety shielding was built around the rotor chamber. Even though the shielding is currently no longer in use, 4 years ago at the time the molecular beam was being built, there were significant instrumental space concerns to assemble both the rotor and the molecular beam within the shielding’s walls. The available space inside the shielding wall had a diameter of 32 in. The rotor chamber occupied 17 in. of the
available space, leaving only 15 in. left in which to fit a molecular beam. As a result, the molecular beam had to be compact enough in order to fit the restrictions in length.

Another important element of the molecular beam design was the speed of the gas expansion. Due to the limitation in the current speed of the rotor, it was crucial to obtain the maximum speed theoretically achievable of a molecular beam. In addition, the experiment required the ability to seed (introduce a molecule of interest inside a molecular beam of inert atoms) molecules of astrobiological importance inside the beam. Literature reported that seeded molecular beams are capable of accelerating analyte molecules to speeds greater than 2 km/s.\(^1\) Furthermore, the addition of heat to the supersonic nozzle of the molecular beam was reported to have significant increase in the speed of the particles.\(^{16}\) Therefore, the molecular beam design implemented a heating element to increase the temperature at the nozzle and a compartment where molecules of astrobiological importance could be seeded and transported by a carrier gas.

Lastly, the beam’s aim needed to be adjustable in order to be directed into the flat paddle region (impacting zone) at the tip of the rotor. This system needed to be able to deliver enough analyte material into the rotor chamber, such that after colliding with the rotor there would be enough products to be detected via mass spectrometry. The design presented in this chapter took into consideration these two important aspects.

The molecular beam’s design was developed using the theory, examples, parameters and equations of vacuum gas flow\(^ {17, 18} \) presented in *Building Scientific Apparatus*.\(^ 6\)

The first step in designing the beam was to determine the experimental constraints for the experiment. A major constraint was to determine the location of the Mach disc (direct shock wave of a supersonic jet) in the region(s) of the differential pumping calculate the optimal
pressures and pumping needed to meet these constraints. Append to this constraint is the beam size, which diameter has to be within the dimensions of the rotor’s paddle. As a result, these constraints will determine the pressures inside the chamber(s), the vacuum requirements (pump capacity and pumping speed), the nozzle(s) diameter for the expansion, and the appropriate skimmer size to narrow the beam’s intensity.

The behavior of the molecular beam depends on pressure. Our design required us to stay in the molecular flow regime or lower (>1 x 10^{-5} Torr). To match the pressure required, the theoretical calculations assumed a stagnant pressure of 650 Torr and a pressure inside the molecular beam chamber of 1.5 x 10^{-3} Torr. Because of pump availability, we chose the turbo molecular pump with the highest pumping speed of 275 L/s with an effective pumping speed (actual speed accounting for any conductance losses) of 200 L/s. In order to maintain the appropriate experimental pressure parameters, we needed to calculate a suitable nozzle diameter for the beam’s supersonic expansion. First, we needed to calculate what conductance would result in the desired experimental pressure (1.5 x 10^{-3} Torr). The throughput of a nozzle tube depends upon the conductance of that tube and the driving force. Additionally, operating at higher pressures gives some control over the flux of the beam and the throughput of the beam. Hence, we proceeded to calculate the vacuum system’s throughput, by the following equation:  

\[ Q = PS \]  

(1)

Where Q is equal to throughput, P equals pressure, and S is the pumping speed.

Then, we used this equation to calculate the nozzle’s conductance:  

\[ C = Q / (P_1 - P_2) \]  

(2)
Where $C$ equals conductance, $Q$ is the throughput, and $P_1$ equals the stagnant pressure (for our calculation is the pressure outside of vacuum), and $P_2$ is the background pressure (pressure inside of vacuum).

After conductance was determined, we calculated the nozzle’s diameter using a different conductance equation for molecular flow regime:  

\[ C = 15d^2 \]  

(3)

Where $C$ is the conductance, and $d$ is the diameter of the aperture.

Ideally, the pressure behind the gas should be such that the tube’s length is greater than the mean-free-path of the expansion, and the mean-free-path is greater than the tube’s diameter. The nozzle tube’s length was not a constraint in our design. However, as described by Moore in *Building Scientific Apparatus*, it could have had an effect in obtaining useful beam fluxes. Reasonable beam collimation occurs when the nozzle tube is at least 10 times greater in length than its diameter.

Once the gas starts exiting the nozzle, the interaction of the expanding jet gas and the background gas produces a shock wave surrounding the gas jet. The free expansion of the molecular beam gas occurs when the pressure difference between the background gas and the stagnant jet gas is enough to create a shock wave. This upstream of gas molecules behaves like a free jet expanding into a “perfect vacuum” and creating a mean-free-path of usable dimensions leading into an aperture. The aperture downstream with a diameter close enough to produce viscous flow is called a skimmer.

The nozzle’s distance from the skimmer is another parameter that was calculated for the design using the equation:
\[ L = 0.67d(P_0/P)^{\frac{1}{2}} \]  \hspace{1cm} (4)

Where \( L \) is the distance from the nozzle to the shock front represented by a skimmer; \( d \) is the diameter; \( P_0 \) is the pressure in the nozzle, and \( P \) is the background pressure.

In addition, the beam’s mean-free-path was approximated using this equation:\textsuperscript{6}

\[ \lambda = \frac{\sigma}{P} \]  \hspace{1cm} (5)

Where \( \lambda \) is equal to the mean-free-path and \( P \) is the background pressure.

Lastly, the spread of the beam was calculated using a cosine distribution and trigonometry to solve for theta by:

\[ \tan \theta = \frac{opp}{adj} \]  \hspace{1cm} (6)

Moore also suggests that depending on the molecular beam’s application, a gas flowing into a vacuum system through an aperture may produce a beam that is too broad to or too divergent. In this case a differential pumping system could be used where one or more apertures are placed downstream. Each chamber is evacuated with a separate pump. The pressure in the first will be highest, so that the large throughput obtained will significantly reduce the load on the next pump.

Because the beam needed to be precisely focused to a small area at the tip of the rotor, we decided to do a three-stage differential pumping to ensure the collimation of the beam. The calculated parameters previously explained were expanded to include the rest of the three-stage differential pumping. The calculated parameters of pressure, throughput, conductance, nozzle diameter, skimmer diameter, and distances between nozzle to skimmer and skimmer to skimmer
are shown in Figure 26. Additional chemical detection elements are also included which will be discussed in chapter 4 of this manuscript.
Figure 26. Elements of the molecular beam differential pumping system: sample tube, helium tank, Nozzle chamber, Skimmers chamber, Rotor chamber, xyz stage, rotor, pumps, gauges, Residual Gas Analyzer (RGA), Cryobaffle (CF), and Thermal Desorption Unit (TDU). Calculations parameters: pumping speed: pump 1 (Ps1), pump 2 (Ps2), pump 3 (Ps3); pressure (Pe), throughput (Q), conductance (C), nozzle diameter (Dm); skimmer diameters: skimmer 1 (Ds1) skimmer 2 (Ds2); distances: nozzle to skimmer 1 (Xs), skimmer 1 to skimmer 2 (Ds1-Ds2), skimmer 2 to rotor (Ds2-R), and mean-free-path (mfp). Values for each parameter are listed and were calculated using the vacuum equation of gas flow.
The next step in the design was to find the elements that would fit within the shielding space restrictions. For the vacuum stages, commercial Kimball physics vacuum chambers, a 6 in. spherical octagonal chamber and a 2.5 in. cube chamber, were purchased for the nozzle and cube vacuum stages respectively. The chambers were rested on an adjustable plate (see figure) to match the rotor’s height. Another dimensional restriction in the building of the molecular beam was the skimmer mounting and the appropriate skimmer-to-skimmer distance to achieve proper collimation. Two skimmers (0.3 mm and 0.5 mm inner diameter) were purchased from Beam Dynamics. Skimmers were screwed tight onto a cylinder using a custom holder piece. Likewise, the cylinder was assembled into a modified 2.5 in. double-sided flange. Both connections were screwed tight with an o-ring underneath for better a vacuum seal. The cylinder and holder pieces were completely customized and were used for the mounting of both skimmers see Figure 27).

Figure 27. Skimmer mounting. From top to bottom: skimmer, upper screws, skimmer holding piece, upper o-ring, cylinder, bottom o-ring, double sided flange, and bottom screws.
The custom cylinders were additionally lengthened to reduce the skimmer-to-skimmer distance to meet the calculated distance required for collimation. In addition, a bellows was incorporated into the design between the cube and the rotor chamber to control the direction of the molecular beam’s path as well as a resting base plate for easier handling of the instrument. A visualization of all the custom parts used in the assembling of the beam is shown in Figure 28.

Figure 28. Molecular beam custom pieces: a, modified double-sided flange; b, skimmer ring holder; c, cylindrical skimmer holder; d, T-shape xyz stage holder; e, nozzle holder; f, base plate; g, nozzle tip heater block; h, L-shape PEEK holder piece
Prior to physically assembling all the elements, the complete design was sketched using Autodesk Microsoft Inventor to ensure restrictions requirements were met see Figure 29. The combined length of all the elements added together was <14 in. which is within available shielding space.

Figure 29 Side view of the molecular beam setup. Top labeled elements: Nozzle, Skimmers 1 & 2, and Bellows. Bottom labeled elements (differential pumping system): Nozzle, Cube, and Rotor vacuum chambers. The total distance of all the elements assembled together was 13.98 in. which is within the available shielding space.

Once the differential pumping system design met the requirements in length, another important designing element was the speed and the seeding of the molecular beam. Experiments on the supersonic expansion of a gas indicate that seeded molecular beams of He and H₂ gas provide the highest molecular velocities due to their light molecular weight and high dilution ratios.⁴ A dilution ratio is the amount of analyte gas seeded in a carrier gas, which parameter may have an influence in the jet flow of the seeded molecular beam preventing clustering. Moreover,
literature suggests that the nozzle heating can increase the molecular velocity up to 4 km/s for molecular beams seeded with organic molecules. In addition, the amount of helium can control the degree of clustering in an expanding jet, reducing dimer formation. For these reasons, we opted for including a heating system in our molecular beam design and to use He as a carrier gas.

A heater block was designed to heat up the tip of the nozzle through contact with a cartridge heater (see Figure 30). In addition, a nozzle holder piece was designed to secure the nozzle and to attach to an L-shape piece that connected to a system of three piezo motorized linear stages. The x, y, and z stages (xyz stage) were mounted together using a custom T-shape holder to allow x, y, and z axis movement of the nozzle, with the purpose of facilitating alignment. Additionally, an insulating square piece made out of PEEK (polyetheretherketone) was placed between the stages and the L-shape holder to prevent the conductance of thermal energy coming from the heating of the nozzle into the motorized stage electronics. To prevent overheating (>60 Celsius) from happening, copper strips were also used to dissipate heat from the stages. The nozzle was connected to a heated capillary tube that led to a mixing gas chamber. Lastly, an LED light bulb was incorporated into the system for alignment purposes.

The xyz stage maximum reach was purposefully set to be an inch from the skimmer in order to prevent contact between the nozzle and the skimmer. The complete assembling can be seen in the following picture:
Figure 30. Top view of the nozzle chamber setup. Elements shown: Skimmer, Heater block, Nozzle, Capillary, Copper strips, L-Shape holder, PEEK piece, T-Shape holder, and xyz stage.

Additional elements were included in the design to introduce gas inside the vacuum, to provide energy to the xyz stage, to monitor the parameters of the system, and to add heat to the nozzle. A visualization of all the ports assembled is provided in Figure 31.
Figure 31. View of the nozzle chamber’s 8 ports. Port 1 holds skimmer 1 and connects to the cube vacuum chamber and eventually to the rotor chamber; Port 2 contains the electrical feedthroughs connections to outside of vacuum electrical supplies, from the inside of vacuum connects to the LED and heating systems; Port 3 holds a viewport to provide out-of-high-vacuum view of the system; Port 4 contains a cold cathode gauge to measure pressure; Port 5, holds an additional viewport; Port 6 connects to the stages’ controller from the outside, from the inside connects to the x,y, and z stages; Port 7 contains connections to a thermocouple from the outside, and from the inside, connects to the heating elements (rotor’s tip and capillary tube); Port 8 connects to the seeded gas chamber from the outside, and from the inside connects to the beam’s nozzle through a capillary.

The seeded gas chamber is divided into 4 gas lines or exits A valve connects to the helium tank. Another line connects to a vacuum pump to evacuate the system. In addition, a
different line is connected to a pressure gauge. A gas line connects to the nozzle chamber. A visualization of the seeded gas chamber is shown in figure 32.

Figure 32. Photo showing the location of the seeding chamber components and gas lines. Elements shown: valve, gauge, helium gas line, nozzle gas line, and pump line.
A complete assembly of the system is shown in Figure 33.

Figure 33. Molecular beam completely assembled showing the location of each of the components. Elements shown: Turbopumps, Gauges, Pressure controllers, Power supplies, Thermocouple, Rotor chamber, Cube chamber, and Nozzle chamber.

3.4 Results and Discussion

Preliminary tests were performed using helium gas. The pressure measurement results fortunately matched the calculated pressures from the theoretical design. Significant pressure changes occurred when moving the nozzle away from skimmer 1; specifically, the pressure in
both the cube and the rotor chamber dropped significantly. No pressure data was recorded due to a shift in the direction of the project’s immediate research goals. Future directions for the project will be presented in chapter 4 where further developments in the molecular beam will be discussed. Lastly, in order to assist future practitioners, a procedure on how to operate the molecular beam is introduced in Appendix B.
References


CHAPTER 4: FUTURE DIRECTIONS

4.1 Introduction

Chapter 4 is dedicated to the project’s future directions. Even though the rotor and the molecular beam have been assembled and are currently functioning, they have not been operated together yet. This chapter is intended to provide guidance on the next steps towards integrating both apparatuses as well as to introduce other experimental applications for the high-speed rotor. The beginning of this chapter will discuss the advances in the incorporation of analytical methods into the rotor experimental design for the chemical detection of rotor collision products. A discussion about the potential molecular candidates for rotor-beam impact experiments will follow. Subsequently, improvements to the current rotor design to increase its nominal rotational speed will be addressed. Lastly, other future experiments involving the use of a high-speed rotor will be presented.

4.2 Detection of Chemical Products from Experiments

Essential to the study of high velocity impact experiments is choosing the appropriate detection method that would allow us to identify any qualitative or quantitative changes to the impact species after a collision with the rotor. Analysis techniques will vary depending on the experiment and on the kind of product that is expected to be sampled from the experiment. The chemical nature and the quantity of products generated from the experiment will define the experiment’s required level of sensitivity, selectivity, resolution, and dynamic range. For instance, if the products produced from a hypervelocity impact are gas chiral isomers (mirror
image molecules that cannot be superimposed by any combinations of rotations) a chemical separation technique will be required in order to discriminate between chiral partners. In this case, the collision products will need the use of a chiral column to be separable via gas chromatography. In addition, a mass analyzer may be also required in order to compare collision products, searching for unique mass peaks between the impacted and unimpacted analyte collision counterparts.

A way to estimate the possible molecular outcome that may come from a hypervelocity impact rotor-beam experiment is by looking at the possible reactions that may occur to analyte when colliding with the surface of the rotor’s tip. Possible reactions that can occur are: bond breaking, racemization, isomerization, radical formation, and ion neutralization reactions. From these potential reactions at the moment of impact, we can expect some of the following chemical mechanism occurring: opening of a strained ring, which would be energetically possible because ring strain have low activation energy; sterically hindered rotation, for example starting with a pure enantiomer and ending with a scrambled rotation; bond breaking, which might produce the formation of radicals as a product of the bond dissociation of impacted volatile species; neutral loss, such as loss of CO₂, CO, although this might be unfavorable unless we start with an odd electron molecule or lose an H radical as in COOH; loss of a volatile neutral, for instance, if a radical becomes neutralized after the subsequent impact with the chamber; racemization or isomerization reactions, described by bond rotations within the molecule--this scenario will require gas chromatography methods with a chiral column; and lastly, the break of a host-guest complexes or breaking of hydrogen bonds. Lastly, because analyte molecules are introduced via a molecular beam, all analytes sampled by the detection technique are expected to be in the gas phase.
After discussing the chemical changes that can occur to impacted molecules, another important experimental aspect is to be able to detect chemical changes of impact molecules in the presence of their original unimpacted counterpart, assuming that not all the impacted particles react after a hypervelocity impact. Therefore, it is imperative that a suitable detection technique be able to distinguish and contrast any chemical changes when the rotor is on or turned off with the same sample being put in the chamber. Therefore, all these parameters play a role in choosing a detection technique.

In choosing an appropriate technique for future experiments, we would like to pick a technique that will be robust, allowing us to detect the vast majority of the previously proposed collision outcomes. Including a chromatography technique with chiral column properties into the set up would be vital as it will allow for enantiomeric and isomeric chemical separation and would bring high selectivity to the experiment. However, this analysis technique may not be available for both volatile and non-volatile collision products. In addition, a mass spectrometer with high resolution, high sensitivity, and increased dynamic range will be needed in order to identify even the smallest quantities of rotor collision products.

Therefore, in order to be able to detect all these possible post collision chemical scenarios, we have designed and have partially built a multi-step chemical detection system. First, knowing that hypervelocity collisions would occur inside the rotor vacuum chamber, we would like to introduce an analytical instrument to allow us to detect any products of unique mass peaks. These would include products from hypervelocity impact chemical reactions such: neutral loss, ion neutralization reactions, and any other type of bond dissociation: breaking of a hydrogen bond, opening or a strain ring, and/or dissociation of atomic bonds. For detection of volatile products for the experiment, we decided to include a residual gas analyzer (RGA) to
detect these types of chemical products. RGA probes use a quadrupole mass filter analyzer with a maximum commercially available mass range of 300 amu, offering a better than 1 amu resolution, which is desired to discriminate between and identify potential species. In addition, its large dynamic range will allow simultaneous detection of small and large gas concentration. Lastly, RGAs operate at partial pressures ranging from $1 \times 10^{-5}$ Torr to $1 \times 10^{-14}$ Torr, which is within experimental parameters. A possible limitation of using an RGA is its short mass range because it can limit the hypervelocity experiments to molecules of molecular mass lower than 300 amu; however, this could be overcome by designing an experiment that would take that into account by using small mass analytes. On the other hand, non-volatile hypervelocity collision products will tend to stick to the walls of the chamber, hence, an RGA quadrupole mass filter will not be able to detect them. In this case, applying heat to the chamber to induce thermal desorption or outgassing could be a way to sample all non-volatile products. Another way to sample non-volatile products would be to use sticky paper mass spectrometry on the walls of the chamber; however, this technique hasn’t been fully developed yet, thus it might compromise the quantification of chemical species.

In addition, chiral collision products will produce the same fragmentation pattern, thus, an RGA detector will not be able to discriminate between isomeric molecules. Therefore, we decided to include a GC-MS detection system, where any unreacted volatile products will be captured, using a cryotrap, prior to separation analysis. We designed a cryobaffle system that would trap any unreacted parent products to its walls by cooling down the walls with liquid nitrogen. Both reacted and unreacted material is expected to stick to the walls of the cryobaffle. The system was designed to be isolated from the rest of the high vacuum system by mechanical valves. The proposed future operation of the cryobaffle after trapping involves bringing the
cryobaffle back to atmospheric pressure and heat it between 80 to 120 degrees Celsius depending on the boiling properties of the compound being studied. During the heating process the volatile trapped material will be released from the walls of the cryobaffle where a flow of nitrogen will carry out of the vacuum chamber and store it into a thermal desorption tube. Meanwhile, the nitrogen flow inside the cryobaffle will bring the cryotrap up to atmospheric pressure. Lastly, a thermal desorption sampling tube will be introduced into a chromatography instrument, which has already been purchased, where the post-collision products will be analyzed and quantified using GC TOF-MS. An overview of the complete step-by-step experimental design is shown in Figure 34. In addition, the figure also describes the types of collision products and expected analytical signal using our detection system for a chiral molecule, a cis 1,2-dimethyl cyclopropane.
Figure 34. Schematic showing the step-by-step proposed molecular chemical detection system after a hypervelocity impact, using cis-1,2-dimethyl cyclopropane as an example. 1. Differential pumping system where hypervelocity collisions occur, and products are expected to be formed. The example molecule, a parent cis-1,2 dimethyl cyclopropane molecule, is expected to turn into its isomeric form, a trans-1,2 dimethyl cyclopropane molecule, after a hypervelocity impact. Two detection paths are shown: one is marked by a red arrow leading to the RGA detector labeled as step 2a. Expected spectral products for the parent molecule are also shown. Because the parent molecule is a rotamer isomer (bond-rotation isomer) the spectral products produced by a hypervelocity impact are indistinguishable. The “separation” path is marked by a blue arrow leading to the cryobaffle. GC-MS detection system labeled as step 2b. The sample is trapped in the cryobaffle and collected using a thermal desorption tube unit labeled as step 3. The sample is then transported using the thermal desorption unit into the GC labeled as step 4. Step 5 is where chemical separation of the products occurs. A chromatogram of the separation of the products the cis-1,2 dimethyl cyclopropane and trans-1,2 dimethyl cyclopropane is shown. Lastly, step 6, shows the quantification of the products using Time of flight Mass Spectrometry (ToF-MS) after chemical separation has occurred.
A physical assembly of the RGA, mechanical valves, cryobaffle, thermal desorption unit, GC-TOF instrument, and turbopump is also shown in Figure 35.

![Image of the complete assembled detection system]

Figure 35. The complete assembled detection system. Top to bottom: Turbo pump, Upper Gate Valve, Cryobaffle, Desorption Tube Unit, Lower Gate Valve, Residual Gas Analyzer, and Vacuum Chamber. Top left corner the GC-TOF-MS apparatus is shown.

Even though the molecular detection system is physically assembled, it hasn’t been used for molecular high velocity impact experiments yet. There are further experimental steps to be determined in order to ensure the experiment’s success. One important element to consider for future rotor-beam experiments is to determine an appropriate analyte molecule of low activation energy and capable of being seeded using a molecular beam. The next section will present
possible suitable molecules that may be seeded in a molecular beam and produce a measurable
chemical change after a collision with the Rotor Induced Collision Cell. In addition, further
designing steps to complete the molecular beam’s seeding chamber system will also be
discussed.

4.3 Completion of the Molecular Beam Assembly and Candidate Molecules for Seeding Experiments

A very important step towards the completion of the Rotor Induce Collision Cell experiment is to determine what gas analytes are suited for high velocity impact experiments. In choosing a compound for future rotor-molecular beam impact beam experiments (whack-a-mole), there are some important factors to consider ensuring the experiment's success. A sequential explanation behind the rationale in choosing an appropriate analyte for the experiment is presented next.

First, the dissociation energy threshold needs to be low enough that the rotor impact will cause a chemical change. Using a purely classical physics approach, the dissociation energy from impact would be directly proportional to the kinetic energy associated with the impact (collisional energy), which is influenced by the molar mass of the projectile molecule and the speed of the impact by the kinetic energy equation:

\[ E = \frac{1}{2} mV^2 \]  

Where \( E \) equals the kinetic energy, \( m \) is the mass of the object, and \( V \) is the velocity of the object.
However, experiments with ions have shown that not all the collisional energy from a single impact goes into dissociation. The collisional energy is distributed into the molecules' chemical bonds causing vibrational excitation. Therefore, if enough collisional energy is produced from an impact, vibrations within the bond could lead to bond rotation, bond rearrangement, and bond dissociation. For our selection of molecules, based on literature we assumed that only 12% of the collisional energy went into the vibrational modes of the molecule; however, this percentage may not be applicable to all potential analyte molecules as the percentage may change depending on the number of bonds within the molecules as well as the type of chemical bonds.

Second, the analyte needs to be suitable for molecular beam seeding experiments. One important consideration is that the analyte must be and stay in the gas phase at the moment of being seeded into the beam and perhaps throughout the experiment. A volatile compound with a low vapor pressure point is preferred because it will allow for greater amounts of analyte to be combined with a molecular beam carrier gas. However, it might also be possible to start with a solid or liquid compound and heat it until it starts to vaporize.

The third consideration is finding molecules of astrobiological relevance for rotor impact experiments. Molecules of astrobiological significance are those that could be the precursors of biological processes. This includes amino acids, fatty acids, carbohydrates, dipeptides, and cyclic organics. In previous experiments performed in our lab we created a stream of glycine and arginine gas using a Knudsen cell (a method to create an effusive beam of a sublimated solid). Literature shows that amino acids such as tryptophan have been used in supersonic molecular beams with the effect of cooling it down for spectroscopic purposes.\textsuperscript{1-3} Testing molecules of astrobiological importance may not be feasible for rotor-molecular beam experiments at the
moment because of their high activation energy of dissociation threshold unless a lower activation energy change such as bond rotation occurs. However, upcoming modifications to increase the rotational speed to our current rotor design may resolve this complication.

The fourth consideration is the nature of dissociation products and chemical detection. Possible dissociation products for the experiment have been previously discussed in the chemical detection section of this chapter. However, other low-barrier compounds may generate products that can only be detected spectroscopically and not chemically; for example, through chemiluminescence.

Initially, we sought to produce bond dissociation reactions from rotor impact experiments. However, due to the reduction in the expected rotor’s rotational speed, we investigated other alternative reactions that would produce detectable chemical changes. For future rotor molecular beam experiments, we suggest the use of rotamers. Rotamers are compounds that contain at least one molecular bond that can rotate, in most cases forming a mirror image of the original molecule. The activation energy that goes into the bond rotation of a functional group can go as low as ~0.07 eV of energy for hydrocarbon chains of two carbons. For example, 1,2-dichloroethane (CH₂C-CH₂-Cl) has three different local minima that correspond to different bond rotations: the first, where the two chlorine are on the same plane as the two carbons but with oppositely directed bonds; second, mirror images of each other; third, the two -CH₂-Cl groups are rotated about 109 degrees from their mirror image position, with energies of rotation of 0.06, 0.22, 0.34 eV respectively. Therefore, there is enough collisional energy in a rotor-molecular beam impact experiment to produce these types of bond rotations. However, there is no astrobiological importance in these compounds, but they can be used as a
proof on concept for future experiments. In addition, only chromatography would be able to chemically separate these types of isomers.

In the future, when improved rotor designs with increased the rotational speed are tested and built, we might be able to test a greater variety of molecules. There are a handful of gas molecules that have been used for impacting experiments compatible in nature to our proposed experiment in which chemical changes were measured. These molecules could also be used for rotor-beam impact experiments. A table of such molecules is presented in Table 2.
<table>
<thead>
<tr>
<th>Authors</th>
<th>Molecules of Interest</th>
<th>Threshold Energy Range</th>
<th>Surface</th>
<th>Dissociation Probability</th>
<th>Products</th>
<th>Astrobiological importance</th>
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<td>Diamond</td>
<td>4% at 8 eV</td>
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<td>Connolly, M. S., et al.&lt;sup&gt;7&lt;/sup&gt;</td>
<td>Cl&lt;sub&gt;2&lt;/sub&gt;CCHCl&lt;sub&gt;2&lt;/sub&gt;</td>
<td></td>
<td>Glass</td>
<td></td>
<td></td>
<td>N/A</td>
</tr>
<tr>
<td>Connolly, M. S., et al.&lt;sup&gt;7&lt;/sup&gt;</td>
<td>CCl&lt;sub&gt;4&lt;/sub&gt;</td>
<td></td>
<td>Glass</td>
<td></td>
<td></td>
<td>N/A</td>
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<tr>
<td>Connolly, M. S., et al.&lt;sup&gt;7&lt;/sup&gt;</td>
<td>C&lt;sub&gt;2&lt;/sub&gt;Cl&lt;sub&gt;6&lt;/sub&gt;</td>
<td></td>
<td>Glass</td>
<td></td>
<td></td>
<td>N/A</td>
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<tr>
<td>Brown, James Corgan, and Michael Menzinger&lt;sup&gt;a&lt;/sup&gt;</td>
<td>Tetramethyldioxetane</td>
<td>1.25 eV</td>
<td>Xenon</td>
<td>Collision energy 1-4.6 eV</td>
<td>Light from acetone excited singlet state</td>
<td>N/A</td>
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<tr>
<td>Bottari, F. J.; Greene, E. F..&lt;sup&gt;5&lt;/sup&gt;</td>
<td>Tetramethyldioxetane</td>
<td>1.25 eV</td>
<td>SS, glass, polyethylene, polyimide, poly (vinylidene chloride), polyethylene terephthalate, paraffin</td>
<td>1.70 eV acceleration</td>
<td>Light from acetone excited singlet state</td>
<td>N/A</td>
</tr>
<tr>
<td>Bechara, Etelvino JH, and Therese Wilson&lt;sup&gt;10&lt;/sup&gt;</td>
<td>Tetraethylldioxetane</td>
<td>1.28 eV</td>
<td></td>
<td></td>
<td></td>
<td>N/A</td>
</tr>
<tr>
<td>Bechara, Etelvino JH, and Therese Wilson&lt;sup&gt;10&lt;/sup&gt;</td>
<td>Cyclohexyldiethyldioxetane</td>
<td>1.58 eV</td>
<td></td>
<td></td>
<td></td>
<td>N/A</td>
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<td>Bechara, Etelvino JH, and Therese Wilson&lt;sup&gt;10&lt;/sup&gt;</td>
<td>3,4-dimethylid-N-dioxetane</td>
<td>1.07 eV</td>
<td></td>
<td></td>
<td></td>
<td>N/A</td>
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<tr>
<td>Farneth, William E., and Douglas G. Johnson&lt;sup&gt;11&lt;/sup&gt;</td>
<td>2,2-dimethyl-3-ethoxyxetane</td>
<td></td>
<td></td>
<td></td>
<td>Weaker emission due to acetone</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Chemical Structure</td>
<td>Energy Level</td>
<td>Additional Information</td>
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<td>Farneth, William E.,</td>
<td>2-acyl-3-ethoxy-2-methylxetane</td>
<td>1.08 eV</td>
<td>Electronically excited biacetyl</td>
<td>Douglas G. Johnson</td>
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<td>Ryang, Hong-Son,</td>
<td>3,4-diethoxy-1,4-dioxetane</td>
<td>1.08 eV</td>
<td>N/A</td>
<td>N/A</td>
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<tr>
<td>Kensuke Shima, and</td>
<td>Adaman HLideneadamantane-</td>
<td>1.75 eV</td>
<td>1,2-dioxetane CL below 75 deg C, this compound</td>
<td>Hiroshi Sakurai</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hiroshi Sakurai</td>
<td>1,2-dioxetane</td>
<td></td>
<td>150-175 deg C</td>
<td></td>
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<tr>
<td>Meijer, E. W., and</td>
<td>1,2-dioxetane (synthesis)</td>
<td></td>
<td>N/A</td>
<td>Hans Wynberg</td>
<td></td>
<td></td>
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<tr>
<td>Wilhelm J. Baader</td>
<td>Sm+ N2O, Ba+ N2O</td>
<td></td>
<td>N/A</td>
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4.4 Improvements for Future Rotor Designs

Based on our own experimental rotor tests previously discussed in Chapter 2, we discovered two interconnected drawbacks that prevented us from increased rotor rotational speed. These were: shape and material, which both have a direct impact on the heating of the electronics.

Initially, our design involved a two-winged rotor with flat paddles located at the tip of the wings. When tested at 100,000 rpm, this rotor prototype got off its axis of rotation and catastrophically failed. The failure was the result of nutation caused by spinning the rotor around its intermediate axis of rotation. This axis of rotation is defined as unstable and has the tendency to flip the rotational object 180 degrees around its center point (for information about the intermediate axis theorem see chapter 2). In order to prevent this, this led us to design a 4-wing rotor in order to reduce the symmetry of the rotational axis and thus reduce the tendency for nutation. In addition, the cross-sectional areas across each wing were optimized using Moon’s equation. However, the addition of wings increased the overall rotor mass, which increased the heat of the bearing and the electronics due to friction during performance. A four-blade rotor with paddles at the tips of its wings is the only design that has currently been tested in our lab.

The properties of the material with which the rotor was built also play an important role in designing future rotor prototypes. A material high in tensile strength that holds its hardness at high temperatures and low thermal conductivity is preferred. Two materials were proposed: titanium and aluminum. Even though some aluminum alloys can reach very high material strength compared to some steels, titanium was chosen above aluminum because it had higher tensile strength and less plasticity at high temperatures. After testing the titanium rotor, we found
that bearing cooling system was able to keep a temperature of less than 60°C. The low
temperature allows for testing of less dense material such as aluminum which could potentially
achieve higher rotational speeds compared to the titanium rotor and produce less heat because of
reduced overall mass. An aluminum rotor is currently in plans of being tested using our rotor
rotational system.
References


APPENDIX A

Operational Instruction

1. Turn on the vacuum until you reach a pressure close to $1 \times 10^{-5}$ Torr. The system vacuum is operated by turning on the 10i Scroll pump until the pressure reaches a pressure close to $1 \times 10^{-2}$ Torr. Subsequently, the turbo pump can be turned on till high vacuum is reached. The maximum pressure obtainable is $1 \times 10^{-7}$ Torr.

2. Cooling system. Turn on the recirculating water lines. There are two of them. Open the lines just enough to where you can see that water is circulating. Make sure that when you open them do not open all the way. In the past, the pressure has broken the lines. In addition, a water leak inside the vacuum would destroy the turbo pump.

3. Rotor power supply. Turn on the BK precision power supply. It is set to 40 V 5 Amp; the settings are already saved on the apparatus. The prefix settings are displayed by pressing on the shift enter button together followed by pressing on the enter button again. This turned on the rotor’s controller box.

4. Rotor Software. Open up the celeroronPilot V4.1. Open up the screen Basic control. Before you start operating the rotor make sure to press the “Levitate” button. Do this a couple of times to make sure the levitation is working properly. You should see changes in the actual speed box when the rotor has been levitated correctly. Then select the speed for your experiment. Note, the pressure in the vacuum system will go up as you turn on the rotor the heating on the material may be the cause of some outgassing inside the vacuum. Monitor the rotor’s temperature closely do not let it passed the 60 celsius mark.
Optics

5. Laser and optics. Turn on the green laser by pressing on the turned on button located on the power supply. Make sure the laser is aligned, if the rotor is working and the laser is properly aligned you should be able to see a pulsed signal.

6. Photodiode detector. Make sure the photodiode is properly aligned to the laser path and that the photodiode is connected to an oscilloscope with a 50 Ohm coupling.

7 Oscilloscope. Make sure to have these settings on the scope when you operate it. DC IMQ coupling. Offset -402.0 mV. Roll 1.000 s/div and a vertical scale of 20 mv/div. You will need to adjust the time knob to be able to detect the signal pulses at high rotational speed.

8. Save the Data. Go to the file. Click on save the waveform. Give the file and Name (speed in rpm-displacement micropositioner-Date). Make sure you save the Data in the correct directory. Then click save. Make sure you wait a little in between saving files for the oscilloscope to have enough time to start to detect any changes in the experiment like increasing the rotational speed or changing the displacement. It takes about a minute or two.

Turning it off

1 Start by turning the rotor off. You will see the peaks in the signal starting to disappear. Press the touch down button to stop the rotor from levitating. Close the Celeroton software

2. Close the recirculating water cooling lines

3. Turn off the power supply.

4. Then turned the laser off.
5. You can leave the oscilloscope on or turn it off as well.
APPENDIX B

1. Turn on the nozzle chamber’s 10i Scroll pump until you reach a pressure close to $1 \times 10^{-2}$ Torr. Subsequently, turn on roughing pumps of the cube and the rotor vacuum chambers. Let them run until the pressures are equalized in all three chambers. Starting with the nozzle chamber again, turn on the turbopump, the pressure should be roughly around $1 \times 10^{-7}$ Torr. If the pressure values do not match the expected values check for leaks in the system. After the expected pressure in the nozzle chamber has been reached, turn on the cube and rotor chamber turbopumps. Let the system run until the pressure equalizes in all three chambers.

2. Log into the computer using the Austin’s Lab Username. The password is NASA. Click on AG-UC2 found on the computer’s desktop. The pop up interface window will open automatically. Click on the COM4 box and then click on the button labeled as launch applet. Click on the main tablet located on top. This will show the xyz stage control display. The current channels # display controls the movement of the stage in the x, y and z directions. The Jog controls the step speed; it controls the movement of the stage. The green light means the instrument is working correctly. If the light turns red restart the system. Locate the stage close to the skimmer’s mouth.

3. Open the helium gas tank line. Make sure you see a ride in the pressure meter. Then open the exit valve located the closest to the copper tube line. When ready to run the beam, open the valve that is connected to port 8 of the nozzle chamber.

4. If desired, turn on the power supply label as LED light for better visualization inside the chamber. The capillary heater has already been assembled into a power supply.
Turning it off

1. Start by closing the helium valve. You will see the pressure start going back down to approximately $1 \times 10^{-7}$ Torr.

2. Let pressure equalize then, turn off the rotor, then the cube, then the nozzle chamber in that order, allowing for equalization of the pressure of the system every time a pump is turned off.

3. After pressure has been equalized to $1 \times 10^{-3}$ Torr, start turning off the rough vacuum pump in the same order.

4. Lastly, disconnect from the xyz stage controller.
Feedthrough connections

Figure 36. Diagram showing the vacuum feedthrough connections of the XYZ stage and LED light