

Silicon carbide nozzle for producing molecular beams

Edward L. Patrick

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Silicon carbide nozzle for producing molecular beams

Edward L. Patrick^{a)}

Science Systems and Applications, Inc., 10210 Greenbelt Road, Suite 600, Lanham, Maryland 20706

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A resistively heated nozzle of silicon carbide (SiC) ceramic was developed for the production of high speed molecular beams for space environment simulation. The nozzle is able to withstand temperatures in excess of 1800 °C and pressures to 30 bars. In a seeded beam of 1% argon in 99% hydrogen, a speed of 3.9 km/s was achieved for the argon component at a nozzle temperature of 1150 °C and a pressure of 23 bars.

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I. INTRODUCTION

Molecular beams have many applications today as diagnostic tools for chemical kinetics, mass spectrometry, semiconductor processing, surface physics, and space environment simulation. Space environment simulation, however, often requires high density beams of high speed gas to approximate the flight conditions encountered by a spacecraft skimming the outer atmosphere of a planet, moon, or comet. Such flow conditions are orders of magnitude higher than those necessary for the delicate work of crossed-beam experiments, and introduce flux problems related to both beam speed and particle density.

A body in low Earth orbit moves at approximately 8 km/s. Upper atmospheric research is one example of where a molecular beam machine (MBM) can be a useful tool for calibrating space flight instrumentation by simulating the local space environment.^{1,2} Simulating that environment for the joint NASA/ESA Cassini/Huygens international mission to Saturn motivated the work described in this article. Orbital encounters by the Cassini spacecraft with the Saturnian moon, Titan, will be in the range of 6 km/s. Although charge exchange and other molecular beam methods can produce neutral beam speeds far in excess of these two cases,³ the density of such beams is characteristically low, particularly in the energy region (<10 eV) of interest to space environment simulators.

II. BASICS OF A COMMERCIAL MOLECULAR BEAM NOZZLE

A. Nozzle operation

To achieve high speeds and densities in a molecular beam, a “free jet”⁴ expansion is used. In the free jet expansion, a high density gas expands through a nozzle into a vacuum where most of the beam gas is evacuated, except for a needle-thin, central portion created by passage through a skimmer into the second stage of the MBM (Fig. 1).

In a typical nozzle, a tiny orifice is drilled into a metal disk, and the disk is welded onto the tip of a metal tube. Any metal suitable for operation in a vacuum system can be used,

however, the usual nozzle material is a refractory metal capable of withstanding the high temperatures necessary for increasing the thermal speed of the gas.

A free jet nozzle converts random thermal motion into a one-dimensional flow.⁴ At room temperature, the speed of hydrogen gas molecules escaping from a nozzle orifice (2 km/s) is higher than the mean thermal speed of the hydrogen molecules (1.4 km/s) in a stagnant volume.

From basic thermodynamics, the lightest gases (e.g., H₂ and He) produce the highest thermal speeds, and hence, the highest corresponding beam speeds. In theory, by “seeding” a light gas with a heavier gas molecule, the “seed” will be carried along at a speed closer to that of the carrier gas. This higher speed significantly increases the energy for the seed molecule above that achieved by the carrier gas alone.⁵⁻⁷ The maximum speed obtainable, however, is also a function of the mean molecular weight of the gas mixture. This imposes an upper limit on the speed and a practical limit on the concentration of the seed gas. Furthermore, the seed gas also experiences “slip” in the streaming gas, because collision effects rapidly vanish in the collision-free region downstream of the nozzle orifice. In practice, this prevents the seed molecules (or atoms) from acquiring their maximum theoretical speed.

One of the scientific instruments aboard the Cassini spacecraft is the ion and neutral mass spectrometer (INMS). Part of the mission of the INMS is to perform *in situ* measurements of the upper atmosphere of Titan during Cassini fly-bys of that Saturnian moon.^{8,9} To simulate the conditions INMS is anticipated to experience at Titan, a commercially available molecular beam nozzle was purchased from Thermionics Laboratories for the purpose of producing high speed, high density molecular beams.

B. Commercial nozzles

The Thermionics nozzle was composed of a 0.635 cm diameter sintered molybdenum tube with a molybdenum electron microscope objective that was electron beam (EB) welded to the tube tip. About the nozzle end of the tube was a length of alumina tubing that electrically insulated the molybdenum tube from an outside heater tube, 10 cm in length, of Hexoloy brand silicon carbide (SiC) manufactured by Car-

^{a)}Electronic mail: edward.l.patrick.1@gssc.nasa.gov

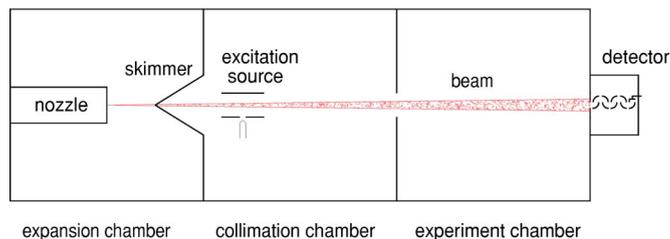


FIG. 1. Diagram of a molecular beam machine (MBM) with an excitation source.

borundum Corporation. Electrical connections to the SiC tube were made by wrapping the tube on both ends with a graphite ribbon. The graphite couplings were clamped by metal bands that tightened about the tube, and also provided an attachment point for copper electrodes that supplied the electrical power. A diagram of the assembled Thermionics tube is shown in Fig. 2, where (a) shows the nozzle ready for operation in vacuum and (b) shows the nozzle assembly with the coolant shroud removed.

A coolant shroud encompassed the nozzle to reduce the amount of heat exposure to the associated vacuum chamber. The shroud, a copper cylinder 5 cm in diameter, and about 10 cm long, with a wall thickness of 0.168 cm, was brazed to a copper coolant line that coupled to feedthroughs using Ca-jon VCR fittings. This permitted easy removal of the shroud to expose the nozzle beneath.

C. Nozzle modifications

Although the original Thermionics configuration kept the nozzle chamber cool, the shroud effectively robbed the nozzle of reflected heater power. To address this, tungsten shielding was introduced as in previous heated nozzle applications at the University of Toronto¹⁰ and the University of

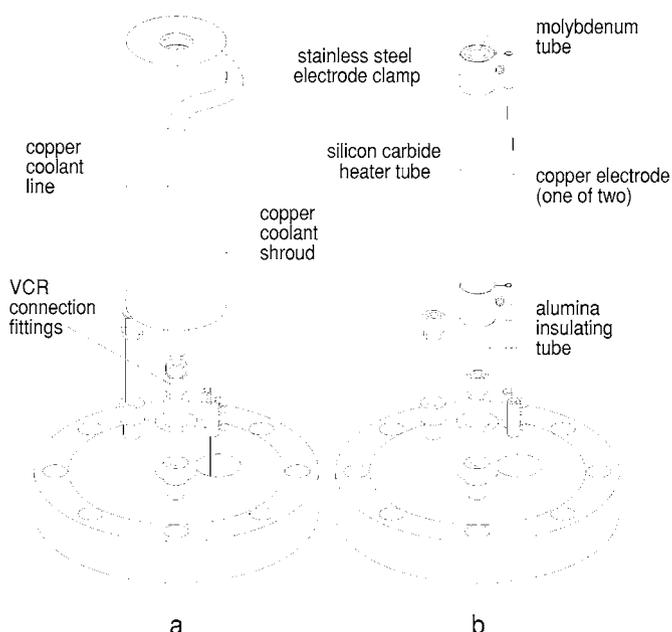


FIG. 2. A commercially available molecular beam nozzle manufactured by Thermionics Laboratories, ready for operation in a vacuum system (a), and with the coolant shroud removed (b).

Minnesota.¹¹ The shielding was made from dimpled tungsten foil that was rolled into a spiral, and then inserted into the coolant shroud. The shroud slipped over the nozzle and was fastened securely by the coolant line connections and by set screws on brackets integral to the shroud through which a guide rod was inserted.

After adding the tungsten shielding to an effective depth of 12 layers, the heater produced the same temperatures as in its original configuration, but at a third of the required power.

D. Operational limits

Once the copper electrodes on the commercial nozzle were replaced, temperature of 1000 °C was routine. In fact, operation was reliable at temperatures up to 1400 °C for hundreds of hours. Above this temperature, aluminum formed in the intervening space between both of the ceramic tubes, and shattered the outer (SiC) tube upon cooling. This was believed due to the reduction of alumina (Al_2O_3) in the presence of hydrogen at high temperatures.

Another difficulty with the commercial nozzle was a “sagging” of the nozzle tube due to a combination of the nozzle weight and its increased elasticity at higher temperatures. Despite the refractory metal construction, heating of the molybdenum tube caused it to sag by an additional amount each time the temperature was increased. These thermal deformations required frequent adjustment of the nozzle, and made an x - y - z manipulating stage an essential part of normal nozzle operation.

III. SILICON CARBIDE NOZZLE DEVELOPMENT

A. Design parameters

Since the SiC ceramic tube was the heat source for the commercial nozzle, fabricating a nozzle completely out of SiC seemed practical. This would reduce the droop seen in metal nozzles at high temperature, simplify the mechanics of the nozzle, and more efficiently transmit heat to the gas.

Producing a SiC molecular beam nozzle required that the closed-end tube have an appropriate resistance per unit length, an ability to be joined to metal, good vacuum/pressure integrity, and an ability to accept a machined orifice of small size.

All four of the above criteria had to be met to produce a SiC nozzle that had practical merit in the laboratory.

1. Electrical resistance

The first question was whether or not suitable tubes of Hexoloy SiC could be produced. This brand of SiC was chosen because of experience using it as a heater for the commercial molybdenum nozzle. Although the cylindrical SiC heater on the Thermionics nozzle had an outside diameter (o.d.) of 1.25 cm., this diameter was necessary to encompass both the 0.625 cm o.d. molybdenum tube and the 0.938 cm o.d. alumina insulating tube. By selecting a smaller tube diameter, the profile of the complete nozzle assembly would be smaller and permit the insertion of additional layers of shielding within the coolant shroud. As a result, smaller SiC tubes with o.d. of 0.938 cm were purchased from the inven-

tory of sizes available from Carborundum Corporation. All of these tubes resemble a test tube as they are fabricated with one end closed.

Electrical conductivity varied considerably between Hexoloy SiC tubes, as they are not manufactured with a view toward controlling their electrical properties. Consequently, about half of the tubes that were purchased exhibited resistances in the range of $10^6 \Omega/\text{cm}$. To function correctly, the tubes needed to draw enough current to become hot, and a resistivity of 10–300 Ω/cm was deemed appropriate to permit the tube to operate with the existing high-current power supply.

2. Pressure integrity

Hexoloy tubes are commonly used as thermocouple protection tubes, so one might assume that they possessed a reasonable degree of vacuum and pressure integrities. However, since Hexoloy is manufactured in a reaction-sintered process using micron-sized SiC granules, there was a question as to the microscopic homogeneity of the material. Small voids in the ceramic could conceivably compromise vacuum and pressure integrities. To verify this, tubes were selected with a high degree of roundness ($\pm 0.05 \text{ cm}$) and secured to a leak detector with Swagelok components using nylon ferrules. The nylon ferrules achieved a good seal against the ceramic, and leak testing revealed that the tubes were leak tight against helium to the limits of detection.

3. Joining to metal

Because of the high operating temperatures desired, joining the SiC tube to metal demanded a junction that could not only withstand the unavoidable thermal expansion at the joint, but would also neatly mate to a metal transition fitting. For these reasons, molybdenum was chosen as the metal for the fitting, as it has a coefficient of thermal expansion of $5 \times 10^{-6}/^\circ\text{C}$ as compared to that of the Hexoloy SiC of $4 \times 10^{-6}/^\circ\text{C}$ at temperatures up to 700°C , and $5.3 \times 10^{-6}/^\circ\text{C}$ at higher temperatures.

Various methods were investigated to join SiC to molybdenum. After reviewing techniques for ceramic-to-metal joining, brazing provided the most promising optimization between temperature resistance, chemical resistance, vacuum integrity, strength, durability, and cost. At this point, the SiC tubes with the highest degree of roundness were identified for machining purposes, and these tubes were brazed into molybdenum fittings by Omley Industries of Grants Pass, Oregon. The molybdenum fittings were then brazed into stainless steel fittings, and the whole assembly welded onto the desired length of stainless steel tubing. Three different lengths of nozzle tube assembly were used during the course of this development effort, and the lengths depended upon the dimensions of the supporting vacuum systems. In all cases, the entire assembly was welded onto a 1.33 in. Con-Flat vacuum flange to make the external connection to the vacuum system.

4. Machining an orifice

To operate as a nozzle, a method had to be chosen for drilling a small (25–100 μm) hole in the nozzle tip. To in-

vestigate possible machining techniques, such as electron discharge machining (EDM), ultrasonic abrasion, and grinding, samples of SiC tubing were taken to the Fabrication Technical Division at the National Institute for Standards and Technology (NIST) in Gaithersburg, Maryland. EDM showed promise as a method for producing holes, but was time consuming and destroyed several electrodes. The depth of penetration was also difficult to control.

While diamond tools were the only viable option for large scale cutting and machining of SiC, using similar tools for drilling not only appeared problematic and costly in both time and resources, but also would suffer similar difficulties as the EDM approach in attempting to control hole size and uniformity. In addition, mechanical drilling introduces vibration and chatter in the ceramic that can create stress cracks.

As an alternative to mechanically machining SiC, an investigation into laser machining resulted in tests performed at Potomac Photonics of Lanham, Maryland. Potomac Photonics satisfactorily drilled holes of various shapes and depths into sample coupons of Hexoloy SiC.

In order to reduce the amount of material to be drilled, the rounded ends of the SiC tubes were shaved with the aid of a diamond wheel (Norton Company) until the end thicknesses were approximately 1 mm. X-ray micrographs were used to verify the tube end thickness.

An existing Thermionics molybdenum nozzle with a 25 μm aperture was attached to a nitrogen gas feed line in series with a flowmeter (MKS Instruments), and in parallel with the SiC tube to be drilled. As the SiC nozzle was being drilled, the procedure continued until nitrogen flow had effectively doubled, at which point the aperture in the SiC tube orifice was deemed to be approximately 25 μm as well. With the assumption made that the hole was successfully drilled, this method was repeated on a total of four more brazed tubes. Optical inspection of the tube orifices showed them to be approximately 25 μm in diameter on the interior side of the tube, and 100–125 μm in diameter at the outside edge of the tube. Subsequent tubes were drilled to a diameter of 35 μm by Lenox Laser of Glen Arm, Maryland, based upon our requested flow conditions. The final result is the SiC molecular beam nozzle as it is used today.

B. Assembly

Dimpled tungsten shielding was inserted between the SiC tube and the surrounding coolant shroud, and acted as a reflector for the intense heat of the nozzle. This method of shielding has been described previously,^{10,11} and a cross section of the shielded nozzle region is shown in Fig. 3.

The tungsten foil had a thickness of 0.05 mm (Alfa Aesar stock No. 10417) and was coiled to nearly its minimum radius of approximately 2.0 cm. The ideal shield construction would place the tungsten foil as closely as possible to the radiating SiC tube. However, the inner layers of shielding eventually degraded to the point where they cracked, crumbled, or curled along an edge, occasionally causing an electrical short to the SiC tube. Spot welding a layer of tantalum between the foil layers is one method used to reduce this effect,¹¹ but the increased gap used here provided adequate clearance about the nozzle to account for thermal re-

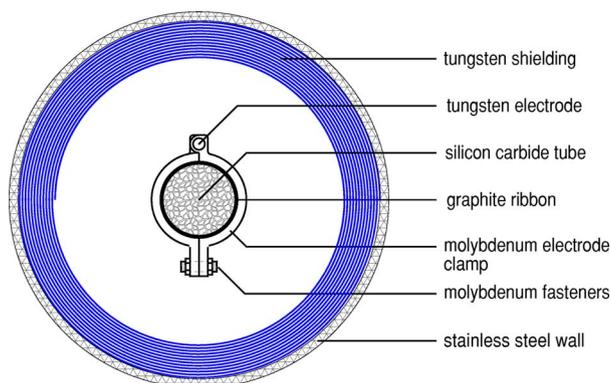


FIG. 3. Cross section through shielding along nozzle axis.

laxation of the assembly hardware and electrodes. The tungsten foil was captured by an Inconel “cage” (Fig. 4) that permitted easy removal of the entire shield assembly for inspection of the nozzle. This also made it easy to replace foil layers without any need for welding.

Electrical connections to the nozzle were made using Tedit graphite ribbon. The graphite acted as both a gasket and an electrical path between a two-piece molybdenum electrode clamp (Fig. 3) and the SiC tube itself. The electrodes were made of tungsten wire, and these were clamped tightly through the molybdenum clamps using studs cut from No. 0-80 threaded molybdenum rod and from No. 0-80 molybdenum nuts, both available from Kimball Physics. Clamped at the nozzle orifice and again midway down its length, the heated region of the nozzle was limited to 10 cm. The nozzle tip was held at positive (+) voltage (<160 V), and the second electrode was connected to ground (0 V).

The heat shield was installed in a shroud assembly that completely enclosed the nozzle assembly. This shroud permitted evacuation in the region between the nozzle and shielding, and supplied gas connections to the nozzle. A coolant line composed of nickel was brazed to the nozzle shroud. All other metal components were fabricated from 304 stain-

less steel, including the ConFlat-compatible vacuum assembly. The molecular beam assembly is shown in Fig. 4. Figure 4(a) represents the complete nozzle assembly, and Fig. 4(b), the nozzle assembly with coolant shroud removed. This lengthy configuration was used in order to position the nozzle deep inside the calibration system with the nozzle orifice closer to the detector.

After a typical installation on the laboratory vacuum system, the entire assembly was leak checked and the nozzle tube was warmed to a few hundred $^{\circ}\text{C}$ to drive off volatiles. After a few hours, the coolant supply was activated and the nozzle was heated until its temperature was high enough to be measured with an optical pyrometer (Spectrodyne DFP 2000). This measurement was performed by sighting through a window along the beam axis. Once the tube became hot enough to be measured with the optical pyrometer, temperature readings were gathered to calibrate the nozzle temperature as a function of nozzle power (Fig. 5). The window was then removed to reduce the risk of operation at high pressures.

Gas connection to the nozzle was supplied through ConFlat and Swagelok hardware. The pure gases He, Ar, and N_2 were connected to the system, as well as a “seeded” gas mixture of 1% Ar in H_2 . The gas pressure was measured using a digital pressure gauge (PSI-Tronix PG5000) or pressure capacitance manometer (MKS Instruments 690A).

A diagram of the SiC nozzle, without shielding, is shown in Fig. 6. Figure 7 is a cross section along the nozzle axis showing the shielded nozzle and its surrounding assembly.

C. Operation

During operation, power was supplied to the nozzle using a high-current supply capable of producing in excess of 30 A (Electronic Measurements, Inc., EMS 150-33-2). Heating the beam began with the current supply set to approximately 1 A. SiC is a semiconductor, and the Hexoloy SiC tubes behave accordingly. As the tube becomes warm over

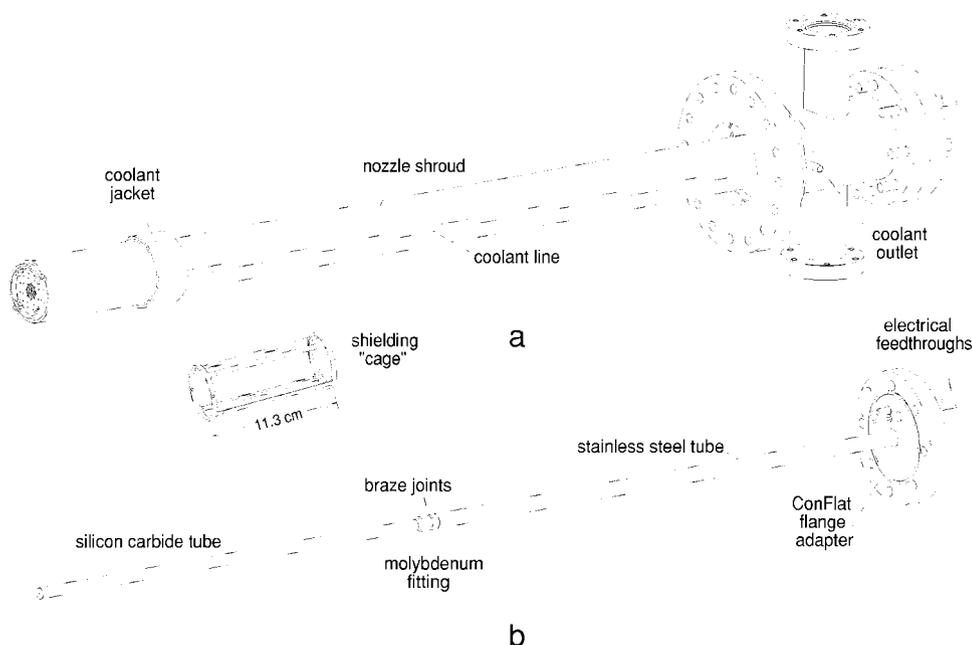


FIG. 4. Nozzle configuration for present molecular beam machine. Figure 5(a) shows the nozzle completely enclosed by the coolant shroud. The cage for the tungsten shielding is also shown. Figure 5(b) shows the nozzle assembly without the shielding and coolant shroud.

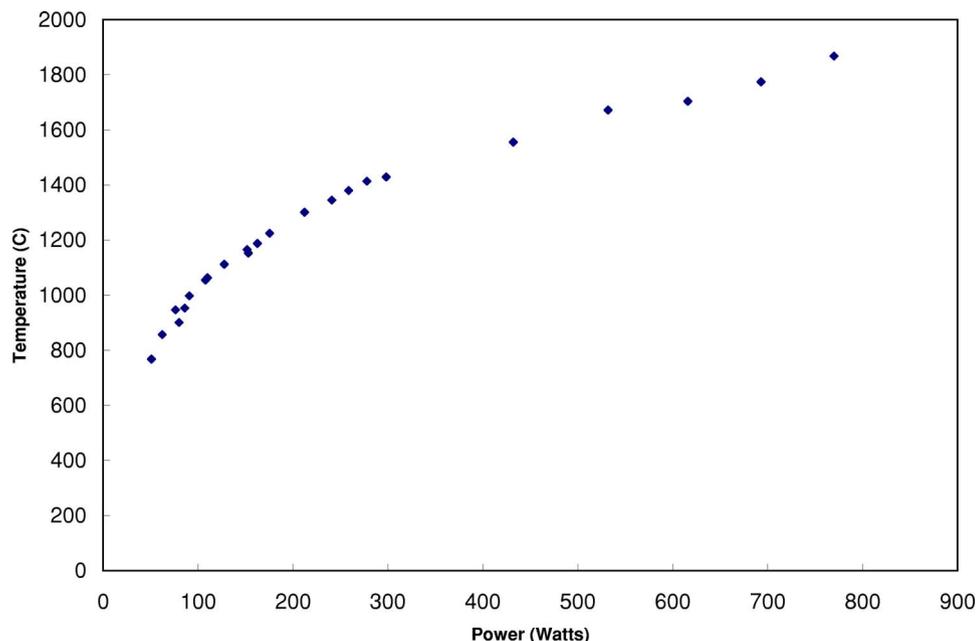


FIG. 5. Nozzle thermal efficiency.

the course of several minutes, resistivity will drop. Eventually, at a temperature lower than that at which it can be observed optically ($<600\text{ }^{\circ}\text{C}$), the tube will “avalanche” and the resistance plummets. Tube lengths showing resistances of 2000–3000 Ω at room temperature typically produced resistances of 4–7 Ω when hot. With 5 A on the power supply, the nozzle reached a temperature of approximately 1150 $^{\circ}\text{C}$.

The beam speed was determined by measuring the time of flight of metastable argon over a measured path.¹² Argon metastables were produced by a pulsed low-energy electron beam and detected by a secondary electron multiplier. For Ar, the typical bias for the filament was 30 V with an electron emission set to around 200 μA in the dc mode. This electron energy was also found to produce metastable peaks in pure He, H₂, and N₂.

Two types of multipliers were used to detect metastables in the beam. The first was a channel electron multiplier (Galileo Electro-Optics Corporation) that operated with a beryllium-copper (BeCu) “first surface” to convert the surplus energy of the metastables into ejected electrons. These electrons were drawn into the throat of the multiplier by a positive high voltage bias on the detector. The second detector used was a discrete dynode multiplier (ETP Scientific). In this detector arrangement, the first surface was the first dynode of a 20-stage multiplier coated with a thin film of aluminum oxide.

The beam was aligned at the target using an *x-y-z* manipulator stage (McAllister Technical Services). The excitation source was pulsed by a deflection amplifier (Physicon Corporation RS 1224) driven by a function generator (Stanford Research Systems DS 345). The metastable arrival pulses were accumulated by a multichannel scaler (Stanford Research Systems SR430) through an amplifier (Stanford Research Systems SR445). The time between the trigger of the excitation pulse and the peak in the arrival pulse distribution was recorded as the time of flight (TOF) of the beam.

To determine the beam flux, the multiplier detector was

removed and replaced by a quadrupole mass spectrometer (QMS) (Ametek Dycor Model QMS 1000). The QMS was installed in the target position behind an orifice of 0.1 cm². The sensitivity of the QMS was determined with the aid of calibrated leaks and a spinning rotor gauge (MKS Instruments SRG-2CE) used as a transfer pressure standard.

IV. RESULTS

The SiC laser-drilled tubes were operated as molecular beam nozzles with hydrogen, helium, nitrogen, and argon.

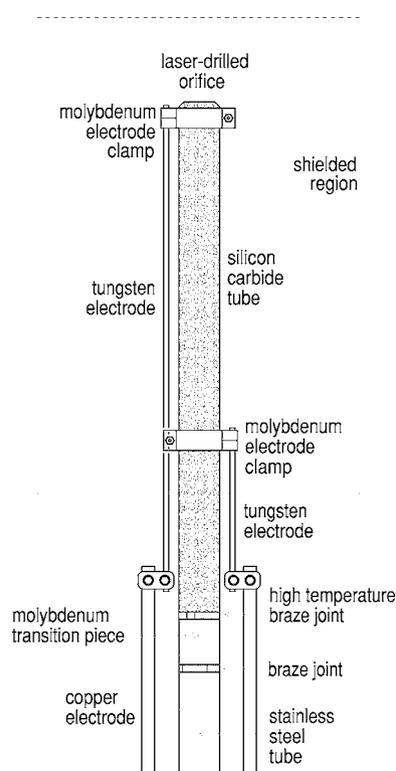


FIG. 6. Diagram of SiC nozzle showing electrode connections (not to scale).

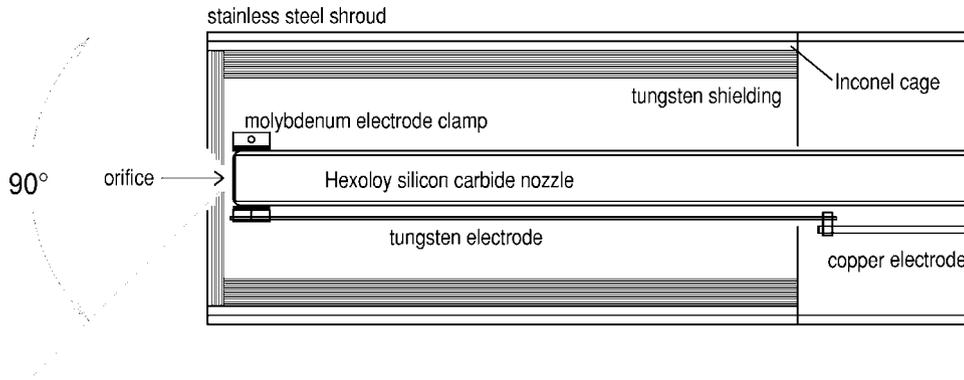


FIG. 7. A cross section along the nozzle axis showing the tungsten shielding and one of two electrode assemblies.

The hydrogen gas was actually a mixture of 1% argon, which allowed the molecular beam to be “tagged” by exciting argon metastables. Metastable TOF detection was achieved in all Ar–H₂, Ar–He, Ar, and N₂ molecular beam tests. Results obtained for a typical experiment in pure Ar and for the 1% Ar in H₂ mixture at 900 and 1200 °C are shown in Fig. 8, along with the theoretical terminal beam speed⁵ for pure Ar at 1200 °C. Raw data for a typical metastable TOF scan for a molecular beam of Ar in H₂ are displayed in Fig. 9.

Performance of the commercial molybdenum nozzle at room temperature was without incident. However, difficulties arose at temperatures above 1000 °C for that nozzle that were not an issue or were not nearly as prevalent in the SiC version. Temperature-related mechanical malfunctions, short component lifetimes, and alignment issues made beam diagnostics problematic. Despite these issues, many metastable TOF measurements were obtained for the commercial nozzle, but the molecular beam flux was never determined. On one occasion, by exceeding its design parameters, the commercial nozzle briefly achieved a high speed of 3 km/s for metastable Ar in the 1% Ar in H₂ mixture at a temperature of 1525 °C.

Heating of the SiC nozzle was routine except under the most extreme conditions. The lifetime of the nozzle at 1000 °C appeared to be many hundreds of hours, but above 1500 °C, only one nozzle survived beyond several hours. Thermal shock due to cycling the nozzle too quickly, from extreme temperature to room temperature, may have contributed to the short lifetimes observed above 1500 °C. This assumption is supported by observations from industry experts on high temperature use of SiC. Data from Carborundum Corporation, the manufacturer of Hexoloy, show an abrupt 33% change in the coefficient of thermal expansion at 700 C.¹³ The typical failure mode was a crack at the ground electrode connection on the SiC nozzle. This manifested itself by an abrupt, but tolerable change in the background pressure. Upon removing the nozzle, the tube would separate at the weakened location. Since the ground junction represents the highest thermal gradient along the tube, a crack at that location was not surprising. A wider contact surface for the electrical ground might alleviate this problem.

One SiC nozzle was operated to 2000 °C, but subsequently cracked upon cooling. A succeeding nozzle was heated to only 1850 °C, and did not crack upon cooling.

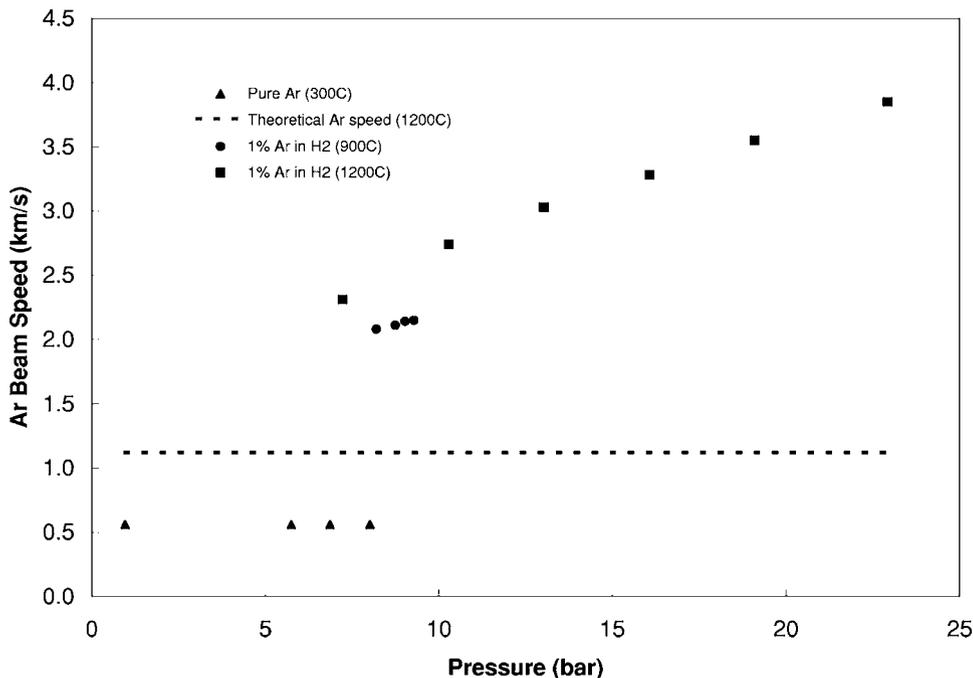


FIG. 8. Typical time-of-flight scans of metastable argon in molecular beams at various temperatures. Measured beam speeds are shown for pure Ar at room temperature (300 °C), and for a 1% Ar–H₂ gas mixture at elevated temperatures (900 and 1200 °C). The dotted line represents the pressure-independent theoretical beam speed for pure Ar at 1200 °C.

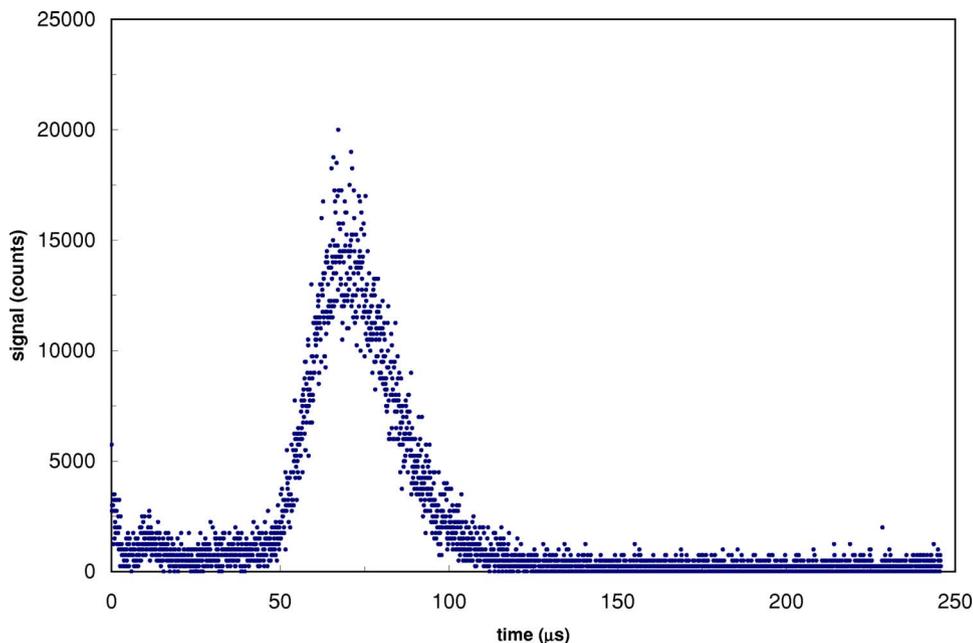


FIG. 9. Time of flight scan for Ar-H₂ mix at 23 bars and 1150 °C.

This could have been due to a combination of reduced temperature and more care taken while decreasing the nozzle power. According to Stross of Spectrodyne, Inc., SiC thermocouple protection tubes used in furnaces will last many times longer when routinely maintained at an elevated temperature and not allowed to return to room temperature.

In both cases where the nozzles were operated at extreme temperatures, each was plagued by episodes of clogging. Reduced pressure downstream of the nozzle will occur due to increased gas viscosity within the nozzle at high temperatures. Consequently, a drop in chamber pressure could be due to increased gas temperature or an orifice obstruction. The system was therefore constantly monitored to observe the moment at which a forming obstruction produced a rapid pressure drop.

The appearance of the SiC tube was remarkably unchanged after repeated operation below 1200 °C, but invariably clogged within minutes above 1500 °C. After several hours above 1500 °C, the nozzle diameter was measurably reduced by a few tens of microns. Both of these effects may be due to sublimation, as a significant multicomponent vapor pressure has been previously measured above a hot silicon carbide surface.¹⁴ Evaporated material is likely condensing about the nozzle orifice, as the orifice was significantly cooler than the adjacent heated region of the tube wall. Furthermore, clogging occurred even during operation with pure argon, implying that the formation of the obstruction is independent of gas composition.

When a nozzle became clogged, the obstruction was removed by heating the nozzle while charged with air. This suggested that the obstruction was a condensable by-product of the hot SiC, and attacked by oxygen. As a corrective technique, this would only be possible for sufficiently small orifices that would not overwhelm a vacuum system with the nozzle inlet charged to atmospheric pressure.

One nozzle was heated to 1850 °C long enough to obtain a temperature reading with the optical pyrometer. With-

out the pyrometer, a welder's mask was needed to view the nozzle at this temperature. After the optical window was removed, the nozzle was charged with the 1% Ar in H₂ gas mixture, and again heated to 1850 °C. Though the nozzle soon became clogged, it was heated at this level for several minutes without any mechanical failure to the tube. At this temperature, the delivery pressure was increased to the limits of the regulator (30 bars) without any ill effects to the nozzle, but this failed to clear the orifice obstruction.

In most cases, there was no observed electrical degradation due to exposure to gases or heat over the nozzle lifetime. However, one nozzle regularly lost its outer conductive layer during operation with hydrogen. Once the SiC becomes hot, it is highly conductive, but obtaining a hot tube demanded a conductive outer wall to draw sufficient current to heat the underlying tube. For the tube in question, conductivity was restored by rubbing graphite onto the outer surface of the tube wall.

The nozzle presently in use was heated to 1170 °C and has never clogged. Despite this reduced temperature over previous nozzles, argon speed of 3.9 km/s was achieved at a nozzle pressure of 23 bars, resulting in a peak in the arrival signal for the argon metastables of 14 400 counts/sec. The integration period that produced this maximum signal also produced half-maximum points at speeds of 3.1 and 4.7 km/s.

Based upon the measurements obtained with the QMS in the target position, the argon beam flux was determined to be approximately 1×10^{12} particles/cm² s at a nozzle pressure of 12 bars and a temperature of 1150 °C. The measured argon beam speed at these conditions was approximately 3 km/s. Even at 23 bars, the argon speed increased with increasing nozzle pressure, suggesting the argon component was not fully "coupled" by collisions to the hydrogen component.

At the most extreme conditions of nozzle temperature

and pressure, the measured speed of the peak in the argon beam component approached 4 km/s. Under these conditions, the pumping capacity of the present vacuum system limited the argon flux to 1×10^{12} particles/cm² s due to beam scattering with the background gas. However, tests performed at lower pressures produced argon fluxes in excess of 1×10^{14} particles/cm² s, and suggested similar fluxes are obtainable at high nozzle pressures with improved pumping. The theoretical treatment of molecular beam fluxes by Ramsey¹⁵ yields a hydrogen flux of 1.7×10^{16} particles/cm² s under the nozzle conditions described above. Consequently, a measured flux of 1×10^{14} particles/cm² s for the 1% argon beam component seems reasonable when compared to the theoretical approximation.

V. DISCUSSION

A silicon carbide (SiC) nozzle is capable of producing molecular beams suitable for simulating the space environment. Though use in an oxygen atmosphere is limited, SiC is capable of long-term operation in inert gases at temperatures below 1500 °C. Under these conditions, the observed degradation of the nozzle is so minimal as to suggest a lifetime of many hundreds of hours.

Compared to oxide ceramics such as thoria and alumina, SiC is a rugged alternative that functions well in hydrogen, and may suggest applications in extreme environments where traditional metal or ceramic tubes are impractical. Silicon carbide has proven to be a mechanically and chemically durable ceramic, although continued work remains to overcome difficulties related to temperature cycling and machining of components.

Future efforts will attempt to improve the beam flux and beam speed by reducing the expansion chamber background pressure and by establishing optimum nozzle operating conditions.

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- ¹J. B. French, *Molecular Beams for Rarefied Gasdynamic Research*, edited by W. C. Nelson, AGARDograph, Vol. 112, (NATO/ACARD, 1966).
- ²J. B. French, N. M. Reid, A. O. Nier, and J. L. Hayden, *AIAA J.* **13**, 1641 (1975).
- ³H. Pauly, in *Atomic and Molecular Beam Methods*, edited by G. Scoles (Oxford University Press, New York, 1988).
- ⁴J. Deckers and J. B. Fenn, *Rev. Sci. Instrum.* **34**, 96 (1963).
- ⁵D. R. Miller, in *Atomic and Molecular Beam Methods*, edited by G. Scoles (Oxford University Press, New York, 1988).
- ⁶R. Klingelhofer and P. Louse, *Phys. Fluids* **7**, 379 (1964).
- ⁷J. B. Fenn, *Entropie* **18**, 11 (1967).
- ⁸W. T. Kasprzak, H. Niemann, D. Harpold, J. Richards, H. Manning, E. Patrick, and P. Mahaffy, *Proc. SPIE* **2803**, 129 (1996).
- ⁹J. H. Waite *et al.*, *Space Sci. Rev.* **114**, 113 (2004).
- ¹⁰C. K. Lam, Institute for Aerospace Studies Report No. 212 (unpublished).
- ¹¹J. O. Ballenthin and A. O. Nier, *Rev. Sci. Instrum.* **52**, 1016 (1981).
- ¹²J. W. Locke and J. B. French, *J. Vac. Sci. Technol.* **7**, 46 (1970).
- ¹³Carborundum Corporation Form A-12071-14 (unpublished).
- ¹⁴J. Drowart, G. De Maria, and M. G. Ingrham, *J. Chem. Phys.* **41**, 1015 (1958).
- ¹⁵N. F. Ramsey, *Molecular Beams* (Oxford University Press, Oxford, 1956).