11th Australasian Fluid Mechanics Conference
University of Tasmania, Hobart, Australia
14-18 December 1992

SPECIES MEASUREMENTS USING MASS SPECTROMETRY IN HIGH MACH NUMBER FLOWS

K.A. SKINNER

Mechanical Engineering Department
University of Queensland
QLD 4072, AUSTRALIA

ABSTRACT

A time-of-flight mass spectrometer has been constructed which demonstrates an ability to detect and to measure quantities of gas molecules present in high Mach number, high enthalpy flows. The instrument has been operated within the environment of a free piston reflected shock tunnel, T4, at the University of Queensland and has allowed measurement of relative concentrations of major species present.

INTRODUCTION

The recent re-emergence of interest in the development of a supersonic combustion ramjet (scramjet) has led to a demand for experimental data to test understanding of the chemistry and flow dynamics of fuel injection into a hypersonic airflow. The most promising fuel is hydrogen. The specific release of energy from a hydrogen-air flame is of the same order as the freestream specific enthalpy indicating that the flow dynamics and the chemical kinetics in a scramjet engine are strongly interdependent.

The ability of shock tunnels to produce high enthalpy gas flows with densities sufficient to allow combustion, has long been established. However, the extreme nature of the flow as well as its short duration has required the development of new instrumentation and techniques to measure the gas parameters.

The possibility of using a mass spectrometer to measure molecular species concentrations within flows produced in high enthalpy impulse facilities, was demonstrated by Crane and Stalker (1977) in the Australian National University's T3 shock tunnel. Crane incorporated a quadrupole mass spectrometer with a dual hollow conical skimmer gas sampling system. The impulse nature of the flow and the inability of the quadruple mass filter to record a range of mass numbers only allowed the relative concentration of a single species of molecule to be measured during each test time produced. To obtain relative concentrations between different species, Crane relied upon sampling multiple tests and an assumption of repeatability between test times produced by the tunnel.

Test times of the order of milliseconds suggest that a more appropriate method of mass separation than a quadrupole mass filter would be using ion time-of-flight. Chemical rate processes have been studied using shock tubes coupled with time-of-flight mass spectrometers. The technique has been useful in examining transient and non-equilibrium effects resulting from the sudden increase in temperature and density of a gas sample near the end wall of a reflected shock tube.

A time-of-flight mass spectrometer has been developed specifically to investigate the mixing and combustion region downstream of a hydrogen injector in a generic scramjet model.

EXPERIMENTAL DESIGN

Geometry and Environment

The test section of the shock tunnel T4 is 450 mm square x 1200 mm long into which extends a contoured nozzle designed to produce a uniform mach 5 flow from a 25.4 mm throat. The diameter of the exit of the nozzle is 261 mm. In this test flow can be placed a model scramjet engine, but for initial operation of the mass spectrometer a direct sample of the test flow was taken. The test section is evacuated to a pressure of 133 Pa or better prior to each firing of the shock tunnel.

A schematic of the mass spectrometer is shown in figure 1. At the upstream end are three co-axial hollow conical skimmers used to sample the test flow. Behind the second and third skimmers are vacuum chambers evacuated by diffusion pumps. Free stream flow passing through the skimmers is formed into a molecular beam entering the inside high vacuum chamber. The mean free path of molecules inside this chamber is one metre. 20 mm behind the final skimmer, an electron beam intersects the molecular beam, causing ionisation. The ions produced are accelerated along a metre long flight tube to an electron multiplier particle detector. The signal from this particle detector drives a preamplifier which provides a signal to a digital storage oscilloscope.

The drift tube containing the ion path from the ioniser to the particle detector should ideally be perpedicular to the axis of the sampler to reduce pressure build up along the ion path during the test time. The maximum angle that could be accommodated was 15 degrees. The mass spectrometer is mounted within the test section with room to allow it pointwise 3D coverage of a volume 50 mm x 100 mm transversly x 1000 mm axially, a large enough region to accomodate combustion models.

Sampling

Extraction of gas samples from high mach number flows has been reported previously by Skinner (1961), Reis and Fenn (1963), and Bird (1976), amongst many others. The advantages of using a flow "skimming" technique is that the composition of the flow in the molecular beam is not affected by approach to chemical equilibrium of the sample gas. The streamtube incident at an orifice at the tip of the cone does

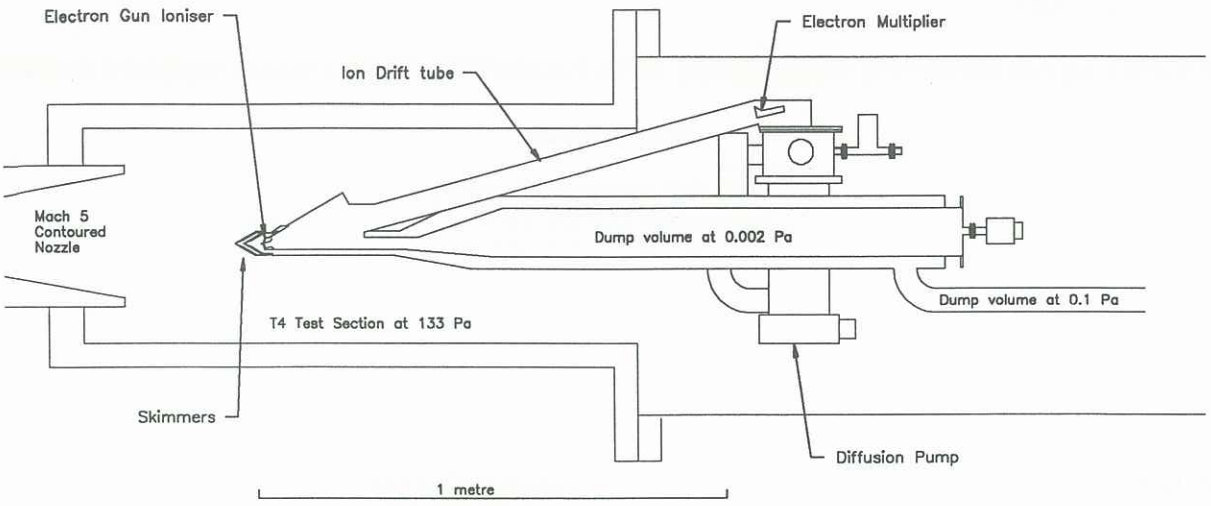

Figure 1. Schematic of experimental layout.

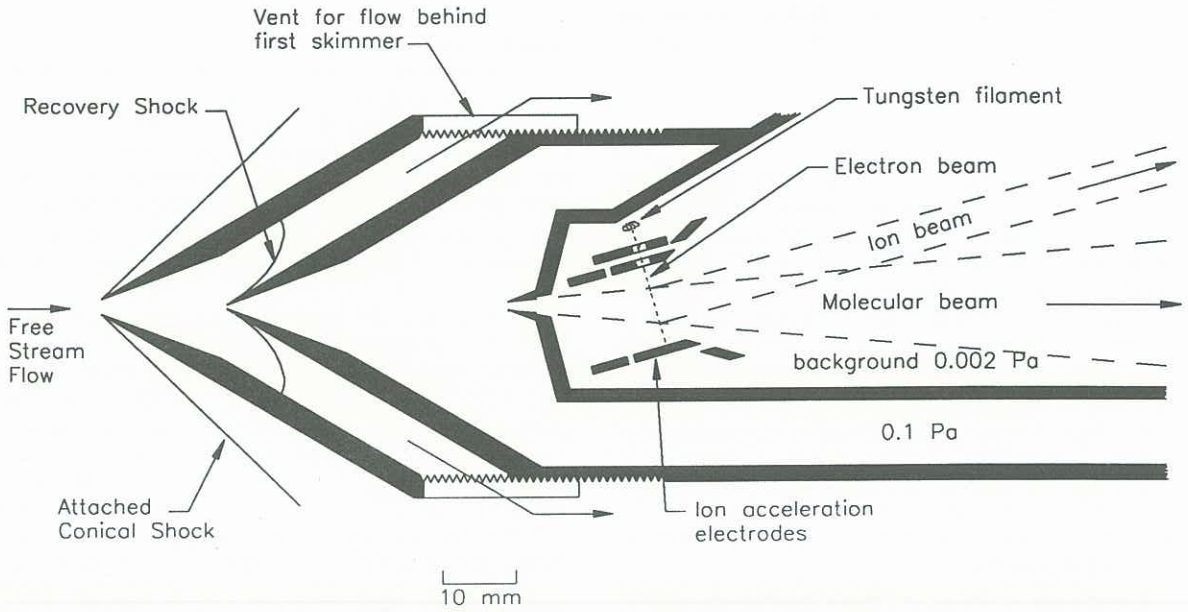

Figure 2. Skimmer arrangement and ioniser.

not pass through the shock wave attached to the exterior of the hollow cone. The first infuence which the skimmer has upon the axial sample of gas is the arrival of the expansion fan off the interior wall. The strength of this expansion depends on the size of the skimmer orifice. For small orifice diameters, the density falls fast enough to halt chemical reactions and freeze the composition of the gas.

Assuming a non-reacting flow inside the skimmer, the properties of the gas could be calculated using a method of characteristics code, (Jacobs and Gourlay, 1991), as long as continuum flow could be maintained. Boundary layer properties within the cone were not considered as only the on-axis gas was retained as the sample. Only the flow between the first and second skimmers could be analysed using continuum assumptions.

The first skimmer had an orifice diameter of 2.0 mm and operated within the continuum flow regime. The gas was expanded from freestream Mach 5 to Mach 10 at the entrance to the second skimmer. The second skimmer was considered the location of the sample, as the gas underwent a strong expansion inside the orifice of this skimmer. The purpose of the first skimmer was to act as a nozzle to reduce the density of the flow entering the mass spectrometer to avoid vacuum deterioration. No inter-skimmer dump cavity existed between the first and second skimmers. Instead the off-axis flow passed through a normal shock, recovering pressure, and venting subsonically back out into the free stream. The location of the shock was at the point in the flow where the recovery pressure through a normal shock equalled the free stream static pressure The second skimmer needed to be ahead of this normal shock so as to maintain supersonic flow at the orifice.

The orifice at the tip of the second skimmer was 0.7 mm in diameter. The flow within this skimmer passes from continuum to free molecular and was therefore not able to be analysed analytically. During this transition species separation occurs due to velocity slip between molecules of different mass, which decreases the relative concentrations of the lighter particles along the axis of the flow. This alteration in the composition of the flow is repeatable, however, and is a function of the density, enthalpy, and mass ratio of the molecules. As such, it could be calibrated for and its influence included in measurements.

The third skimmer orifice is 1 mm and acts as a beam collimator in the rarefied flow.

Mass Separation

Behind the third skimmer a compact electron gun ionises a portion of the molecular beam. A Tungsten filament operating at 6 Watts provides electrons which are accelerated into a cylindrical electrode at a potential of 250 V. The flow of electrons is controlled by a gate electrode at +/- 15 V which can produce a pulse 200 nsec in duration. The 250 V cylindrical electrode is aligned with the drift tube and additional electrodes on either side control the potential gradient in the ionisation region. The positive ions are accelerated into the drift tube. The ions drift free from he ioniser with an energy of 250 eV towards the particle detector. For singly charged ions, flight time, T, varied with mass, m, according to the equation:

$$T = L\sqrt{\frac{m}{2E}}$$

where L is the length of the drift tube, and E is the energy of the ion.

CALIBRATION

The performance of the three conical skimmers was difficult to assess experimentally. Shock attachment at the lip of the first skimmer was verified from luminosity photographs. No means of measuring pressure distributions within the flow were available, however the stagnation point heat transfer in the molecular beam is sensitive to the presence of a bow shock across the second skimmer. A heat transfer gauge of the thin film variety was used to measure the stagnation point heat transfer directly behind the third skimmer.

For a test gas of air at 10.5 MJ/kg the heat transfer behind the third skimmer was measured at 5 W/m² which corresponded to a beam velocity of 4500 m/sec and a particle flux of 4 x 10^{17} /mm/sec. In the presence of a bow shock across the second skimmer both the stream velocity and the axial particle density would decrease, yielding a lower heat transfer rate.

The operation of the mass spectrometer was tested outside the shock tunnel by attaching an evacuated chamber and a solenoid valve in place of the first skimmer. This could be pulsed open for 10 msec allowing a sample of gas in through the second skimmer. In this configuration, the second skimmer acted as a laval nozzle with a sonic throat at the tip. The third skimmer sampled the flow from the second skimmer and although the collimation of the beam behind the third skimmer was not as narrow as in T4, the density of the beam was of the same order.

The use of the mass spectrometer in this fashion allowed the calibration for the ion collection efficiencies of the species in the gas behind the valve. Using known gas mixtures, the ratio of areas of peaks was calculated for different molecules and this ratio could then be applied to mixturesof unknown concentrations. The ionization cross sections for most molecules present in the test flows are available with errors less than 10%. Dissociative ionisation of diatomic molecules is less acurately known and the collection efficiencies of atomic species from pre-dissociated molecules could not be measured outside the shock tunnel.

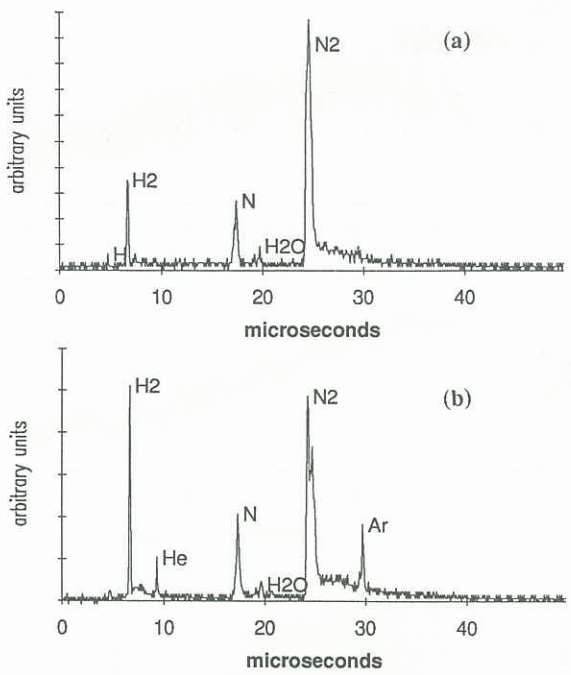

Figure 3. Sample spectra produced from a hydrogen-nitrogen test gas in T4 showing, (a) during test time, (b) later, showing driver gas arrival. The time is measured from the electron gun pulse.

The remaining requirement for relative species measurements is for the the error introduced by mass separation between skimmers two and three. This was performed in the shock tunnel because of its dependence on enthalpy. Known mixtures were used as a test gas and a depletion factor obtained to account for the relative decrease in concentration of the lighter molecule.

PERFORMANCE

The mass spectrometer was installed in the test section of the shock tunnel and used to record mass spectra from samples of test flow from a Mach 5 nozzle. The electron gun was pulsed on for 200 nsec every 0.55 msec, producing a packet of ions which were then detected and recorded on a storage oscilloscope. The oscilloscope recorded output from the electron multiplier every 100 nsec for a duration of 6.4 msec, allowing 117 complete spectra to be obtained during each firing of the shock tunnel. The limitating resolution of the area under each peak is determined by the sampling rate of the data storage. A smaller error in peak size can be obtained at the expense of reduced duration of recording by increasing the sample rate of the oscilloscope. In this initial application the motivation was the detection of the onset of driver gas contamination in the test gas, and so the increased length of record was chosen at the expense of peak resolution. Even so the spectra obtained were mostly well defined.

Figure 3 shows samples of spectra obtained at different times during a flow of nitrogen-hydrogen test gas with a

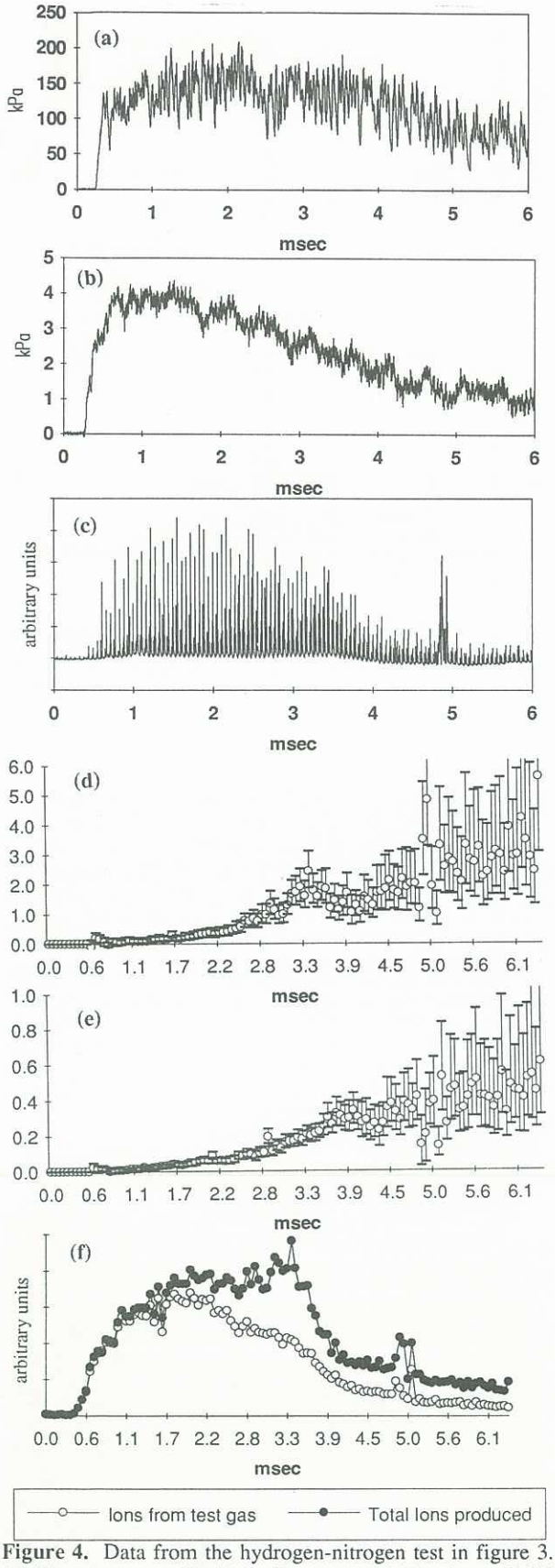

Figure 4. Data from the hydrogen-nitrogen test in figure 3. (a) Pitot pressure. (b) Static Pressure. (c) Envelope of 117 spectra produced consecutively during shot. (d) [He]/[N2] in free stream measured for each spectra. (e) [Ar]/[N2] in free stream measured for each spectra. (f) Relative contribution of test gas to the total number density throughout the flow duration

helium-argon driver gas. The noise in the baseline was generated within the preamplifier. The presence of a peak can often be recognised when the area of the peak is only 2% of the area of the peak of N_2 (typically the largest peak in most flows studied). This demonstrates the ability of the spectrometer to detect and resolve the species which may be encountered in the combustion region of a scramjet engine, as well as to detect the onset of driver gas contamination.

Measurements of relative mole fractions, $[He]/[N_2]$, $[Ar]/[N_2]$,and pressure traces for this run are shown in figure 4. These are calibrated using measurements of peak sizes from runs with test gas mixtures of helium-nitrogen and argon-nitrogen at the same condition. The composition of the driver gas was known to be 15% Ar - 85% He. The independently calibrated measurements of relative concentration arrive at the same ratio.

The instrument was used for 54 shots of the shock tunnel. The major difficulty was in maintaining the vacuum. The high impulse loading stressed joints around the drift tube and caused small leaks which were able to be repaired in-situ. The tungsten filament and electronics did not suffer overly from the tunnel environment. Vibration did not have any effect. The three skimmers were not physically degraded by the flow, although dirt built up inside them which required regular cleaning

CONCLUSIONS

The mass spectrometer has been demonstrated as a feasible instrument for high quality measurements in hypersonic flow. The ability of the mass spectrometer to detect molecular species present at levels of only 2% by number has been shown. The current limitation on this is the background noise generated in the signal preamplifier, which leaves the possibility of a more sensitive instrument.

The ability to produce measurements of relative species concentrations from the output of the mass spectrometer has also been shown. With suitable calibration, the error can be less than 5% for major species, the error increasing as the strength of the signal decreases.

The instrument is reasonably robust and has been used to investigate the test gas produced by a free piston reflected shock tunnel. Further work is planned in the near future applying it to studies of the combustion region of a model scramjet.

ACKNOWLEDGEMENTS

The author wishes to acknowledge Professor Ray Stalker for valuable discussion and The Australian Research Council and the National Aeornatical and Space Administration for financial assistance.

REFERENCES

BIRD GA (1976) Transition regime behaviour of supersonic skimmers. Phys Fluids, 19, 1486-1491.

CRANE, KCA and STALKER, RJ (1975) Mass-spectrometric analysis of hypersonic flows. J.Phys D., 10, 679-695.

JACOBS, PA and GOURLAY, CM (1991) An Interactive Method-of-Characteristics Program for Gas-Dynamic Calculations. Int. J. Appl. Engng. Ed., 7, 242-250

REIS, VH and FENN, JB (1963) Separation of gas mixtures in supersonic jets. J. Chem. Phys., 39, 3240.

SKINNER, GT (1961) Molecular beam for the study of high-temperature-gas collision processes. Phys. Fluids, 4,1172