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ARC JET DIAGNOSTICS TESTS

Final Report

NASA/ASEE Summer Faculty Fellowship Program--1988

Johnson Space Center

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ABSTRACT[1]

Two objectives were addressed during a 10 week 1988 NASA/ASEE summer faculty fellowship at the Johnson Space Center Atmospheric Reentry Materials Structures Evaluation Facility (ARMSEF). These objectives were the evaluation of mass spectrometry for the measurement of atomic and molecular species in an arc jet environment, and the determination of atomic recombination coefficients for RCG (reaction cured glass) coated HRSI (high temperature surface insulation) materials subjected to simulated reentry conditions.

Evaluation of mass spectrometry for the measurement of atomic and molecular species provided some of the first measurements of point compositions in an arc jet tunnel environments. A major objective of this project centered around the sampling residence time. A three staged vacuum sampling system pulled the molecules and atoms from the arc jet to a quadrupole ionization mass spectrometer in 400 milliseconds. Conditions investigated included a composition survey across the nozzle exit at 3 cm z-distance from the nozzle exit for 3 different currents. Also, a point composition survey was taken around a shock created by the the presence of a blunt body.

Results for the composition survey across the nozzle exit showed concentration gradients for oxygen and its related secondary species: oxygen atoms, nitric oxide, and carbon dioxide. This gradient was more pronounced at higher currents and locations closer to the nozzle exit. The gradients were skewed from the centerline with maximum concentration occurring at a theta of 1.0 degree. A composition survey across the shock showed nitric oxide concentrations dropped while water concentrations increased about 6 fold. Other species also showed small changes. By using the existing copper pitot tube, little disturbance occurred to the arc jet operations.

Recombination measurements on RCG coated HRSI materials showed low catalytic recombination coefficients for both nitrogen and oxygen atoms. The experiments were performed on a 5.08 cm radius HRSI hemispherical model (LI-900) coated with a reaction cured glass coating. Gas mixtures investigated included pure nitrogen, 11%, 14% and 23% oxygen in nitrogen. Flowrates and currents ranged from 0.036 to 0.056 kg/sec, and 300 to 550 amps respectively. The results showed that the nitrogen recombination coefficient is relatively constant at 0.014 over a temperature range of 1350 to 1550 K. These results compare favorably with those reported by Scott (1981) and Kolodziej and Stewart (1987). The analyses of air runs were not as conclusive. Experimental repeats within a run were good, however, repeats outside of a run showed a variation in surface temperatures of 60 K. Oxygen concentration appears to have an influence, however, more measurements are recommended.

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[1] This report is dedicated to Dr. Frederic A. Wierum - a pioneer in Arc Jet Diagnostics

INTRODUCTION

This report is divided into two major sections. The first section will discuss mass spectrometry of atomic and molecular species in an arc jet. The second section will discuss atomic recombination measurements on RCG coated HRSI materials.

SECTION 1

EVALUATION OF MASS SPECTROMETRY FOR ARC JET SPECIES IDENTIFICATION

BACKGROUND

Quadrupole ionization mass spectrometry is a well developed analytical technique used for the determination of gas compositions via mass separation. The principle centers around an ionization filament which emits electrons. These free electrons strike a fraction of molecules and atoms as they pass through the ionization cage. These molecules and atoms lose an electron as a result of this collision and a charged positive ion results. Under the presence of a rf voltage superimposed on a dc voltage established by a two sets of oppositely charged rods (quadrupoles), certain charged ions can pass through the quadrupoles and strike a detector (a faraday cup in this instance). By ramping a potential across the quadrupoles a "scan" can be performed which will measure the m/e (mass to charge ratio) present in the mass spectrometer at any given time. Ions created in the process can also fragment and thus a parent molecule like a nitrogen will have a signal at 14 and 28 plus a small amount at 29 for the NN^{15} isotope. Further details about mass spectrometry may be found in a book by McLafferty.

Mass spectrometry has been attempted in arc jet environments before with limited success. A major limitation was residence time. Real time responses were on the order of 30 minutes - a response time much too long. A mass spectrometer sampling system developed by R.J.Willey at Northeastern University has enable rapid response of an order of 1 second for gas samples from an atmospheric environment. This rapid response was achieved by a staged vacuum system. Therefore an earlier drawback could be eliminated if the sampling system could be adapted to an arc jet environment.

Another concern in mass spectrometry as applied to arc jet environments is the insurance that what is measured is what appears in the stream. The presence of high atom concentrations in the stream present a possible sampling problem because of atomic recombination in the probe and sample line. Recombination reactions can occur in both the gas phase and along the probe walls as the gas sample travels from the source to the mass spectrometer. A preliminary study completed at Northeastern University showed that homogeneous gas phase recombination reactions could be virtually eliminated by the running at pressures below 20 microns at a residence time of 1 second. Wall reactions could be eliminated by using a specially designed probe constructed an inert wall material (quartz).

EXPERIMENTAL

A block diagram of the mass spectrometer system is shown in Figure 1. The initial study used the existing pitot tube (constructed of copper) as the sample probe. The pitot tube was tied to the mass spectrometer by a 2 m 0.188 I.D. Resistoflex tubing (Teflon^{TM} lined) and 1.5 m 1/4" O.D. Eastman Nylon^{TM} tubing. Four valves separated 3 vacuum stages and the mass spectrometer chamber. These valves were a solenoid valve which allowed remote sampling capability, 2 Nupro SS-4H bellow vacuum valves and one Nupro SS-4BG-TW bellow vacuum valve. The vacuum pumps were standard mechanical vacuum pumps manufactured by Sargent Welch, Duo Seal and Cenco. The mass spectrometer chamber was evacuated by a Balzer's turbo molecular pump Model # TPH050 and this pump was backed by an Edward's Model E2M-1 high vacuum pump.

The mass spectrometer used was an UTI AMX-100. The mass spectrometer has a m/e range of 1 to 100. Thus all light gases below a molecular weight of 100 can be observed. Signals from the mass spectrometer were interfaced to an IBM-XT computer through a Cybord A/D and D/A converter (Isaac Model 91-I). Programming written at Northeastern University allowed for the collection of mass scans or selected peak scanning as a function of time.

Experimental conditions investigated were:

Currents: 400, 800, 1100 amps

Pitot z-distances from the nozzle: 3 cm, 23 cm, 43 cm

Radial Positions from the centerline: +/- 7.9, 6.32, 4.74, 3.16, 1.58,0 degrees

Gases: Air, Nitrogen, and Argon

RESULTS AND DISCUSSION

Shown in Figure 2 is the mass spectrometer response at m/e= 30 (Nitric Oxide) for a composition survey at 3 current levels across an arc normal to the nozzle exit plane and located 3 cm from the nozzle exit. Several phenomena are evident. As the current is increased, nitric oxide concentrations increase, in this case from 2% to 11% at the centerline. Further, the results showed more nitric oxide at the centerline then at the edges of the nozzle thus indicating a concentration gradient across the nozzle. Gradients were also observed for oxygen molecules, oxygen atoms, and carbon dioxide.

Also shown in Figure 2 is a pitot pressure survey. Note that it does not show as high as % maxima. Thus the peaks seen at 800 and 1100 amps for nitric oxide suggest that centerline concentrations for this species are higher. Another source for nitric oxide may be the reaction of nitrogen atoms with oxygen atoms in the probe. However as is discussed below, nitrogen atoms are reaching the mass spectrometer and it is currently suspected that the nitric oxide measured is coming from the arc tunnel. The skewness shown in the figure may represent an arc column slightly out of line.

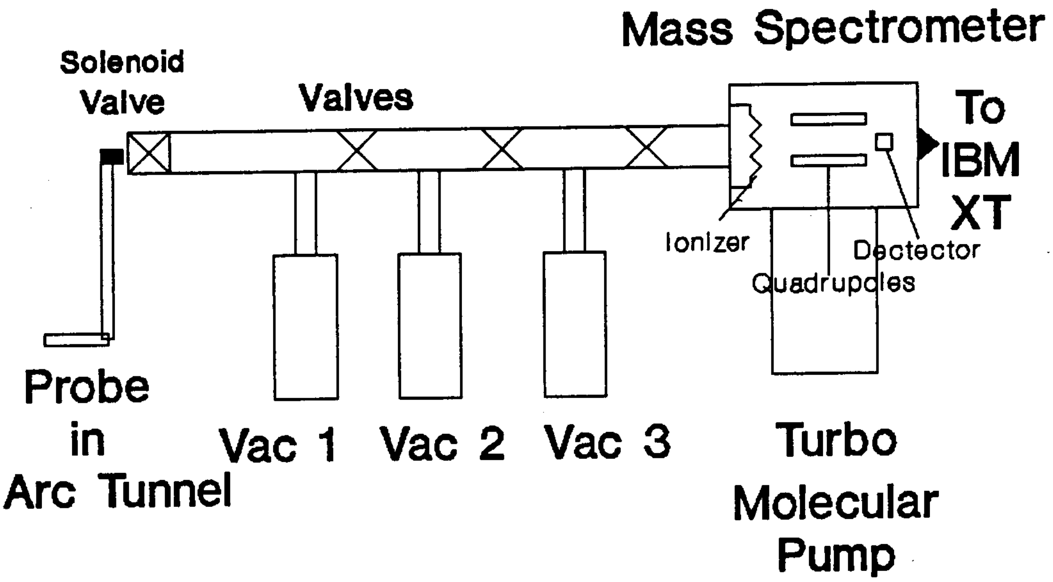

Figure 1. Block Diagram for the Mass Spectrometer System.

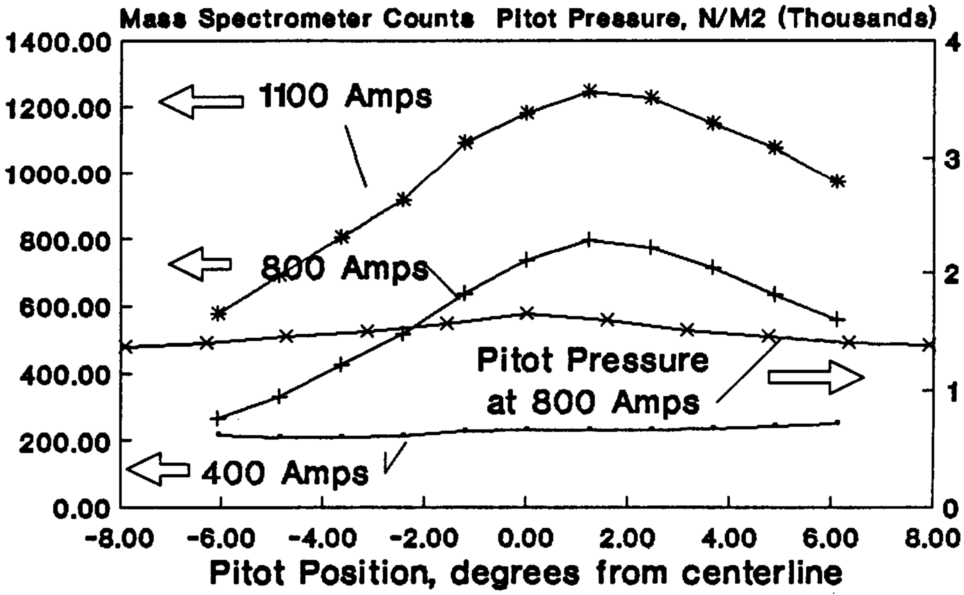

Figure 2. Example of Nitric Oxide Concentration for Composition Survey across the Nozzle Exit Conducted at Three Currents.

Figure 3 shows the corrected mass spectral response at m/e = 14 (N), 16 (O), 28 (N_2), 30 (NO), and 32 (O_2). Corrected response means that the signals for each of the m/e ratios have had parent molecule fragments subtracted. For example, the m/e signal at 14 will have a contributions from N_2 and NO. Thus the 14 signal was corrected by subtracting the contribution due to N_2 and NO. Note in this figure how O_2 trends drop slightly as current is increased, however, it is a significant response. The source of the O_2 is suspected to be from wall recombination of oxygen atoms. Using a wall recombination coefficient of oxygen of 0.06 for copper lined sections of the probe system and 0.0005 for the inert wall probe areas results in an approximation that 70% of the oxygen atoms may be lost to wall recombination. It is estimated that this figure can be lowered to 30% by simply using the quartz sample probe.

High nitrogen atom concentrations are somewhat puzzling. The homogeneous reaction models predicted that in the presence of O atoms, N atoms would convert to nitric oxide in the probe. However, the m/e signal at 14 is significant at about 14% of the total mass spectral response even after the signal is adjusted for fragments from N_2. Thus, nitrogen atoms do reach the mass spectrometer.

An indication of nitric oxide concentrations around a shock is shown in Figure 4. Figure 4 shows the nitric oxide concentration decreasing dramatically inside a shock. An explanation for this observation is that nitric oxide dissociates across the shock to nitrogen and oxygen atoms. If the source of nitric oxide was probe reactions, one would expect that nitric oxide concentration would increase across a shock. Spectrographic work by Willey (1987) showed the primary emitter in the free stream to be nitric oxide. Thus conditions may exist where nitric oxide concentrations are above 1% in the arc jet. Mappings for other species showed the definite shock boundary, however, their concentrations were relatively constant on both sides of the shock with the exception of water. Water increased about 4 to 6 fold on the backside of the shock. This result suggests that some moisture is being pulled in from the ejector system. Further information about this project will be available in a separate report.

CONCLUSIONS

1. Sampling times of less than a second can be achieved.

2. Nitric oxide is a good probe molecule.

3. Concentration gradients occur across the nozzle and are strongest for nitric oxide.

RECOMMENDATIONS

1. Install the quartz sample probe and 1/2" teflon or nylon lines throughout the sampling system so a non-catalytic sampling system can be compared to the existing pitot tube sampling system.

2. Verify the presence and percent concentrations of nitric oxide observed at higher currents spectrographically.

3. Install pyrex orifices in place of valves and use inert valve bodies for locations where valves are necessary (solenoid valve and mass spect chamber valve).

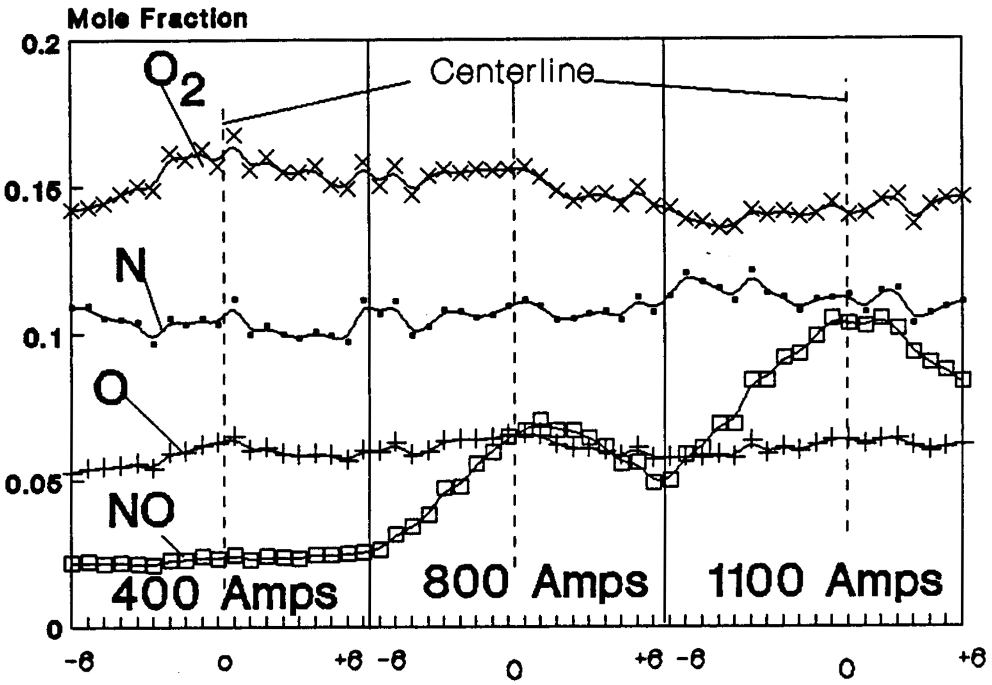

Figure 3. Mole Fractions of Oxygen, Oxygen Atoms, Nitrogen Atoms, and Nitric Oxide as a Function of Pitot Position and Current.

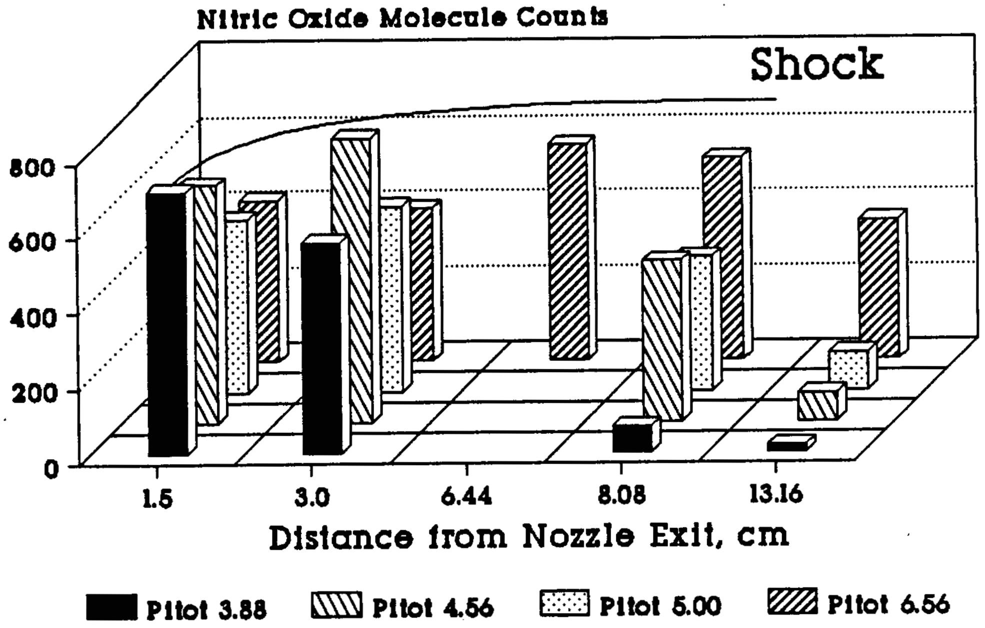

Figure 4. Mass Spectrometer Signal Counts at m/e=30 (NO) for a Shock Survey around a Blunt Body.

SECTION 2

CATALYTIC MEASUREMENTS ON HRSI AND RCC MATERIALS

BACKGROUND

This objective involved the determination of atomic recombination rates on HRSI (high temperature reuseable surface insulation) and RCC (reinforced carbon carbon) materials at various gas compositions, flow rates, and enthalpies.

Knowledge of catalytic responses on thermal protection systems are of tremendous importance in heat shield design. The difference between a non catalytic surface and fully catalytic surface influences the rate of heat transfer to the surface during a reentry manuever, and therefore, the difference in temperature measured at the surface. The ideal surface would be non catalytic i.e. atoms would strike the surface and reflect off. On the other hand, on a fully catalytic surface all atoms which strike the surface recombine to the ground state, and the energy released in this process would be transferred to the surface. Thus the surface can heat up tremendously. Demonstration of the catalytic effect on surfaces was reported for STS-2 which had 2 catalytic points on its thermal protection system (Stewart, 1981) and subsequent analysis predicted surface temperature increases of 270 K at these catalytic points (Scott, 1982).

Data analysis followed an approached developed by Scott (1981,83). The analysis begins by using simple flow conservation equations for an inviscid stream tube and frozen flow relations across the shock wave (Scott, 1983). Shock properties are determined using the measured quantities of enthalpy, stagnation pressure, and mass flowrate along with the calculated effective area taking into account the boundary-layer effect. The effective area was determined using the NATA code (Bade and Yos, 1976). Then the Fay and Riddell (1958) relation as modified by Goulard (1958) and further refined by Scott (1981) was used to generated curves for the ratio of heating rate to fully catalytic heating rate versus recombination coefficient. Since recombination can occur for both nitrogen and oxygen atoms the approach requires separating the influence of nitrogen before analyzing results for air. This is done by running tests in pure nitrogen conditions and obtaining the nitrogen recombination coefficient in terms an Arrhenius expression which would serve as an input into the air analysis. Further details about the method can be found in Scott (1981, 83).

EXPERIMENTAL

Hemispherical test articles made out of HRSI (LI-900) RCG (reaction coated glass) were place in the 5-MW arc jet chamber at JSC. The arc column consisted of 5 heater packs attached to a 2.25 inch throat which was connected to a 15^{0} solid angle nozzle with an exit diameter of 15 inches. Further details about the facility made be found in Rochelle et al. (1983). The test articles were placed in the right arm while a comparable metallic hemispherical model instrumented with slug calorimeters was placed in the left. The slug calorimeter served as a reference heating rate measurement. The heating rate was determined by measuring the rise in temperature divided by the change in time during the period the calorimeter was inserted into the stream. The constant for the calorimeter was 0.930 BTU/ft^2/^{o}F The recombination coefficients for the calorimeter were assumed constant at 0.6 for nitrogen and 0.15 for oxygen.

The procedure was:

1. Arc on.
2. Run for 2 to 5 minute period to allow gas flows and arc conditions to stabilize.
3. Swing in the left arm (calorimeter) into the stream for a period of 1 to 2 seconds.
4. Swing in the right arm (the model) into the stream wait until the stagnation point thermocouple reads constant, take a test point.
5. Swing in the pitot tube into the stream, take a test point to obtain the stagnation pressure.
6. Change to the next current setting.
7. Repeat steps 3 to 6 until full ranges have been covered.

The range of conditions studied were:

Nitrogen	0.038 kg/sec (0.08 lb/sec)	at 400 to 550 amps
11% (mass) O_2 in N_2	0.038 kg/sec (0.08 lb/sec)	at 300 to 400 amps
14% O_2 in N_2	0.056 kg/sec (0.14 lb/sec)	at 300 to 340 amps
23% O_2 in N_2 (Air)	0.038 kg/sec (0.08 lb/sec)	at 350 to 450 amps

The sequence of testing was:

7/12/88	Air - 12 points followed by Nitrogen - 1 point
7/14/88	Nitrogen - 7 points followed by 14% O_2 - 3 points
7/15/88	Air - 3 points followed by 11% O_2 -6 points followed by N_2 - 3 points

The experimental procedure included several repeat points in order to check repeatability of the results.

RESULTS AND DISCUSSION

Table 1 presents conditions tested and resultant heating rates and temperatures measured. Figure 5 shows that good repeatability could be achieved within a run, however, between runs temperatures for air conditions varied by about 60 K. Another observation made is that in the presence of 11% oxygen, the model temperatures were hotter than the Air-1 case. Heating rates also followed a similar trend. Figure 6 shows the heat flux normalized by the square root of the static pressure. In this figure one sees that the trends are uniform, however, an unexplained difficultly is why the measured normalized heating rate was about 2 times the value predicted by the Fay-Riddell correlation (shown as the straight line). The Fay-Riddell prediction was also checked through the NATA code which showed it value to be slightly higher than calculated by the catalysis program primarily because of higher predicted shock properties of temperature and pressure. It should also be noted that the NATA code calculated a Lewis number of 1.27 while the catalysis code calculated a Lewis number of less than 1 at around 0.9 (dependent on the experimental point). Lewis numbers less than 1 result when the total atomic mass fraction is high. This signifies that the ratio of diffusion transport to the surface is less then thermal transport away from the surface.

Figure 7 shows the recombination coefficient determined for nitrogen. The curve is relatively flat representing a process that has low activation energy (gas phase recombination reactions also have low activation energies). The magnitude of the results compare favorably with those reported by Scott (1981) (his values ranged from 0.014 to 0.02 in the similar temperature range). The results also compare with Kolodziej and Stewart (1987) in which they reported values from 0.008 to 0.016. Both papers reported relatively low activation energies which also compare with these results.

Figure 8 shows the recombination coefficient determined for oxygen. These curves show the difficultly of data repeatability between runs. Further, one would expect the results for the 14% O_2 to fall between Air-2 and the 11% O_2 if the Air-1 data could be thrown out for an obvious reason. Attempts to explain the day to day discrepancies included verification of voltage measurement, current measurement and water flow rates. The voltage measurement check involved connecting a Simpson meter across the supply lines. The Simpson meter measurement checked with the value measured by the Norbitrol. Current calibrations were done several times with no major discrepancies found. Finally, the water turbine meter position was changed so that no sudden flow disturbance occurred upstream. No flow change was observed for this correction. In a review of arc jet data, the only major discrepancies was with the oxygen supply pressure and actual flowrate. However, the product of these two were the same for both air runs as it should be. Further, the column pressures were very similar indicating that the arc jet column conditions were identical. So what explanations remain to be explored? Surface change is the most likely candidate. Perhaps emissivity changes or perhaps nitrogen changes. Another explanation may be thermocouple relocation, however, two independent nitrogen runs were repeatable. Another possibility may be the model location was not repeatable and this should be explored further. In summary, further testing is required.

TABLE 1

SUMMARY OF CONDITIONS STUDIED FOR CATALYTIC RECOMBINATION ON RCG COATED HRSI MATERIAL

Run Number	Gases	Flowrate kg/sec	Current amps	Enthalpy MJ/kg	Calorimeter W/cm^2	Surface Temp. K
2-749-1	Air	0.0368	350	7.31	50.66	1448.6
2-749-2	Air	0.0368	376	7.75	56.06	1467.5
2-749-3	Air	0.0368	403	8.20	60.08	1497.3
2-749-4	Air	0.0368	426	8.66	63.72	1518.0
2-749-5	Air	0.0368	450	9.11	64.42	1539.8
2-749-6	Air	0.0368	451	9.14	64.95	1540.1
2-749-7	Air	0.0368	425	8.71	60.12	1527.1
2-749-8	Air	0.0368	401	8.25	56.59	1500.5
2-749-9	Air	0.0368	376	7.80	52.53	1485.8
2-749-10	Air	0.0368	349	7.34	47.57	1458.0
2-749-11	N_2	0.0363	402	7.89	42.20	1349.8
2-751-1	N_2	0.0381	400	7.65	53.74	1410.7
2-751-2	N_2	0.0381	451	8.46	60.07	1460.0
2-751-3	N_2	0.0381	501	9.13	61.81	1490.2
2-751-4	N_2	0.0381	524	9.42	66.88	1506.0
2-751-5	N_2	0.0381	550	9.83	68.21	1523.9
2-751-6	N_2	0.0390	500	9.04	64.44	1493.2
2-751-7	N_2	0.0368	400	7.74	50.08	1411.6
2-751-8	14% O_2	0.0558	301	5.25	46.95	1468.0
2-751-9	14% O_2	0.0563	326	5.64	49.86	1508.7
2-751-10	14% O_2	0.0563	341	5.84	53.48	1529.2
2-753-1	Air	0.0359	351	7.43	61.18	1502.3
2-753-2	Air	0.0359	374	7.80	66.56	1527.1
2-753-3	Air	0.0359	401	8.30	69.21	1557.0
2-753-4	11% O_2	0.0368	300	6.45	51.96	1455.9
2-753-5	11% O_2	0.0368	326	6.97	52.94	1479.1
2-753-6	11% O_2	0.0368	351	7.35	58.24	1502.1
2-753-7	11% O_2	0.0368	376	7.79	60.89	1521.9
2-753-8	11% O_2	0.0395	400	7.77	65.07	1552.2
2-753-9	11% O_2	0.0372	326	6.89	51.54	1485.9
2-753-11	N_2	0.0368	400	7.69	50.92	1402.2
2-753-12	N_2	0.0368	501	9.32	67.50	1495.7
2-753-13	N_2	0.0368	550	10.05	71.48	1536.1

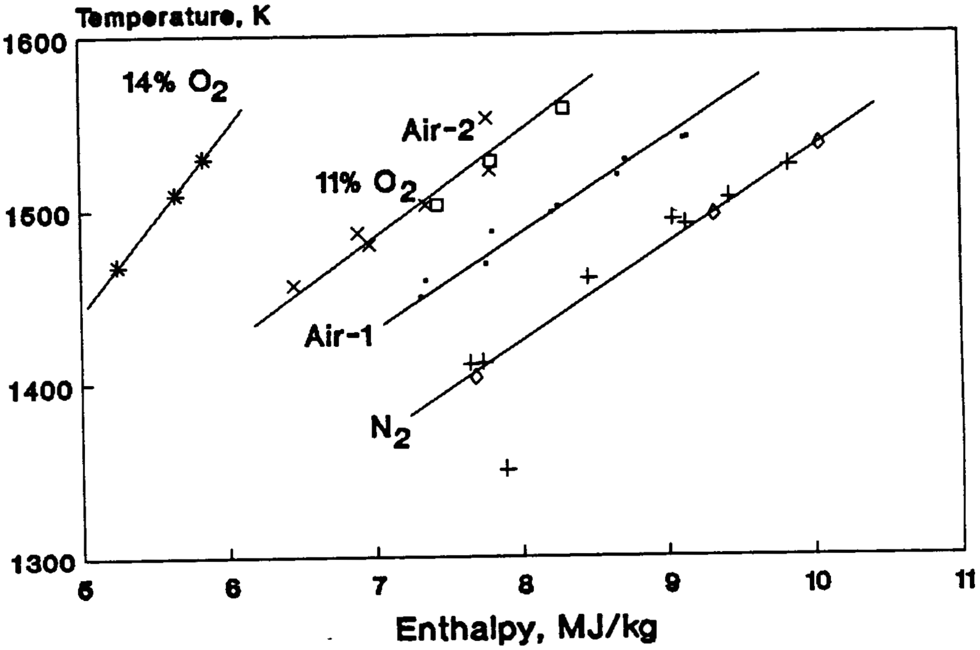

Figure 5. Surface Temperatures Measured at the Stagnation Point of an HRSI RCG Coated Hemisphere as a Function of Enthalpy for all Test Conditions.

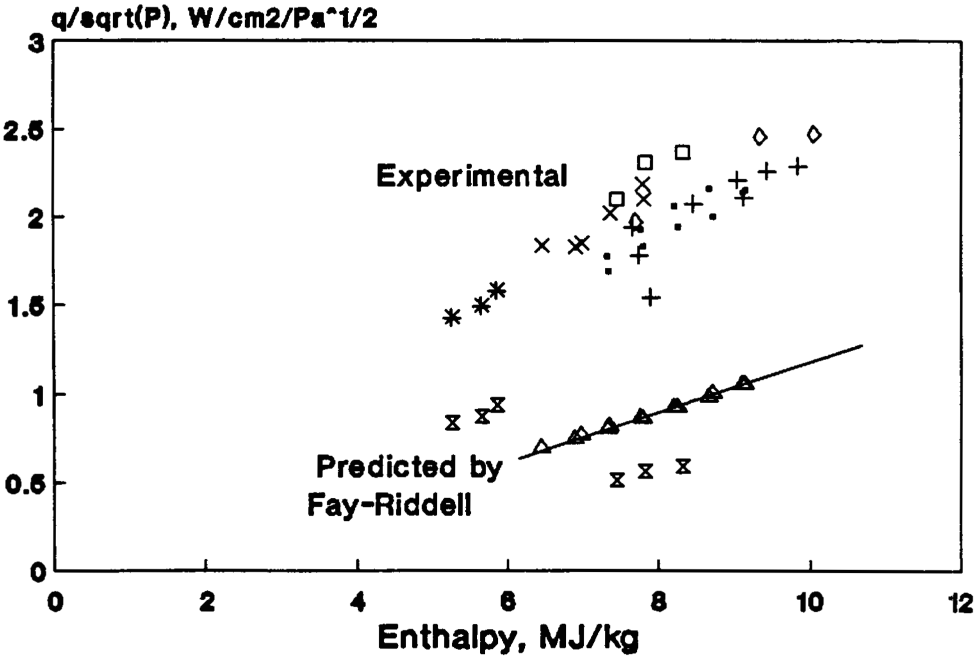

Figure 6. Normalized Heat Flux Measured as a Function of Total Enthalpy.

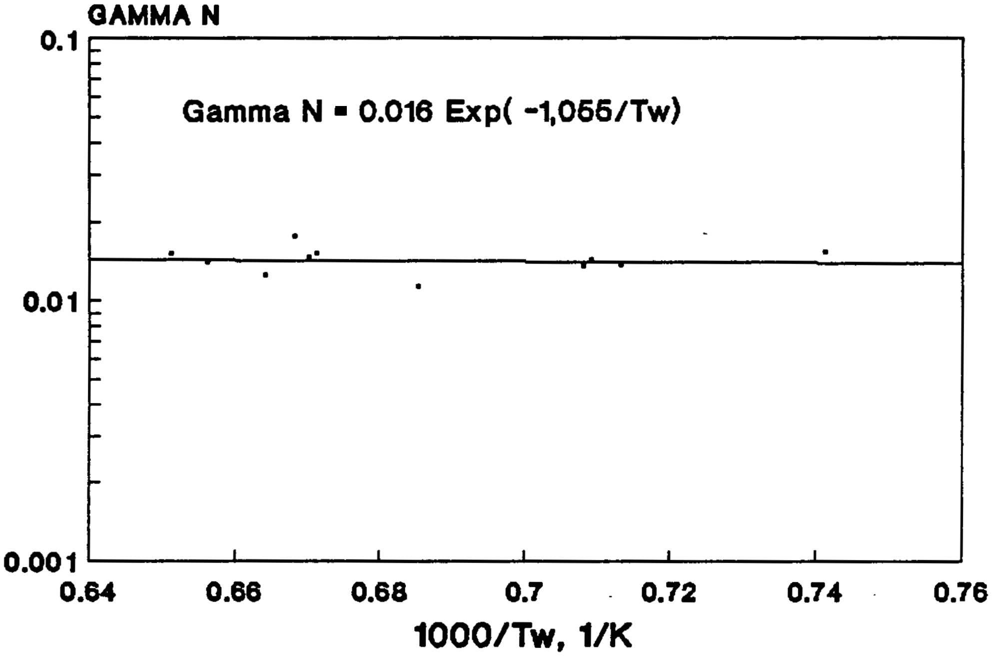

Figure 7. Nitrogen Recombination Coefficient as a Function of Temperature - Arrhenius Plot.

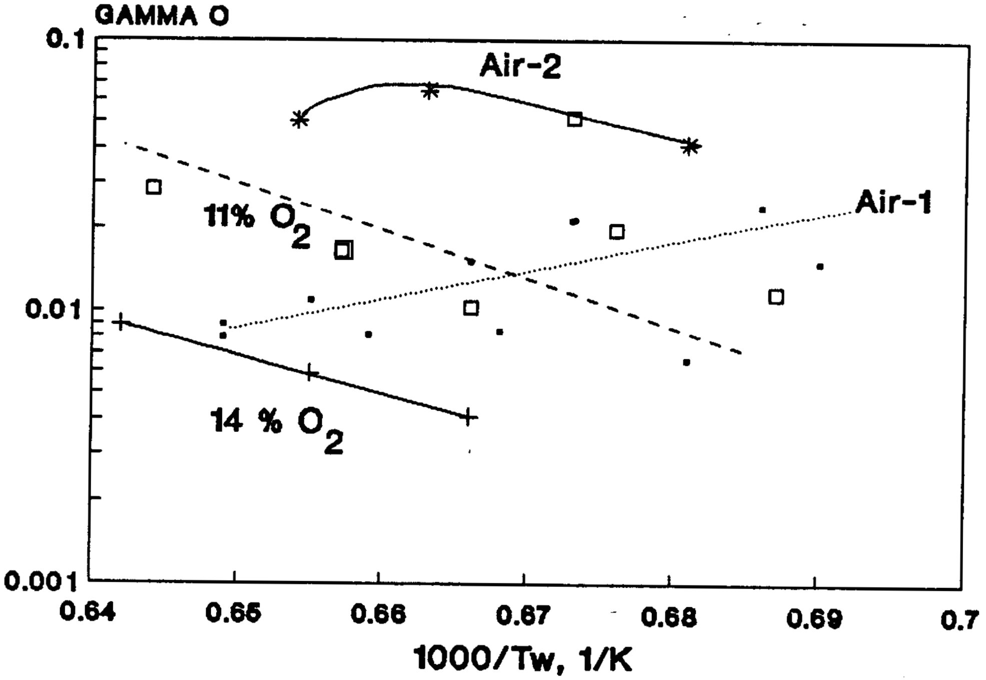

Figure 8. Oxygen Recombination Coefficient as a Function of Temperature - for Various Run Conditions.

After the test program was completed, the surface of the slug calorimeter was analyzed by energy dispersive X-Ray spectroscopy (EDAX). The results showed that the silver coating agglomerated on the surface exposing the copper surface underneath. Further elemental analysis showed that the small clumps of were primarily composed of silver with a trace of oxygen and copper. The valley areas were primarily composed of copper with some oxygen, however, the oxygen level was not high enough to represent cuprous oxide or cupric oxide on the surface.

CONCLUSIONS

1. HRSI - RCG coated thermal protection materials are relatively inert to nitrogen atom recombination.

2. Repeatability within runs was possible, however, outside of runs, results were not as repeatable.

3. The scatter of data observed between runs for oxygen recombination experiments cannot be attributed to differences in voltage, current, or water flowrate measurement errors.

3. The silver coated calorimeters changed surface structure morphology during the test program with silver agglomerates forming on the surface.

RECOMMENDATIONS

1. Continue experimental measurements. Temperature ranges between 1600 and 1800 K should be investigated.

2. Attempt catalytic measurements on RCC (Reinforced Carbon Carbon) materials.

3. Determine why the surface temperature differed by about 60 K for identical arc heater conditions between the two air runs.

4. Include emissivity measurements in all catalysis test programs.

5. Insure that all arc jet measurements are reliable before testing.

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