Mass Spectrometric Study of Various Coated Targets Utilizing PUPR-MC Plasma Machine for NASA Solar Probe Space Mission

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ABSTRACT

The NASA Solar Probe Space Mission will be a historic operation, flying for the first time into one of the last unexplored regions of the solar system, the Sun's atmosphere or corona; hopefully revolutionizing our knowledge of physics regarding the origin and evolution of the solar wind phenomenon. One of the spacecraft's most prominent features is the Thermal Protection System (TPS), composed of a large carbon-carbon conical shield, designed to withstand the Sun's violent temperatures. Thermal testing was performed on various coatings on the carbon-carbon targets in order to study mass loss components using mass spectrometry. Mass spectrometry is an analytical technique used to measure the mass-to-charge ratio of ions. The composition of a gaseous sample is found by generating a mass spectrum where the masses of the elements present in the sample. Using a quadrupole mass spectrometer, the effects of the exposition to low density plasma on various coated targets were analyzed at PUPR Mirror/Cusp (PUPR-MC) plasma machine. A series of five tests were performed for this experiment. The first four tests consist in creating plasma with four different gases, and studying the effects of each gas on plasma using mass spectrometry, in order to decide which plasma resembles the Sun's atmosphere or corona; the gases utilized were: (1) residual gas, (2) argon, (3) nitrogen, and (4) hydrogen. The fifth test consists in the introduction of various coated targets, representing the spacecraft's shield, inside PUPR-MC plasma machine for approximately twelve hours, to study the reaction of the coatings to the plasma best resembling the Sun's atmosphere. After studying the first four tests results, it is evident that each gas has a distinctive effect on the plasma. For the fifth test following the study of the mass spectrometry results, it is clear that the quadrupole mass spectrometer was able to detect mass loss components for the introduced targets, and the presence of the coatings were successfully identified inside PUPR-MC plasma machine, therefore assisting in the shield coating selection for the Solar probe aircraft.

I- INTRODUCTION

***Figure 1:** Solar Probe Mission*

Solar Probe will be a historic mission, flying for the first time into one of the last unexplored regions of the solar system, the Sun's atmosphere or corona. Approaching as close as 3 RS above the Sun's surface, Solar Probe will employ a combination of in-situ measurements and imaging to achieve the mission's primary scientific goal: to understand how the Sun's corona is heated and how the solar wind is accelerated. Solar Probe will revolutionize our knowledge of physics regarding the origin and evolution of the solar wind. Moreover, by making only direct, in-situ measurements of the region where some of the deadliest solar energetic particles are energized, Solar Probe will make unique and fundamental contributions to our ability to characterize and forecast the radiation environment in which future space explorers will work and live. The Solar Probe Mission will revolutionize our knowledge of physics regarding the origin and evolution of the solar wind, by making the only direct, in-situ measurements of the region where some of the deadliest solar energetic particles are energized. The baseline Solar Probe is a 3-axis stabilized spacecraft designed to successfully survive and operate in the intense thermal environment that it will encounter during its voyage around the Sun. The spacecraft's most prominent feature is the Thermal Protection System (TPS), comprising a large 2.7-m diameter carbon–carbon conical primary shield with a low-conductivity, low-density secondary shield attached to its base. The TPS protects the spacecraft bus and instruments within its umbra during the solar encounter. The bus consists of a hexagonal equipment module and a cylindrical adapter. It provides an efficient mechanical structure that accommodates the instruments and spacecraft subsystems and handles the loads from the TPS and the launch loads.

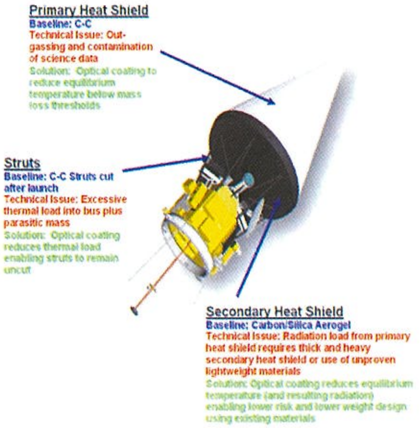


***Figure 2:** Solar Probe Aircraft*

A mass spectrometer allows identifying masses of individual atoms and molecules that have been converted to ions from a given sample. This technique is unique because it provides fingerprint identification for the structural and chemical properties of these molecules. Among the instruments used in research to identify isotopic composition, we can mention the magnetic mass spectrometer, the Wien filter, the visual light spectrometer and the quadrupole mass spectrometer. The quadrupole mass spectrometer used in this experiment, one of a specialized subset of mass spectrometers, able to measure background gases in a vacuum chamber, is shown in Figure 3.

Figure 3: *Dycor Dymaxion Quadrupole mass spectrometer*

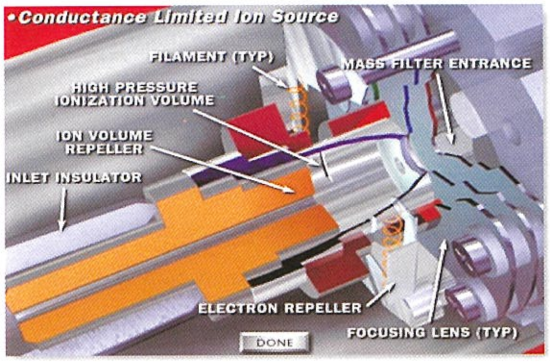


Figure 4: *Closed Ion Source (Ionizer)*

A mass spectrometer is comprised of three systems:

(1) Sampling System
(2) Mass Spectrometer Hardware
(3) Data System

The sampling system serves as a connection between the outside sample environment and the vacuum environment required by the mass spectrometer. Once the sample reaches the mass spectrometer hardware, three processes take place:

(a) Ionization
(b) Separation
(c) Detection

During ionization, sample molecules are tuned into ions, which are then focused towards the quadrupole to be detected. The process occurs in the ionizer (Figure 4), which consists of a filament, filament electron repeller, ionizer body, ion volume and two focusing lenses. The filament produces electrons, as current flows through the filament, it is electrically heated to incandescence towards the ionizer body by the potential difference in the filament and the ionizer body, and the electrons collide with the sample in the center of the ion volume and create ions. Once the positive ions are formed, they are extracted from the ion region towards the quadrupole mass filter by a difference in potential. Lens 1 has an applied negative voltage that, due to a difference in electrostatic potential, attracts the newly formed positive ions, passing them though lens 2. Lens 2, in turn, focuses the electrons into the quadrupole. Once the ions reach the quadrupole mass filter, separation occurs. The ions are filtered according to their mass-to-charge (m/z) ratio. Each ion has an identifiable mass. The quadrupole mass filter is built of four electrically-conducting parallel cylindrical rods. A constant direct current (DC) voltage and an alternating radio frequency (RF) voltage are applied along the length of the rods. Through proper electronic tuning, these voltages set the criteria for the ions that pass through the quadrupole. Ions that successfully pass through the quadrupole are again focused towards the detector using an exit aperture, which has an applied negative voltage that attracts the positively charged ions. The simplest detection setup consists of a Faraday cup detector. A simple description on the performance of an ideal Faraday Cup can be found in: DiDlippo, F., & et, a. (1994), Accurate Atomic Masses for Fundamental Metrology.

Phys Rev Lett., 73 (15). An electron multiplier is used for amplified sensitivity. A Faraday cup detector is a closed structure except for an opening that allows the ions to enter. As the positive ions exit the quadrupole mass filter (Figure 5) striking the detector, a current is created. This current is sent to the preamplifier for amplification and then to the data system for display. The data system keeps the overall control of the system, and performs the data acquisition. Access to collected data is accomplished through the Dycor Process 2000 software shown in Figure 6. The adjustments of all instrument parameters affecting the sampling, ionization, separation and detection of the ions, are software controlled. All data acquisition parameters are also set using the software.

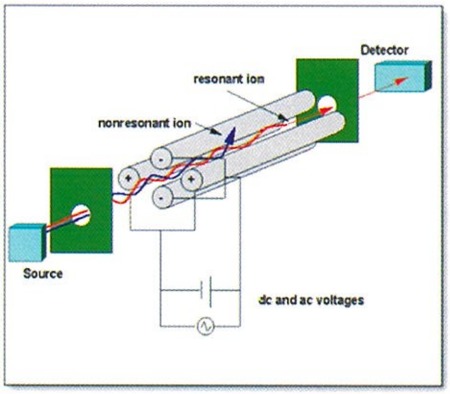
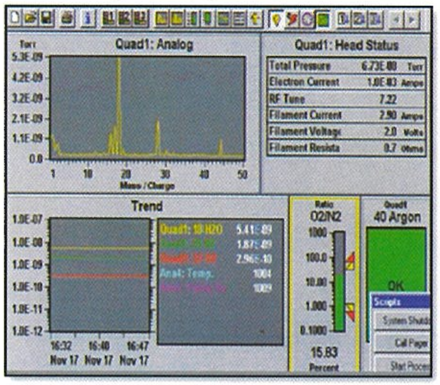


Figure 5: *Quadrupole Mass Filter*



Quad1: Head Status

Total Pressure	6.73E-08	Torr
Electron Current	1.0E-03	Amps
RF Tune	7.22	
Filament Current	2.90	Amps
Filament Voltage	2.0	Volts
Filament Resista	0.7	Ohms

Quad1: 18 H2O	5.41E-09
[illegible]	1.87E-09
[illegible]	2.96E-10
Anal: Temp.	1004
[illegible]	1009

Figure 6: *Dycor Process 2000 Software*

PUPR-MC plasma machine (Figure 7) has the advantage of producing high density ion concentration plasma at regularly low temperatures. A series of five tests have been performed for this experiment in order to study the behavior of the plasma created by diverse gases.

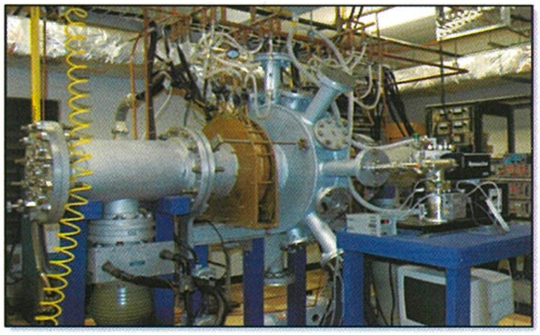


Figure 7: *PUPR-MC Plasma Machine*

The first four tests consist in creating plasma with four different gases, and studying the effects of each gas on the plasma using mass spectrometry, in order to decide which plasma resembles the Sun's atmosphere or corona; the gases utilized were:

(1) Residual Gas
(2) Argon (Figure 8)
(3) Nitrogen (Figure 9)
(4) Hydrogen (Figure 10)

Figure 8: *Argon Plasma*

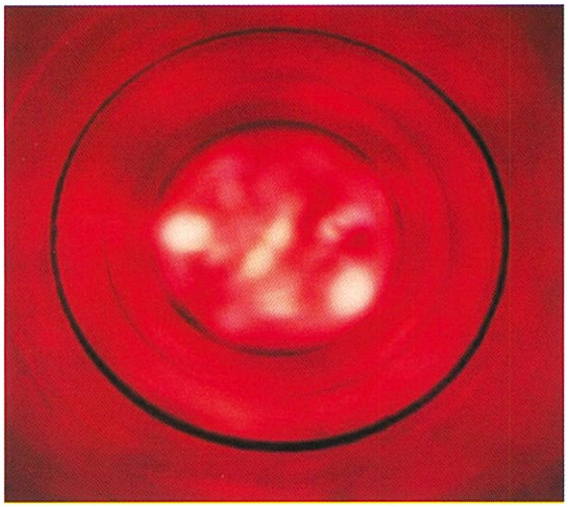


Figure 9: Nitrogen Plasma

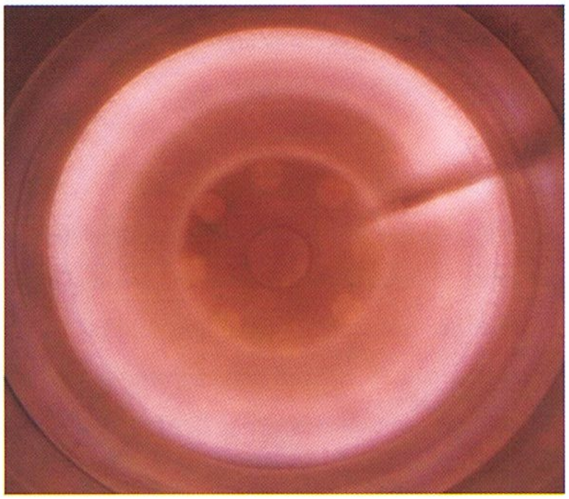


Figure 10: Hydrogen Plasma

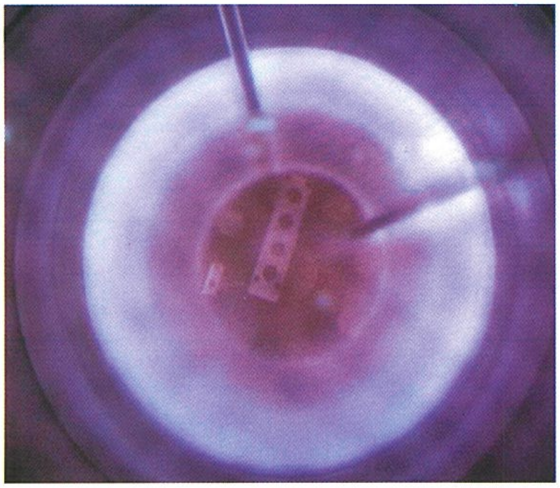


Figure 11: Coated Targets Exposed to Hydrogen Plasma

The fifth test (Figure 11) consists in introducing various coated targets inside PUPR-MC plasma machine, to study how the different coatings react to the plasma that best resembles the Sun's conditions for an exposure of approximately twelve hours. Treatment can also be done for shorter periods of time and the same effects can be reproduced, however, different ion densities have to be used.

II- PROCEDURE

PUPR-MC plasma machine chamber used for this experiment is 3m long with two side cylinders with 30cm of radius, and a central cylinder with 50 cm of radius. Two mechanical pumps are used to bring the chamber pressure to $1x10^{-3}$ Torr, a stage referred to as pre-vacuum. Once the pre-vacuum is complete, two diffusion pumps are activated to obtain high vacuum, obtaining pressures between 10^{-6} and 10^{-8} Torr. When high vacuum is obtained, the power supply is turned on, and a current of approximately 400 A is passed through a pair of Helmholtz coils (each one separated 15cm from the center of the plasma chamber) to create a magnetic field either in Mirror or in Cusp mode. A high power microwave signal is then applied to ionize the gas in the chamber creating plasma. The microwave power is oscillating at 2.45 GHz, the electron cyclotron resonance frequency when the magnetic field surface in the machine is larger than 875 Gauss. Once this task is completed and the mass spectrometer has completed its pre-vacuum and warm-up sequence, the desired settings for each test are programmed utilizing the Dycor Dymaxion 2000 software. The mass spectrometer capillary valve (Figure 12) is then opened to commence the sampling. The data acquired is then stored and converted, also using the Dycor Dymaxion 2000 software, so can later be tabulated, graphed and analyzed using any spreadsheet software.

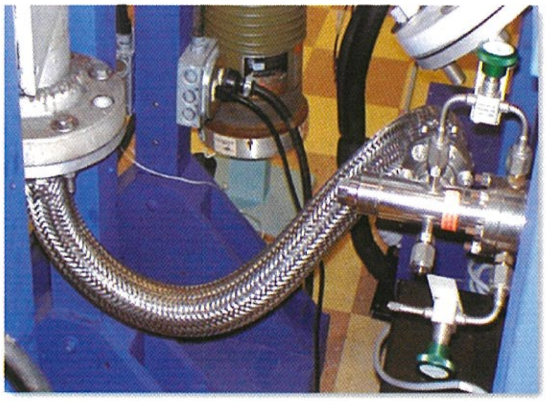


Figure 12: Capillary Valve

For the fifth test, a holder was manufactured to place the coated targets (shown in Figure 11), to be later mounted to the end of an electrostatic probe. By placing a negative voltage bias on the holder in the form of a probe (Figure 13), high energy ions are attracted and immersed into the metallic matrix microns into the surface of the coated target, consequently attracting the plasma. This potential maintains a constant surface temperature, and is applied for approximately twelve hours. The plasma density is directly proportional to the saturation current of the ions and inversely proportional to ion temperatures. These ion temperatures vary with the intensity of the magnetic field of the machine, and the frequency of the microwaves that create electron resonance in the machine chamber.

The mounting system used to treat the stainless steel samples in the machine uses a 3/8" diameter stainless steel tube. A micro-coaxial cable runs through the tube, and it is attached to a screw, which is in direct contact with the target holder. Liquid ceramic is used to insulate the probe structure and avoid short circuits created by the plasma field. The back of the probe is closed with a stainless steel flange with BNC connections to the power supply.

Once the experimental setup is complete, the Dycor process 2000 software is programmed to store data every 10 minutes during the twelve hours exposure. Data is then analyzed to search for any mass loss components on the coated targets.

III- RESULTS

Once all five test were completed, the data was converted in order to be tabulated and graphed using a spreadsheet software. In the following Figures 14, 15, 16, 17, and 18, a set of mass spectra for each of the plasmas mentioned above is illustrated.

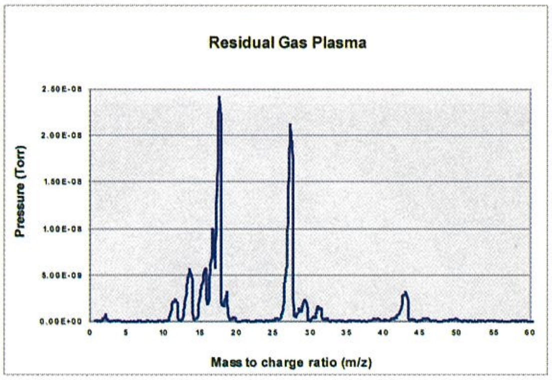


Figure 14: Residual Gas Plasma

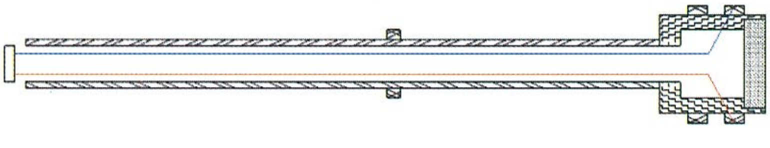


Figure 13: Probe Schematic Utilized in Experimental Setup

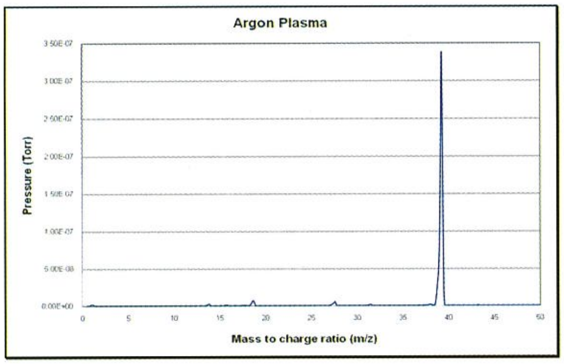


Figure 15: Argon Plasma

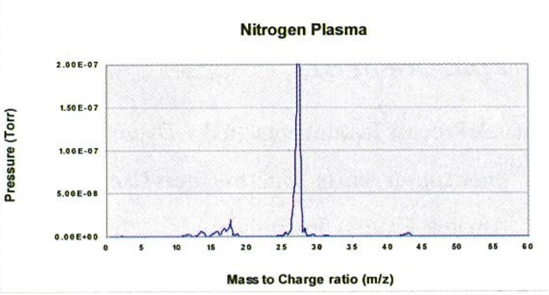


Figure 16: Nitrogen Plasma

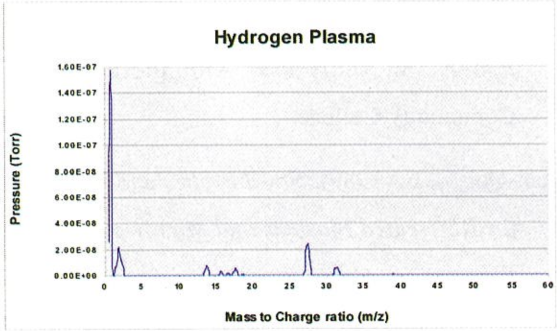


Figure 17: Hydrogen Plasma

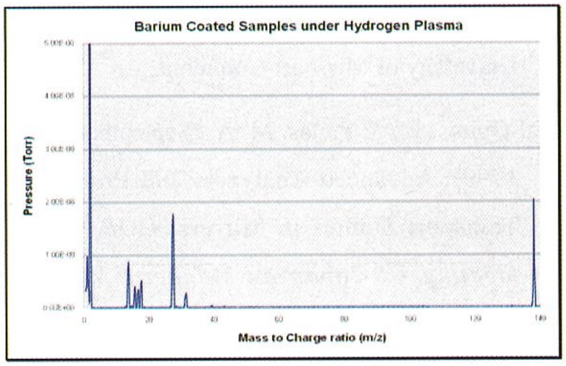


Figure 18: Coated Sample under Hydrogen Plasma Spectrum

The mass spectrometry data showed that various elements were present for the residual gas plasma (Figure 14), the most notable being nitrogen with a mass-to-charge (m/z) ratio of approximately 28, and water vapor with a mass-to charge ratio of 18. These elements are present due to vacuum leaks in PUPR-MC plasma machine, where nitrogen can be found in atmospheric air and water vapor is abundant due to the level of humidity found at the machine location. This data served as comparison base for the other plasma samples. From the argon plasma (Figure 15), which has a mass-to-charge ratio of 40, it can be observed that the presence of water vapor and nitrogen went down significantly. This is because the argon plasma high temperature most likely evaporated most of the other elements existent inside the plasma chamber. The nitrogen plasma spectrum can be appreciated in Figure 16. The nitrogen plasma approximately maintained the same levels of water vapor as the argon plasma, but it tends to becomes somewhat unstable. Hydrogen plasma (Figure 17), on the other hand, was the most stable plasma throughout this experiment, and due to the high energy required to ionize this gas, it produced the highest temperatures, nearly eliminating any impurities found inside PUPR-MC plasma machine. Even though nitrogen was present in the spectrum, something interesting was observed during the hydrogen plasma testing: when hydrogen was being injected into the chamber, nitrogen levels were present in the chamber for a longer period of time compared to the previously mentioned plasmas. This behavior is normally due to residual N_2 gases that remain in the chamber until it is entirely pumped out. The reason for this behavior is currently under investigation, and further tests will have to be performed in order to study this anomaly. The fifth test consisted in introducing various coated targets inside PUPR-MC plasma machine while monitoring any mass loss from the coated samples. The gas chosen for this experiment is hydrogen, due to the high energy readings obtained in previous tests. As an example, Figure 18 shows the spectrum of one of the coated samples submitted to hydrogen plasma after approximately six hours of exposure.

IV- CONCLUSIONS

In conclusion, the Dycor Dymaxion mass spectrometer effectively identified the argon, nitrogen and hydrogen gases. It was also successful in identifying the mass loss components on the coated targets. The effects of each gas on the plasma could be appreciated from the mass spectrometry results illustrated above. Argon plasma was better at eliminating chamber nitrogen impurities. Nitrogen plasma was the least stable of all the plasmas created, and further study and analysis will be performed to correct this problem. The plasma with the highest temperature and stability was hydrogen plasma, which was able to eliminate nearly all impurities except for nitrogen. For the fifth test, it is clear that the quadrupole mass spectrometer was able to detect mass loss components for the introduced targets, and the presence of the coatings were successfully identified inside PUPR-MC plasma machine, therefore greatly assisting in the shield coating selection for the Solar probe aircraft.

V- ACKNOWLEDGEMENTS

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