TITLE: Overview of Characteristics and Experiments in IPM Plasmatrons

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ADP010736 thru ADP010751
OVERVIEW OF CHARACTERISTICS AND EXPERIMENTS IN IPM PLASMATRONS

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ABSTRACT

Characteristics of IPG series plasmatrons as well as there application for reentry simulation and testing of thermal protection materials are discussed in the presented paper on the basis of 35-years experience of Plasma Laboratory of IPM RAS. Successful application of plasmatrons for simulation of reentry conditions and testing of thermal protection materials is based on using of plasmatron’s advantages such as purity of plasma flow, its high stability, excellent reproducibility as well as wide ranges of realized pressure and heat flux. Using of subsonic regimes together with plasmatron’s ability for independent smooth regulation of regime parameters such as pressure and power injected in plasma make a plasmatron the most flexible and powerful instrument for simulation of thermochemical action of shock layer on the surface of descent space vehicle, especially when it is necessary to solve problems concerned radiative & convective heating and/or non-equilibrium heat transfer. To the present day long-term aging tests (up to 100 15-minutes testing cycles for one sample) in dissociated gas flow were fulfilled only using plasmatrons. Also there are discussed tests of ablative thermal protection materials, studies of thermo-chemical interaction between dissociated flows and reusable thermal protection materials as well as some «non-space» applications of plasmatrons as deposition of diamond films and testing of industrial materials on heat shock.

1. INTRODUCTION: ADVANTAGES OF INDUCTION PLASMATRONS

In world practice gas heating by electric energy, enjoy wide application for the simulation of re-entry conditions to test reusable thermal protection materials. However traditional methods like ohmic heating and electric arc heating don’t give needed performance characteristics. Ohmic heaters do not allow to reach high temperatures and electric arc heaters pollute gas flow by products of electrodes erosion. On the contrary induction method of gas heating most completely fits the requirements of simulation of physical parameters of hypersonic vehicles aerodynamic heating.

Discharge device of a plasmatron includes two main elements – quartz tube, that is used as discharge channel and induction coil (inductor), on which runs high-frequency current, that induces circular currents in discharge. In such device there are no electrodes, which is the main source of flow contamination and disturbances in arc-jet facilities. Discharge in subsonic flow of air, discharge channel and inductor of the powerful plasmatron IPG-3 are presented in Fig. 1.

Figure 1. Inductively coupled plasma in the IPG-3 plasmatron (discharge channel diameter ~ 200 mm).

In the plasmatrons of IPG series hot gas does not contact with discharge channel wall due to flow vortex in discharge channel and that ensures high purity of plasma flow. Also flow vortex makes discharge very stable in wide range of pressure and discharge existence is not limited in time.

Induction plasma generators have a high ability to simulate aerodynamic heating of hypersonic vehicles. Due to using contactless heating of gas flow by induction current, these facilities create exceptionally pure plasma flow of any gases also they show high stability and excellent reproducibility of regimes. Last but not least advantage of a plasmatron is the feasibility to make long-term aging tests in real time. Taken together, these advantages make induction plasma generators best suited to fulfill aging tests of reusable thermal protection materials. Besides, using of pure subsonic plasma jets allows to simulate most precisely thermochemical action of shock layer plasma on thermal protection materials of descent space vehicles. In particular, plasmatrons give an opportunity to
simulate flight conditions such as total enthalpy and pressure, temperature and concentrations profiles within boundary layer and hence heat flux and surface temperature.

2. PLAMATRONS OF THE IPG SERIES: STEP BY STEP

During the last 35 years four induction plasma generators of IPG-series have been created in IPM under the supervision of Dr. M. Yakushin. Electrodeless high frequency discharge in gas flow was used in these facilities for generation of subsonic and supersonic high enthalpy jets of air and other gases. The progress of the IPG plasmatrons followed the directions of the increase of power, discharge channel diameter, total pressure range, making it possible to use different gases. Table 1 shows main characteristics of IPG facilities, which were in use previously (IPG-1, IPG-2) and which are in operation now (IPG-3, IPG-4). Compared to first generation, existing plasmatrons increase 15 times in power, 3 orders of magnitude in pressure range.

The following technical problems were solved during the creation of the IPG facilities:
- a compromise between energy and gas dynamics parameters was established;
- excellent stability and reproducibility of operation regimes were achieved;
- tube generator was agreed upon plasma load;
- quartz discharge channels and gas injection system were designed to withstand operation without any forced cooling;
- effective methods for plasma ignition were developed.

One of the main aims of IPG series development was to ensure high working ability and high reliability in operation for all parts and elements of facilities due to using of reliable approaches which are as simple as possible as well as schemes and principles verified by experience. Vertical orientation of discharge channel with plasma flow directed upward has been applied for all four facilities and it allowed to avoid problems concerned operation at high pressures where Archimedean forces disturb discharge oriented horizontally but gasdynamic stabilization is not so effective as at low pressures. The problems were solved successfully to make a choice of optimal conceptual sketches, materials for manufacturing and commercial equipment of general use. Units and elements of facilities have been designed to keep high reliability under heavy duty operation conditions including joint action of high temperature, chemically active gases, low pressure, significant radiative & convective heat fluxes, high-frequency electromagnetic fields, which are attended by phenomena caused by skin-effect.

To secure the safe working conditions for maintenance personnel reliable means of protection have been designed in the facilities of IPG series.

Particular attention has been given to IPG facilities universality, which allows among other things quick going from subsonic to supersonic operation mode and vice versa. The time of this conversion is about a few minutes. So samples and models could be changed quickly between testing cycles during aging tests of thermal protection materials and full-scale design elements.

The IPG-1 facility was used for the simulation of convective & radiative heat transfer from chemically equilibrium boundary layer to the surface at atmospheric pressure. Ablative materials for reentry vehicles «Voskhod», «Soyuz», «Zond–4», «Luna–16» were investigated at the intensive blowing of vapor into initial air plasma flow [1-8]. Also the first into initial air plasma flow [1–8]. Also the first

<table>
<thead>
<tr>
<th>Table 1. Main parameters of the IPG plasmatrons</th>
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<tr>
<td>Electric power supplied to plasmatron, kW</td>
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<tr>
<td>Generator anode power supply, kW</td>
</tr>
<tr>
<td>Frequency, MHz</td>
</tr>
<tr>
<td>Discharge channel, mm</td>
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<tr>
<td>Snagnation pressure, atm</td>
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<tr>
<td>Gas mass flows rate, g/s</td>
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<td>Working gases</td>
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experiments with planet atmospheres as working gases were carried out using this facility. The outward appearance of the IPG-1 is shown in Fig.2.

![Figure 2. The outward appearance of the IPG-1 plasmatron (1963-1977).](image)

Expanded programme of testing was realized in the IPG-2 during Buran programme and samples (30 mm diameter) of black ceramic tile [9-11] and different types of carbon-carbon materials with antioxidative coatings were subjected aging tests with duration up to 100 cycles. 5 coatings for ceramic tile and about 50 coatings for carbon-carbon materials were put through the IPG-2 facility. Non-equilibrium convective heat transfer from dissociated air, oxygen and nitrogen flows to thermal protection materials was studied using the IPG-2 at pressures $P = 0.05-1.0$ atm and main results on catalycity of Buran’s thermal protection materials were obtained using this facility [9, 12-16]. The outward appearance of the IPG-2 is shown in Fig.3.

![Figure 3. The outward appearance of the IPG-2 plasmatron (1977-1989).](image)

The main problem solved using the IPG-3 facility is laboratory testing of full-scale ceramic tile and other full-scale design elements under conditions corresponding to the peak heating part of “Buran’s” re-entry trajectory [9-11, 17].

The IPG-3 plasmatron of 1 MW class is the most powerful facility of the IPG series. Its outward appearance is shown by Fig.4.

![Figure 4. The outward appearance of the IPG-3 plasmatron (1983-....).](image)

The creation of the IPG-4 facility introduces radical alteration in the plasmatron technology of IPM. The IPG-4 facility fully corresponds to modern requirements in performances and potentialities, and permits to solve wide spectrum of problems in experimental high temperature gas dynamics. The fundamental difference of IPG-4 from preceding facilities of the IPG series is that the opportunity was realized for the first time to obtain stable high-enthalpy jets in the pressure range $P = 0.01-1.0$ atm at constant gas enthalpy. Pressure and enthalpy ($H = 10-40$ MJ/kg) can be regulated smoothly. It allows to vary the degree of the disequilibrium of dissociated boundary layer on the model from frozen to equilibrium. It makes it possible to extend the area of
trajectory parameters simulation for the hypersonic vehicles in existence and under consideration.

Up to now the IPG-4 plasmatron is «working horse» of Plasma Lab. Due to its universality and low cost of exploitation the IPG-4 is in operation practically every day. It was used to make test campaigns for ESTEC/ESA, Aerospatiale, SEP and many others as well as to study surface catalytic and non-equilibrium heat transfer in different dissociated gases [18-25]. The outward appearance of the IPG-4 is given in Fig.5.

![Figure 5. The outward appearance of the IPG-4 plasmatron (1989-....).](image)

3. MODERN PLASMATRONS OF THE IPG SERIES

The IPG-3 and the IPG-4 plasmatrons meet modern requirements to facilities for the testing of hypersonic vehicle thermal protection. These requirements are derived from the necessity to work on the following problems:

- the investigation of the thermochemical stability of thermal projection materials under conditions modeling aerodynamic heating;
- the determination of the catalytic properties of surfaces at high temperatures;
- the study of ablative materials including intensive radiative & convective heating at high pressures (up to 1 atm and higher);
- the experimental and numerical studies of plasma flow in
  * discharge channels of induction plasma generators;
  * sub- and supersonic non-equilibrium high enthalpy jets;
  * dissociated boundary layers over models;
- synthesis of new coatings in high enthalpy jets of carbon-hydrate- containing gases by using chemical vapor deposition (CVD).

The IPG-3 and the IPG-4 plasmatrons, which are in operation now, differ in their technical potentialities and it leads to different application.

The IPG-3 facility is used to solve problems of practical character, i.e. testing of working capacity of large-scale samples of thermal protection materials and full-scale (150-500 mm) elements of thermal protection system of «Bor», «Buran» and advanced hypersonic vehicles as follows. High-enthalpy air flows were used for testing of full-scale ceramic tile with its real fastening to skin imitator (100 testing cycles of 10-minutes duration of each cycle at T = 1250°C), ceramic tile in simulated non-standard situations, full-scale part of man-hole cover with window, light-weight structures of carbon-carbon with antioxidative coating (200 x 200 mm, 2 mm thickness) as well as large samples of thermal protection materials (diameter ~100 mm).

The IPG-4 application is directed to research works, for example, non-equilibrium heat exchange between high-enthalpy dissociated flows and different surfaces such as metals, quartz, ceramics, carbon-carbon materials with antioxidative coatings. Database was created on catalytic properties of thermal protection materials. Also thermochemical stability of thermal protection materials have been studied including determination of maximum working temperatures of TPM, making aging tests, studies of surface degradation together with changes of surface emissivity and catalyticcity as the functions of temperature and time. Comparative study was made on Buran's materials behavior in different dissociated gas environments.

Consider the design of the IPG-4 facility. Schematic diagram of test chamber in Fig.6 (see next page) and schematic diagram of IPG-4 facility with its infrastructure and scientific equipment are shown in Fig.7.

Test chamber consists of low pressure test chamber shell, inductor chamber, induction heater, fast positioning device, instrument positioning device, water-cooled shields.

Test chamber shell is designed to produce and to maintain specified pressure under the effect of high temperature and chemically active gas environment, as well as to protect maintenance personnel. The shell of cylindrical form is made one-sectional. It is equipped with elliptic bottoms, man-holes with corresponding covers, mating units, optical windows and lead-in for high frequency feeder.
**Figure 6.** Conceptual sketch of the IPG-4 facility. 1 - test chamber, 2 - load matching unit of RF-generator, 3 - RF-generator unit, 4 - main control panel, 5 - control panel of cooling system, 6 - plasma-water heat exchanger, 7 - vacuum line, 8 - vacuum pump, 9 - exhaust line, 10 - shadow instrument "IAB-458", 11 - pyrometer, 12 - AGA-780 infrared thermovision system, 13 - monochromator, 14 - grating spectrograph, 15 - Fabry-Perot scanning interferometer, 16 - data logging system ORION-3530, 17 - videocamera.

Inductor chamber contains 5-turns inductor and discharge channel. That is designed to provide atmospheric pressure around an inductor. In addition, water-cooled walls of inductor chamber is a shield from powerful electromagnetic field, which is generated by inductor.

**Induction heater** includes discharge channel, gas feed unit, top and bottom interfaces and nozzle unit (if it is necessary).

**Discharge channel** and gas feed unit are designed for making optimal gas flow, which guarantees the maintenance of stable steady state discharge in gas flow.

**Nozzle unit** is designed for forming and accelerating of plasma jet up to supersonic or high subsonic velocities. Conical supersonic nozzles do not used in the IPG-4 facility, since jet diameter (at available capacity of vacuum system) is too small for testing of typical samples of 15-30 mm diameters. However application of sonic nozzles with critical section diameters 15-40 mm allow to make such tests. Also it should be

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**Figure 7.** Conceptual sketch of the test chamber of the IPG-4 facility. 1 - low pressure test chamber wall, 2 - inductor chamber, 3 - nozzle unit, 4 - discharge channel, 5 - ignition device, 6, 7 - positioning devices, 8 - cooled shields.
emphasized, that when developing nozzle unit for the IPG-2 facility it was observed experimentally instability of discharge under operation with supersonic nozzle. To eliminate this effect gas injection system and discharge channel of the IPG-2 were renovated to obtain stable discharge. This experience was used in the design of the IPG-4 facility and instability was not observed in this on.

Criterion of discharge stability for operation in supersonic regime was found at simple and rough assumptions: (i) discharge is vortex stabilized (not by water-cooled walls of discharge channel) (ii) plasma volume is more lesser than volume of discharge channel including gas supply pipe up to inlet valve, and (iii) pressure drop between inlet section of nozzle and valve is to be more lesser than pressure drop on valve. This criterion, which was used to obtain stable discharge, is presented by formula:

\[
\frac{\partial G_{\text{out}}}{\partial P} - \frac{\partial G_{\text{in}}}{\partial P} > \frac{\tau_1}{\tau_2} \cdot \alpha
\]

where \(G_{\text{out}}\) - mass flow through sonic section of supersonic nozzle, \(G_{\text{in}}\) - mass flow through inlet valve, \(P\) - pressure in discharge channel, \(\tau_1\) - \(V/n\) (\(V\) - volume between inlet valve and critical section of nozzle, \(n\) - volume flow of gas through discharge channel in stable regime), \(\tau_2\) - time of discharge expansion from channel axis to its wall in motionless gas, \(\alpha\) - value depending on design of inductor, discharge channel and on way gas injection into discharge channel, etc. One can easily obtain from presented criterion that \(\tau_1/\tau_2 < V/G\) and to increase stability of discharge one has to decrease \(V\), to increase mass flow rate of gas and as well as to use inlet valve operating in subsonic regime, etc.

Top and bottom interfaces are intended for fastening of discharge channel and gas feed unit to test chamber and inductor chamber.

Positioning devices are designed to insert models and gauges into plasma jet, to withdraw these ones after measurements or tests, as well as to move them smoothly along and across a jet during measurements. Two-direction positioning device allows to move the holder with gauges along X, Y, directions. Express positioning device allows to move a model or a sample into specified position in the jet and to withdraw them. Introducing and withdrawing times of express positioning device are about 1 s. Both devices allow to move sample/probe along jet axis during experiment.

Water-cooled shields were designed to protect test chamber wall from hot gas and they are mounted along walls inside the shell

Gas supply system of IPG-4 facility allows to use different gases. This system includes control section and gas flow control panel. The panel is placed so that to guarantee convenient observation and control.

Evacuation and exhaustion system is designed for (i) the producing of specified pressure in the test chamber, (ii) the cooling of hot gases, which were heated in discharge, before evacuation, (iii) the exhaustion of cool evacuated gases into atmosphere. The conceptual sketch of this system includes the following units: gas cooler, water-cooled vacuum pipeline, vacuum pipeline, control and stop valves, exhausting fan.

Plasma-water heat exchanger is designed for the cooling of hot gas flowing out from test chamber. The cooler represents water-gas heat exchanger which is made of corrosion-resistant materials. It is equipped with gauges to measure and to control gas and water temperatures.

Water-cooled vacuum pipeline is placed between test chamber and gas cooler. Vacuum pipeline has anti-vibration inserts, which are placed near vacuum pumps to protect vacuum pipeline, gas cooler and test chamber from the adverse action of pumps vibrations. All above-mentioned units are made of corrosion-resistant material.

Plunger vacuum pump has got capacity of \(0.3 \text{ m}^3/\text{s}\) at \(P = 0.01 \text{ atm}\). There is an opportunity to connect vacuum systems of the IPG-4 and IPG-3 facilities and to use all three pumps with total capacity \(1.3 \text{ m}^3/\text{s}\)

Control and stop valves are made of corrosion-resistant materials. They have remotely operated drive.

The exhausting fan of corrosion-resistant design is placed external to room in exit part of exhaust line.

Cooling system is used for water cooling of different elements of other systems such as inductor chamber, nozzle unit, heat exchanger, vacuum pipelines, tube of RF-generator etc. Each cooled element has got individual cooling circuit that is equipped with rotameters and thermometers. Thermometers are mounted in inlet and outlet cooling water pipelines to measure water temperature and mass flow. Such simple, cheap and reliable system allows to make complete calorymetry of the facility. Electronic flowmeters and thermocouples are used to measure inlet and outlet parameters of cooling water for in precise scientific measurements (heat flux, enthalpy) together with computer data logging system.

Tube generator is used as power supply system. The automatically operated switch connects the tube generator to the commercial power supply line (380 V, 50 Hz, 3 phases). Commercial tube generator is based on the powerful generator triode of "Y-66A", which is of 100 kW power at 10 kV voltage across an anode. Combined air-water cooling is used for this tube. Tube generator includes electric circuits, which transform the voltage of commercial power supply line (50 Hz) into high frequency voltage (1.76 MHz). Among these are voltage controller, anode transformer, high voltage rectifier, anode circuit. Anode voltage controller allows
to vary rectified and stabilized anode voltage between 0 and 9.5 kV. High voltage transformer (380/8400 V) is of 100 kW power. The variation of tube generator power is realized by fine controlled linkage between anode circuit and load circuit. Load circuit includes the bank of capacitors and 5-turns water-cooled induction coil (so-called inductor), which is made of copper tube. Load circuit is tuned to the frequency 1.76 MHz under operation conditions.

Subsonic and supersonic flows created by the IPG-4 plasmatron are shown in Fig. 8.

![Figure 8. Subsonic(a) and supersonic(b) air plasma jets of the IPG-4 facility.](image)

Conceptual sketch of the IPG-3 facility is similar in general to that one of the IPG-4. One of the most significant distinctions between the two facilities is conceptual sketch of test chamber. To ensure atmospheric pressure around inductor small inductor chamber is used in the IPG-4 (test chamber diameter 800 mm), but in the IPG-3 test chamber of 1200 mm diameter is divided into two chambers of law and tests of ablative materials under radiative & convective heating. These works established methodology, technical approach and main specific features of tests in IPM.

Complex diagnostics of physical parameters of high-enthalpy dissociated gas flows in plasmatrons of the IPG series includes experimental and numerical techniques as follows:

- measurements of heat fluxes to model surface using stationary water-cooled calorimeters;
- total enthalpy measurements using enthalpymeter with gas sampling;
- measurements of total pressure profiles using water-cooled Pitot tubes;
- spectrometric temperature measurements in discharge and in high-temperature core of subsonic flows;
- pyrometric measurements of surface temperature of samples surfaces in the range $T_w = 300-2200 \text{ K}$ using optical pyrometers $(\lambda = 0.55-0.65\mu m)$ and infrared thermovision systems $(\lambda = 3.5-10.8 \mu m)$;
- numerical calculations of free subsonic flow and flow over models of viscous dissociated reacting gas using Navier-Stokes equations at $M << 1$ as well as numerical calculations of non-equilibrium boundary layer on a surface of the models under conditions of experiments in plasmatrons.

Data of subsonic high-enthalpy dissociated flow diagnostics are considered carefully in cited literature. A summary Table 2 shows parameters of subsonic high enthalpy air jets, which have been realized in the facilities of the IPG series which are in operation now. These subsonic jets most closely correspond to the requirements of hypersonic heat transfer simulation at blunt body tip-nose radius $R \sim 1 \text{ m}$.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>IPG-3</th>
<th>IPG-4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Generator anode power supply, kW</td>
<td>60-750</td>
<td>15-80</td>
</tr>
<tr>
<td>Total enthalpy, MJ/kg</td>
<td>10-40</td>
<td>10-40</td>
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<tr>
<td>Total pressure, atm</td>
<td>0.01-0.3</td>
<td>0.01-1.0</td>
</tr>
<tr>
<td>Gas temperature near exit section of discharge channel, K</td>
<td>7000-11000</td>
<td>4500-10500</td>
</tr>
<tr>
<td>Velocity, m/s</td>
<td>500-1100</td>
<td>20-950</td>
</tr>
<tr>
<td>Radius of test model, cm</td>
<td>1.5-17.5</td>
<td>0.5-4.0</td>
</tr>
<tr>
<td>Heat flux, W/cm²</td>
<td>10-1000</td>
<td>15-600</td>
</tr>
<tr>
<td>Reynolds number</td>
<td>50-150</td>
<td>50-200</td>
</tr>
<tr>
<td>Mach number</td>
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<td>0.02-0.5</td>
</tr>
<tr>
<td>Efficiency, %</td>
<td>55</td>
<td>40-64</td>
</tr>
</tbody>
</table>

4. TESTS OF ABLATIVE MATERIALS UNDER RADIATIVE & CONVECTIVE HEATING

The first experiments with real heat protection materials, which have been made 35 years ago, were tests of ablative materials under radiative & convective heating. These works established methodology, technical approach and main specific features of tests in IPM.

First of all, from the very beginning there were no attempts to obtain maximally possible Mach and Reynolds numbers, but it was realized simulation of thermochemical action of shock layer plasma on material near stagnation point using subsonic plasma flows at $M << 1$. Enthalpy, pressure and velocity gradient in stagnation point were chosen as close as possible to those in real flight. To obtain radiative heat flux close to that in flight the length of discharge and jet was chosen close to typical thickness of shock layer. Complete simulation theory for non-equilibrium convective heat transfer in stagnation point was developed significantly later in 80's years and later [26-28].
Experiments were carried out at atmospheric pressure and condition at the axis of equilibrium air plasma flow were as follows: $H = 40 \text{ MJ/kg} \ (T = 8500 \text{ K}), V = 30 \text{ m/s}$. Total heat flux to stagnation point of hemispherical water-cooled model of 30 mm diameter was 400 W/cm² with radiative heat flux ~30% of this value. Maximum surface temperature of tested samples achieved ~3000 K. Main directions of work were investigations of spectra of pure air plasma, spectra of boundary layer, temperature and species concentration profiles across boundary layer, determination of spectral absorption coefficients of pure air plasma and destruction products of ablative materials injected in boundary layer.

Of course, effective enthalpy determination together with other measurements, which are conventional for ablative materials, have been applied to many different candidate materials and materials used in real flights.

Sample of ablative material tested in high-enthalpy air flow of the IPG-1 facility is shown in Fig.9. Profiles of temperature and species concentrations across boundary layer on ablative material used in real flights are given in Figure 10 and Figure 11 respectively.

**Figure 9.** Ablative material in air plasma flow of the IPG-2 facility (note, that plasma flow at atmospheric pressure and temperature 8500 K is not seen near very bright boundary layer).

**Figure 10.** Temperature distributions across the boundary layer on ablative material. 1-3 – rotational temperatures determined by $N_2$ and $N_2^+$, 4 – vibrational temperature determined by $CN$ molecule.

**Figure 11.** Relative concentration of species across the boundary layer on ablative material.
5. TESTING OF REUSABLE THERMAL PROTECTION MATERIALS AND FULL-SCALE ELEMENTS OF THERMAL PROTECTION SYSTEMS

5.1. Aging tests of carbon-carbon materials with antioxidative coatings

Aging tests of carbon-carbon materials with antioxidative coatings were carried out using the IPG-2, the IPG-3 and the IPG-4 facilities. Main parameters of those were presented above. The IPG-2 facility was main test instrument for testing of small samples during Buran program to obtain temperature, pressure and time dependencies of mass loss rate. The IPG-3 facility was used to test large samples or full-scale structures. The IPG-4 facility is used now instead of disassembled IPG-2.

All experiments were carried out using stagnation point configuration at constant anode power of generator. Used models was cylindrical with flat ends and samples were about half model diameter. It guaranteed the uniformity of heat flux to heated surface of sample. The difference between maximum and minimum temperatures over front sample surface did not exceed 20°C even for large samples. The samples were of disk form of 2-10 mm thickness. The designed holders and models allowed samples to be removed from model between the cycles of testing for visual and microscopic inspection, photographing and weighing. Duration of each testing cycle varied from 10 to 30 minutes in different test programmes and corresponded to specified time of peak heating part of the reentry trajectory.

First aging tests of carbon-carbon material with different SiC based antioxidative coatings were made in 1980 and two samples withstandted 100 testing cycles of 15 minutes duration at P = 0.06 atm and initial surface temperature 1500°C in the IPG-2 facility. Time dependencies of sample mass and surface temperature for the best sample of the lot in question are presented in Fig.12.

One can see from Fig.12, that surface temperature of sample decreases with number of cycles. Special study showed that this effect is caused by decrease of surface surface catalicity due to silica layer formation on front surface of the sample as a result of plasma-flow interaction but not change of surface emissivity. The samples with other coatings of the same branch showed that thin cracks of coating results in increase of mass loss rate, but the other coatings demonstrated effect of self-healing even when hole of 0.5 mm diameter was made in the coating by powerful CO₂-laser.

Carbon-carbon material with glass-silicide antioxidative coating which was used for manufacturing of nose-cap and leading edges of Buran and of coarse it was subjected systematic study. The antioxidative glass-silicide coating, which is based on high-melting borosilicate glass doped with MoSi₂, was developed by NIIGrafit for deposition on siliconized C-C materials [29].

30-cycles aging tests were carried out using the IPG-2 and the IPG-3 facilities. In the IPG-2 facility tests were made at constant surface temperature 1480°C and pressure 100 hPa. In the IPG-3 facility tests were made at constant pressure 16 hPa anode power of HF-generator 325 kW. It corresponded to surface temperature 1500°C in the beginning of the first testing cycle. Time dependencies of mass loss rate during 30-cycles aging tests are shown in Fig.13 (see next page). One of the curves corresponds to test of samples of 30 mm diameter in the IPG-2 facility and the other one shows data obtained in the IPG-3 facility at average temperature Tₛₐₚ = 1477°C, which was found over all test cycles. Taking into account pressure dependence of mass loss rate and influence of scale factor two presented curves are in acceptable agreement.

The result of thermochemical action of dissociated air is seen in Fig.14 which shows the coating surface on front and back sides of the sample. Note, that back side of the sample had practically the same surface.
1A-10

**Figure 13.** Mass loss rate of siliconized carbon-carbon material with glass-silicide coating as a function vs the number of 10-minutes test cycles.

1. the IPG-2 facility, sample of 30 mm diameter $T_w = 1480^\circ$C, $P = 0.1$ atm,
2. the IPG-3 facility, sample of 84 mm diameter $T_w \approx 1480$, $P = 0.016$ atm

Temperature as front surface but it was exposed to stagnant non-dissociated air since fibrous material was used for back side thermal insulation. As a result back side surface is practically identical to initial surface state before test.

### 5.2. Testing of material of thermal protection tile

Careful study of properties of tile material described in [30] were carried out using the IPG-2 facility including 100-cycles test of one sample, studies of coating degradation in for different surface temperatures and in different gas environments. However the most important results was obtained very simply. Comparison of tile surface morphology after one testing cycle and after one real flight of Bor-4 vehicle showed identical changes (see Fig.15a,b). And plasmatron was the only test facility that demonstrated this effect!

**Figure 14.** Surface of antioxidative glass-silicide coating for Buran's nose cap after 30 10-minutes testing cycles at $T_w = 1450^\circ$C (SEM photo). 

a - front surface of the sample exposed to dissociated air, 
b - rear surface of the same sample after testing.

**Figure 15.** Tile coating surface after one-cycle test in plasmatron (a) and after real flight of «Bor-4» (b).

### 5.3 Tests of full-scale thermal protection tile

Testing of full-scale black ceramic tile was carried out in the IPG-3 facility. Experiments were performed,
using air, at pressure 0.026 atm, total flow enthalpy 17.3 MJ/kg, and surface temperature at the center of front surface of tile 1250°C.

The surface temperature distribution was derived from infrared thermovision system data. Tiles were mounted into the cylindrical sample holder of the same material so that the tile surface was perpendicular to the axis of symmetry of the subsonic jet. The model was configured to simulate all designated features of the tile heat protection. Up to 100 cycles of tile testing were conducted, with each cycle being 10 minutes long. The front surface of the tile, which was initially black, became grey after testing as a result of repeated exposure to the dissociated air flow. This "surface greying effect", which results in decreasing the total hemispherical emissivity \( e_h \), was studied later and its thermochemical nature was found. Surface degradation was caused by interaction of the tile coating material with atoms of oxygen. Tile before testing together with tiles after 1 and 100 testing cycles are shown in Fig. 16. Note, that tile after 100 cycles is grey but not black, however its total hemispherical emissivity at 1250°C decreased non-significantly.

**Figure 16.** Full-scale Buran's ceramic tiles with black glassy coating: a – before testing, b – after 1 testing cycle, c – after 100 testing cycles.

Tile surface study performed with a scanning electron microscope (SEM) showed that the near surface layer of the tile coating changes with the increasing of testing time. Open pores were generated in the near surface layer after the only cycle of testing. The development of the near surface porous layer, as the number of testing cycles increases, is illustrated by the SEM photographs in Fig.17. Test conditions were \( T_w = 1250°C \) and \( P = 0.026 \text{ atm} \).

The maximum and average thickness of the near surface porous layer depends on the number of testing cycles, as shown in Fig.18 (one can see all experimental points are in good agreement with square root dependence besides 100-cycle point of maximum thickness). In spite of these changes in near surface layer, the tile did not acquire any fissures or shape changes, even after 1000 minutes of exposure to the high temperature dissociated air. The results demonstrate the important requirement that the surface catalytic activity, with respect to heterogeneous recombination of nitrogen and oxygen atoms, remain constant after the 100-cycle exposure testing.

**Figure 17.** Section of black tile coating after 30 testing cycles at \( T_w = 1250°C \), \( \tau_{cycle} = 600 \text{ s} \).

**Figure 18.** Maximum and average pore layer thickness as a functions of testing cycle number \( (P = 100 \text{ mbar}, T_w = 1250°C, \tau_{cycle} = 600 \text{ s}) \).

Full-scale elements of thermal protection systems

The greatest model (\( \Theta 525 \text{ mm} \)) ever tested in the IPG-3 plasmatron is full-scale window of Buran's manhole cover with its fastening and nearest tiles. Window includes three glasses with heat reflecting coatings on rear sides. Photo of this model before test is shown in Fig.19 (see next page).

Because manhole cover is located on the side of vehicle, heat fluxes to this element are not high and model axis was arranged at angle \(-70°\) to flow axis. The choice of regime was made by heat flux using thin black heat protection tile which covered glass of window. Surface temperature was measured by infrared thermovision system and heat flux was determined by stationary surface temperature. In experiments
maximum temperature of front surface of quartz glass achieved 800°C.

The greatest full-scale carbon-carbon light-weight structures tested in the IPG-3 plasmatron have got dimension 220x220 mm. These elements were tested in cylindrical mask of 350 mm diameter and tests were made in stagnation point configuration.

5.4. Simulation of non-standard situations

The non-standard situations, which are possible during the exploitation of reusable thermal protection materials, are sufficiently different and, moreover, the most dangerous situations are different for various types of materials. The simulation of non-standard situations using plasma-jet facilities requires much more ingenuity than the routine tests, and individual approach is necessary for each situation. As a rule, the careful analysis of a wide range of fine physical and chemical effects is required for the correct design of experiment. Sometimes the experiment in plasmatron is the last step in the long sequence of investigations, which include complicated computer calculations and hypersonic wind tunnel experiments. It should be emphasized, that neither numerical modeling nor wind tunnel experiment are not able to give the total solution of the problem.

Non-standard situations for heat protection materials of "Buran" have been simulated in the IPG-3 facility, because it gives the opportunity to test full-scale tiles. However, advance thermal protection elements can have considerably larger dimensions, than existing tiles. In this case it is impossible to test real-scale thermal protection elements and the IPG-4 facility can be acceptable choice when simulation can be realized using models lesser than 100 mm in diameter.

The non-standard situations, that have been studied in the IPG-3 plasmatron, can be subdivided by the following manner:

- reentry along a trajectory with over-design heating, up to the emergency reentry, when a reusable vehicle, after saving the payload, loses it's serviceability (totally or partly);
- damages of thermal protection element coating, such as a loss of a part of coating, cracking, coming off a substrate;
- total or partial loss of a thermal protection element;
- water saturation of porous or fibrous thermal protection materials;
- overequilibrium heating of noncatalytic coating, which has been contaminated during exploitation.

Each item of this list deserves careful consideration in individual article but hereinafter we consider only tests of water saturated tiles as typical representative of kind of tests in question.

Atmospheric water influence on porous or fibrous materials of heat shield is one of the most complicated problems during vehicle exploitation. Saturation of such materials with water will be considered for the example of ceramic tiles. The technique of hydrophobization was developed and tested in exploitation for ceramic tiles of "Space Shuttle". However, it turns out, that sometimes the tiles have contained water. In this case it is necessary to determine the amount and the location of water in tiles (it is the subject of a separate investigation) and to predict correctly the behavior of tile, basing on laboratory experiment data. Since saturation of tiles with water can lead not only to coating coming off and to the loss of coating, but also to the appearance of inner damages, the residual strength of material after tests is important criterion.

Obviously, water contained in tiles freezes at putting a vehicle into orbit and becomes ice. The difference between thermal expansion coefficients of tile material and ice can lead to the rupture of material fibers during further cooling and heating, which cause the decrease of material strength.

The essential aspect should be emphasized: ice can be produced in tiles not only during putting into orbit, but also at a launch position. In this case mechanical properties of ice, and, possibly, predicted behavior of
tiles considerably differs from the those in previous case.

The use of literature data on mechanical properties of ice can lead to the great mistakes in prediction, since even under the nature conditions there are more than 20 kinds of ice with different characteristics. Structure and properties of ice essentially depend on a large number of parameters, such as temperature gradient, rate of water cooling, gas and salt content of water, etc. It is obvious that the very specific kind of ice - friable ice, which consists of long needle-shaped crystals and is formed by vacuum pumping of water vapors - this ice differs essentially from routine kinds of ice by its structure and properties. Therefore, during tests of water-saturated tiles careful simulation of pressure decrease rate is important as well as the rate and conditions of cooling, corresponding to possible situations at launch position.

One more important aspect, which can be easily overlooked at cursory analysis of the problem, is chemical composition of water. The fact is that alkaline metals, contained in water as salts, and primarily K and Na, are deposited on fibers surface at the vaporization of water during reentry. These metals have extremely high diffusion coefficients in silicate glasses, which leads to their quick propagation over the whole volume of fibers at high temperature. The increase of K and Na content in silica leads to the decrease of its softening temperature and to the material shrinkage, especially in case of tiles, repeatedly saturated with water. Therefore, water with the chemical composition, corresponding to the composition of rainfall at the location of launch position, should be used in the experiments with tiles, multiply saturated with water. The mentioned examples demonstrate clearly the main problems associated with testing of water-saturated tiles. Besides, there are some technical problems, which have been resolved. For example, practically all vacuum pumps "don't like" water vapors, etc.

Tiles saturated with 30, 100, 300 and 500 g of water were tested under different conditions of freezing but in the same regime: $T_{w}=1250^\circ$C (for dry tile), $P=0.026$ atm, $t_{cycle}=10$ min. It was found that only in one experiment at 500 g of water was observed additional coating damage due to ice vaporization. In all other test coating was not damaged and residual tension strength of tiles was found the same as before tests. Temperature field was measured by infrared thermovision system and time dependencies of maximum, average, and minimum surface temperatures for tile containing 30 g of water are presented in Fig. 20. Note, that maximum surface temperature at water vapor blowing from tile surface is higher than without blowing.

![Figure 20 Time dependencies of maximum, average, and minimum surface temperatures for tile containing 30 g of water.](image)

6. STUDY OF THERMOCHEMICAL PLASMA-SURFACE INTERACTION

Optical spectral analysis is one of the most promising methods for the study of plasma action on reusable/non-ablative thermal protection materials (TPM). Although optical spectra of destruction products of reusable thermal protection materials are “poor” (in contrast to “rich” spectra of destruction products of ablative materials), they give reasonably good possibilities to study physiochemistry of plasma-material interaction. Application of spectral analysis makes it possible to observe in situ degradation of antioxidative coatings during aging tests of any duration and analysis of boundary layer spectrum permits to find, what kinds of species are at a loss from substrate/coating during test, to see time dependencies of those losses etc.

Many essential features of thermochemical action on thermal protection material by dissociated air flow may be found using optical spectra analysis in combination with making tests in different plasma environments such as air, nitrogen, oxygen and argon. As a rule mass loss of carbon-carbon materials with antioxidation coating is generally resulted from the oxidation but atomic oxygen is essentially more active than molecular. Comparative study of optical spectra of boundary layer allows to understand what components are arisen in boundary layer due to thermal vaporization and what components are arisen in boundary layer due to thermochemical action of the initial flow. Obviously, above mentioned approach is to be combined with data on temperature, pressure and time dependencies of mass loss rate, as well as photos of surface obtained using SEM etc.

Consider two examples of spectral analysis application for study of plasma-material interaction. Emission optical spectra were obtained during tests of C-C
material with glass-silicide coating in dissociated air and nitrogen flows. All experiments were carried out at constant pressure $P = 100$ hPa using the IPG-4 facility.

More than 20 rather intensive spectral lines of molybdenum were found when sample was exposed to dissociated air flow. Also sensitive lines of BI, SiI, MnI, KI, NaI were observed in boundary layer overflowed by dissociated air flow. Note, that simple qualitative chemical analysis gives two interesting results. Specially made plasma jet tests showed that there are no even weak sensitive lines of molybdenum while sample is exposed to dissociated nitrogen flow but lines NaI, KI, BI, SiI were found in these spectra. These result suggest that molibdenum atoms arising is caused by the thermochemical action of oxygen but the other components arise in boundary layer due to thermal evaporation. The band of wavelength 377-394 nm, containing sensitive lines of molybdenum MoI379.83 nm, MoI386.41 nm and MoI390.30 nm, is shown in Fig.21 for dissociated air flow (a) and dissociated nitrogen flow (b).

![Figure 21. Spectra of boundary layer over surface of glass-silicide coating ($\lambda = 337-394$ nm). a - dissociated air flow, b - dissociated nitrogen flow.](image)

So, one can make conclusion that it is atomic oxygen that causes such damages to coating. The most effective way of molybdenum loss is associated with the oxidation of $\text{MoSi}_2$ followed by evaporation of exceptionally volatile molybdenum oxides and it is one of the leading mass loss processes.

It was found later, that intensities of molybdenum lines in boundary layer become unstable before the beginning of catastrophic destruction of the C-C material with glass-silicide coating, but at the same time intensities of other lines of main components of the coating such as silicon and boron don't demonstrate such behaviour as it is seen from Fig.22.

![Figure 22. Time dependencies of sensitive lines intensities of different atoms which appeared in boundary layer due to coating degradation.](image)

As it is seen from Figure 22, intensities of lines of silicon and boron before the beginning catastrophic destruction are enough stable and those begin to increase only with the increase of surface temperature. At the same time intensity of molybdenum line begin to pulse $\sim 10$ s before the beginning of catastrophic destruction. Peak intensities can be 2-3 times higher than its quasi-stationary level. There is no such peaks at usual operating temperatures, they appear only before destruction. This result shows that destruction is caused by intensive oxidation of $\text{MoSi}_2$ particles, containing in the coating, by atomic oxygen from oncoming flow.

At the same time observable peaks shows, that before boiling heavy oxidation is blocked time by time with formation of protective film of oxides, and finally it becomes so strong, that the formation of film does not realized. So, study of specific features in behavior of boundary layer over tested sample before catastrophic destruction allowed to obtain new information on mechanism of destruction even for well-known material & coating. Of course, this approach is mostly fruitful for new TPM/coatings.

Thus, it was found, that before catastrophic destruction one can find few peaks of molybdenum lines intensity. There is no any peaks at usual operating temperatures. 100% repeatability in appearance of those peaks allows to consider them as «spectral precursors» of catastrophic destruction.
Study of spectral precursors of catastrophic destruction can be easily made practically in each laboratory, working on TPM testing in plasma jets. First of all, it can give important information about physico-chemical processes, that initiate catastrophic destruction of tested reusable TPM. Of course, that information can be used to improve tested material.

7. STUDY OF NON-EQUILIBRIUM HEAT TRANSFER

One of most important fields of activity in Plasma Lab of IPM is a study of non-equilibrium heat transfer. This works were initiated by programs of development of space planes «Space Shuttle» and «Buran», since peak reentry heating of these vehicles depend significantly on surface catalycity of thermal protection materials. Really, heat flux to full-catalytic surface near stagnation point is greater by several fold than heat flux to non-catalytic surface. It required to renovate old and to develop new methods of catalycity determination.

Experimental & theoretical method of surface catalycity determination developed in IPM is based on comparison of measured heat fluxes to tested material with results of numerical heat flux calculation where effective probability of heterogeneous recombination is varying parameter. Main specific features of used approach were as follows. Fist, catalytic tests were carried out under simulated reentry conditions which are as close as possible to flight conditions. Second, codes for numerical calculations were specially developed for treatment of experimental results obtained in subsonic jets of plasmatrons.

Method of catalycity determination is described in individual article of Dr.Kolesnikov in the present Lecture Series.

8. TESTS IN PLANETARY ATMOSPHERES

First reusable light-weight thermal protection materials for spaceplanes were developed in the frames of Shuttle' and Buran' programmes. Ultralight-weight fibrous heat-insulative and carbon-carbon materials became available and now they are not too expensive. Successful exploitation showed their high reliability. Joint influence of all these circumstances allowed to formulate a question about possible application of new materials in advanced planetary probes instead of conventional ablative materials. However, in the absence of blowing one has to consider possible influence of heterogeneous recombination on heat flux. Taking into account, that Martian atmosphere is rarefied, such influence is very possible. Actually, calculations presented in [ ] showed, that heat flux distributions over front surface of entry vehicle depends significantly on surface catalycity in relation to reactions $\text{CO}+\text{O} \rightarrow \text{CO}_2$ and $\text{O}+\text{O} \rightarrow \text{O}_2$.

Since 1996 to 1999 Plasma Lab of IPM together with TsAGI, TsNIIMASH and IM MSU have participated in Project 036 of International Scientific and Technology Center intended for development of heat protection system for interplanetary flight and IPM team was responsible for determination of surface catalycity under conditions of entry into Martian atmosphere.

That is why first of all it is necessary to obtain of stable, steady-state jets of $\text{CO}_2$-plasma and to expand operating envelope of the facility as far as possible. That first step is one of the key points in work on catalycity determination, because without highly developed, stable, steady-state regimes of facility it is very difficult (or impossible) to obtain reliable experimental data on heat transfer.

When developing plasmatron operation with new working gas, the main aim in first stage is to clarify basic possibilities of facility, i.e. to obtain operating envelope in coordinates «pressure - power». Operating envelope in these coordinates is one of the main characteristics of plasma jet facility (arc-jets and plasmatrons), because that one is determined by basic properties of used generator and pumping system, but together with test chamber they are just these parts of facility which are the most conservative and difficult for renovation. For example, operating envelopes in coordinates «pressure - heat flux to catalytic/non-catalytic wall» can be easily changed by changing of model's form and dimension, by change of model's position in a jet and, finally, by using of new design decision of discharge device as a last measure.

Practically, the most difficult task is to obtain at least one stable, steady-state regime. If that one is available, success of step-by-step procedure of operating envelope expansion is generally a matter of time. Pressure 100 hPa and moderate anode power ~40 kW were selected as start point for work on determination of operation envelope in $\text{CO}_2$, because existing geometry of discharge channel was developed to be optimal for operation in air at 100 hPa, and moderate power allows to have enough power to support discharge existence on one hand and to avoid quick overheating of discharge channel on other.

Mass flow rate of carbon dioxide $G=2.8\;g/s$ was used in first experiments, because excellent results were obtained with that value in air. Later, $G=1.8\;g/s$ was found as optimal value. Criteria of mass flow rate optimization were the following: (1) good quality of plasma jet, (2) absence of discharge channel overheating and (3) ranges of parameters ($P$, $N_{\text{it}}$), where foregoing conditions are fulfilled, are to be as wide as possible. To clarify first point, it should be
noted, that visible length of jet was used to estimate plasma jet quality in first experiments, because it is known that quick destruction of jet does not allow to have high heat flux to a model. As to third criterion, it is very important to have a feasibility to obtain pressure and power dependencies of heat flux, dynamic pressure etc. instead of individual points, since it allows to avoid rough mistakes and to ensure clear, understandable presentation of results. Mass flow optimization allowed to realize stable discharge without limitation in operation time in wide range of pressure and power.

Operating envelope in coordinates «pressure - anode power» is shown in Fig.23 for subsonic regimes of the IPG-4 facility operation in CO₂ atmosphere.

![Figure 23. Operating envelope in coordinates «pressure - anode power» for subsonic regimes of the IPG-4 facility operation in CO₂ atmosphere.](image)

Presented data have been obtained at mass flow of carbon dioxide 1.8 g/s. This value is optimum for the IPG-4 facility operation with CO₂ flow at P < 200 hPa. As it is seen from Fig.1, total range of power regulation provided by HF-generator can be realized at high pressures. At low pressures and low currents (i.e. low power) simultaneous influence of several factors appears and the border of discharge existence area results a curve, however in pressure range 10-100 hPa, i.e. in the range of significant curvature of the border, heat conductivity is kept as basic physical mechanism of discharge propagation. The main result that can be found from Fig.1 is that operating envelope (in coordinates «anode power - pressure») is limited for CO₂-environment mainly by capacity of exhaust system and regulation range of HF-generator (11-1000 hPa, 12-76 kW). So pressure range covers two orders of magnitude and ratio between maximum and minimum available levels of power is about four.

To determine catalycity it is necessary to have available regimes where catalycity influence on heat flux is significant, therefore to find such regimes is necessary to make heat flux measurements with catalytic and non-catalytic calorimeters. However, there was no experimental data on surface catalycity in dissociated CO₂, so it was necessary to find high-catalytic and low-catalytic reference materials. In the first stage. In the first stage of work copper was found as material with the highest catalycity [ ] and flow enthalpy and velocity were numerically calculated by measured heat flux, dynamic and static pressure.

Measured and calculated parameters for three regimes of the IPG-4 facility are given in Table 3.

### Table 3. Parameters of regimes of the IPG-4 plasmatron operating with CO₂ as working gas

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Two reusable TPM were studied with both materials were used in real reentry to Earth atmosphere. First one is Buran’s ceramic thermal protection tile with glassy coating based on SiO₂-B₂O₃-SiB₄ system [30] and Buran’s siliconized carbon-carbon material with glass-silicide antioxidative coating for carbon-carbon nose-cap and leading edges. Obtained effective probabilities are shown in Figure 24.

![Figure 24. Temperature dependencies of effective recombination probabilities for tested materials.](image)
9. ALTERNATIVE WAYS

Not only problems concerned reentry problem were investigated in Plasma Lab of IPM. Flexibility of using and good control of regimes allowed to use plasmatrons for heat tests of industrial ceramic thermal protection materials and full-scale elements of industrial heat exchangers which must withstand heat shocks and extremal heat loading.

The other challenging direction of work was deposition of diamond and diamond-like films using IPG-4 plasmatron. Films were deposited even in air plasma flow and their catalycity was determined [31], but unfortunately these efforts did not find financial support.

10. CONCLUSION: LESSONS LEARNED FROM 35-YEARS HISTORY OF IPM PLASMA LAB

1. Experimentalists working with large facilities intended for test support of challenging space projects must be good forecasters, because facility is to be ready in general before decision on project realization.
2. To obtain good scientific results in modern high-temperature gas-dynamics it is necessary to unite experimentalists and theorists in one team under common supervision.
3. To present reliable test results, test laboratory is to be independent on material developers and vehicle designers.

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