GP12.00015

Preliminary Design and Results from the Riverside Research Plasma-Materials Interaction Experiment

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Abstract

Hypersonic vehicles are often exposed to extreme environments in which heat fluxes to the vehicle's surface can pose several challenges for the integrity of the shielding material. At hypersonic speeds, the temperature surrounding the vessel can exceed 3000 °C and the local environment becomes partially ionized. Under these conditions, oxidation effects become a significant concern. Despite the importance of understanding the role oxygen plays in materials testing, facilities capable of creating controlled oxygen plasmas with sufficient plasma and material diagnostics that can achieve sample temperatures of interest (i.e., >1800 ℃) are scarce. Recently, our team began to develop a facility capable of heating small material samples to high temperatures in a controlled cold gas environment. Compact samples (i.e., 75 mm×75 mm×5 mm) are heated to >1800 °C using a simple heating element. The heated sample is exposed to gas mixtures of known concentrations and operating pressures to study oxidation effects in a controlled environment. The current suite of diagnostics available to our laboratory can measure electromagnetic properties of the heated samples in the X band (8 – 12 GHz) and mid infrared (IR) range (66-230 THz) as well as monitor global environment characteristics. Here we present an update on the progress of this facility and preliminary results of materials characterization after a controlled gas exposure to a heated sample. Planned plasma source and diagnostic upgrades are also shown.

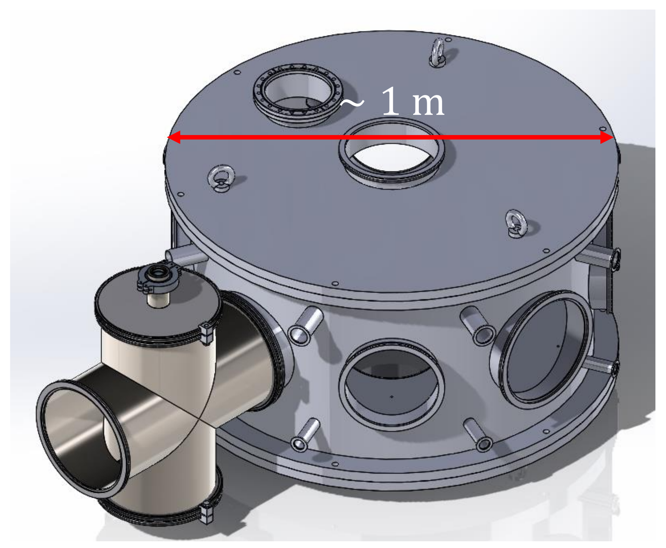
About Riverside Research

Riverside Research is a national security nonprofit dedicated to translating scientific research from the laboratory into real-world applications for the benefit of our government and our nation. We perform multidisciplinary research and development across various fields of physics and engineering to meet the needs of our customers. We offer trusted, independent solutions.

Motivation

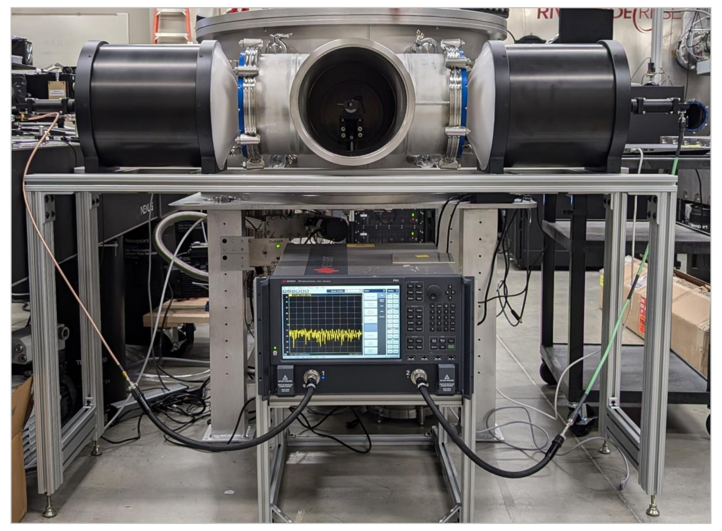
Hypersonic research is multifaceted. An ideal material deployed on a hypersonic vehicle must withstand extreme temperatures without losing structural and protective integrity, have favorable radiation windows (typically in the radio frequency range), and maintain these properties in harsh reactive environments. This work aims to develop a facility capable of testing novel samples under extreme conditions. **The Plasma-Materials Experiment at Riverside Research will heat samples to temperatures ≥ 1800 ℃ while exposing these samples to gases and reactive plasmas of known compositions.** The suite of diagnostics available for this experiment, and those that are planned to be added, will monitor sample temperature (IR camera), measure reactive species compositions during plasma-material interactions (OES/TALIF), monitor global chamber gas compositions (RGA), and measure dielectric properties of the sample-under-test in mid-infrared and X-band frequencies (VNA). **A full description of diagnostic capabilities can be found on poster GP12.00014.**

Existing Riverside Research Facilities are Well-suited for Materials Investigations

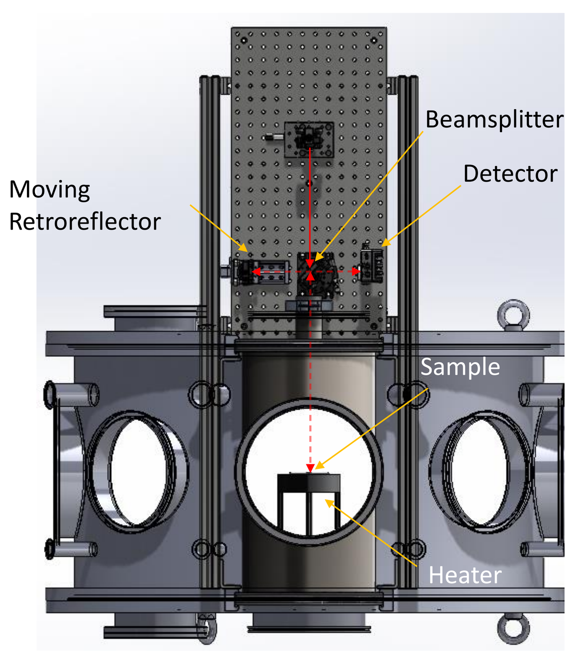


A one-meter diameter chamber with an ISO-250 four-way cross fastened to one side (left) provides several points of diagnostic access. Base pressures $\leq 1 \times 10^{-6}$ are achieved with sensitive control of gas mixtures using low flow MFC. Two separate configurations facilitate dielectric measurements in the X-band ($8-12$ GHz) or mid infrared ranges ($66-230$ THz).

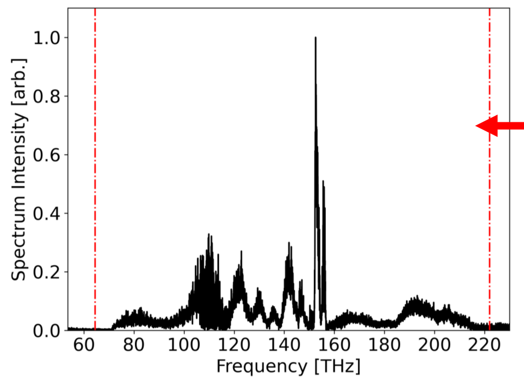
For measurements in the X-band range, two 12 in. diameter antennae are oriented antiparallel to each other. Each antenna has a focal length of 12 in. At the focal plane of the antenna a spot size (3 dBm level) of approximately one inch is achieved, allowing for relatively small samples to investigated. The antennae are swept from 8 GHz to 12 GHz using a two-port Keysight N5225B VNA, capable of driving frequencies between 10 MHz and 50 GHz. Reflected (S_{11}, S_{22}) and transmitted (S_{12}, S_{21}) signals are collected and recorded by the VNA to derive dielectric properties.



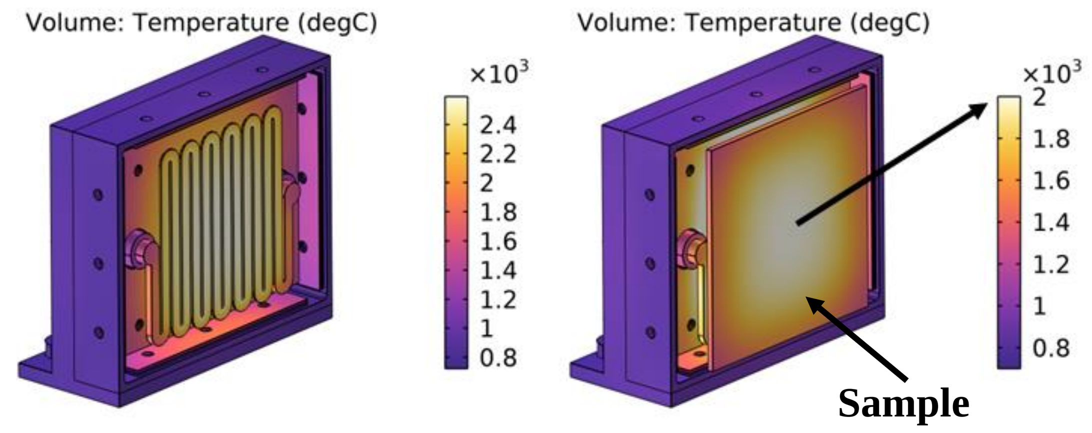
Experimental configuration for dielectric measurements in the X-band frequency range.



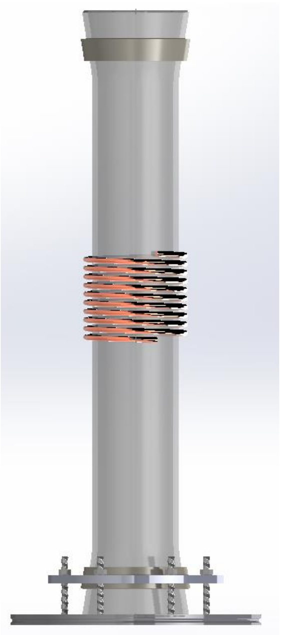
For measurements in the THz range, an interferometry configuration is used [*Chao, et al.*, 2012]. Radiation between ~ 66 and ~ 230 THz is delivered to the interferometry beam path by a Thorlabs SC4500 Mid-IR Supercontinuum Laser, corresponding to wavelengths between $1.3\ \mu m$ and $4.5\ \mu m$.



Planned Facility Upgrades Bridge Dielectric and PMI Experiments

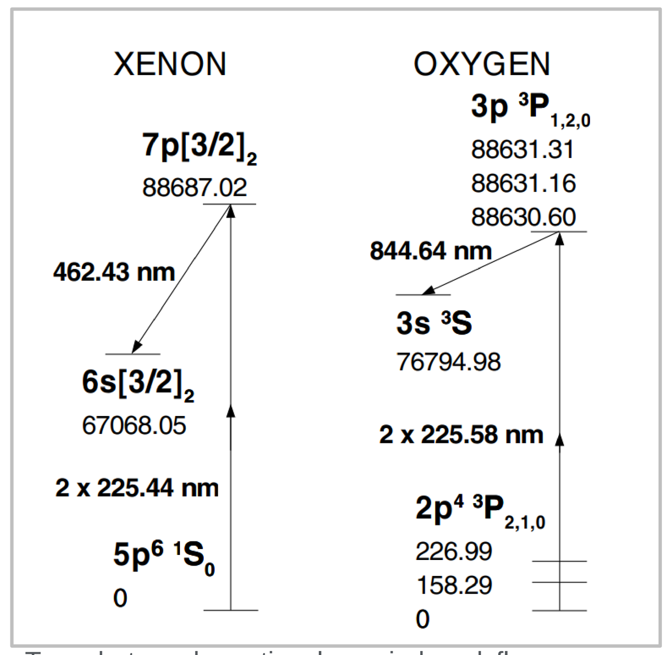


A custom vacuum compatible heater to heat small (~ 75 mm $\times$ 75 mm $\times$ 5 mm) samples to ~2000 ℃ without the need for large and expensive equipment has been designed at Riverside Research. Auxiliary heating enables the sample to be placed in an environment where the gas composition is precisely controlled. To further expand the capabilities of our facility, an inductive plasma source is being developed. A key feature of this design is that the plasma generation mechanism does not encounter the plasma, allowing for reactive species (i.e., oxygen, nitrogen, etc.) to be used without damaging the plasma source.



A two-photon absorption laser induced fluorescence (TALIF) system will be implemented to monitor reactive species concentrations (e.g., atomic oxygen) during plasma-material interactions. TALIF (generically LIF) is a laser spectroscopic technique that directly measures the distribution function of an atom, ion, or molecule.

- Two photons are absorbed simultaneously to excite an electron from a lower lying state to an excited state.
  - Fluorescence intensity is collected as a function of laser wavelength to construct the velocity distribution function.
- Select species are absolutely calibrated to derive absolute densities of interrogated species (right).

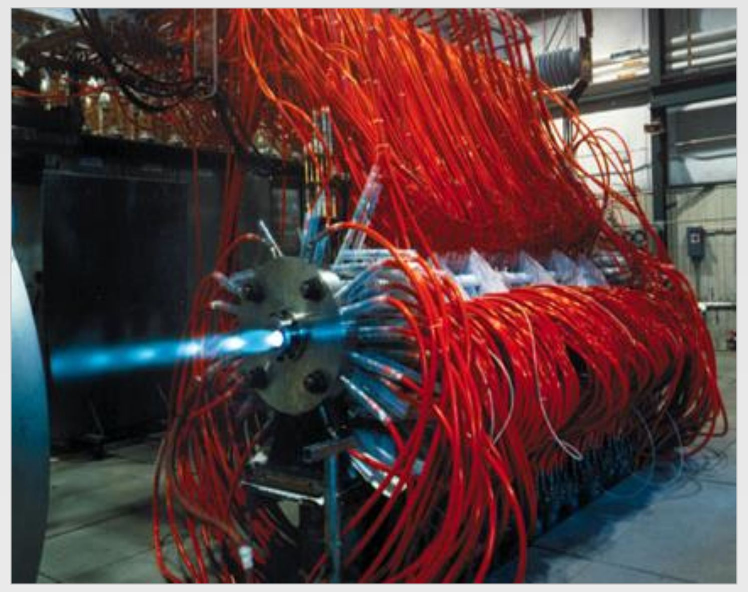


Two-photon absorption laser induced fluorescence scheme for oxygen and xenon (calibration gas). Image reproduced from [*Niemi et al.*, 2001].

A Lioptec Liopstar pulsed dye laser is slated for this diagnostic

- $\Delta t_{\text{Pulse}} = \sim 6\ ns$
- $\lambda = 193$ nm $- 900$ nm
- 10 Hz repetition rate

How is it Done Today?



U.S. Air Force - This image was released by the United States Department of Defense with the ID 950101-D-1111A-001

Current methods for testing samples under extreme plasma conditions are commonly executed at large arc jet laboratories (e.g., The Arc Jet Complex at NASA Ames). While these facilities achieve extreme environments, the gas composition is not easily controlled or changed, there are relatively few of these complexes in the country and they can be excessively large and are expensive to operate. This makes testing samples under extreme conditions a long and costly process.

Next Steps

- Construct and implement custom heater for high temperature operation.
- Reconfigure chamber for inductive plasma source and optimize operation for reactive plasmas.
- Develop and implement TALIF diagnostic for species concentrations monitoring.
- Test samples under room temperature/vacuum/high heat/plasma environments.

Key Takeaways

- Current facilities in the Plasma Laboratory at Riverside Research are capable of measuring material properties in X-band and mid IR frequencies.
  - Samples can be investigated at atmospheric pressures or in vacuum environments.
- Planned upgrades will enable testing materials under extreme conditions.
  - Custom vacuum heater will heat samples to temperatures ≥ 1800 ℃.
  - Inductive plasma source will enable sample exposure to reactive plasmas.
- **Riverside Research facilities are open to contracts with outside partners. Please contact Dr. Stephen Parsons at sparsons@riversideresearch.org for additional information or work opportunities.**

Acknowledgments

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References

[1] Chao, L, Sharma, A., Afsar, M. N., 2012 IEEE International Instrumentation and Measurement Technology Conference Proceedings, 2012.
[2] Niemi, K., Schulz-von der Gathen, V., Döbele, H.F., Journal of Physics D: Applied Physics, 34 (15), 2001.