



# Characterization of Material Response during Arc-jet Testing with Optical Methods – Status and Perspectives

Michael Winter, UARC – UCSC, NASA Ames

5<sup>th</sup> Ablation Workshop  
Lexington, KY, Feb. 28 to March 1, 2012

## Acknowledgments:

- NASA Contract NAS2-03/44 to UARC, UC Santa Cruz
- NASA-SCAP (Strategic Capabilities Assets Program)

Thanks to TS Division at NASA Ames.

## Outline:

- Motivation
- Examples for existing emission spectroscopy data
- Most recent results taken in the Ames arc-jets
- Proposed experimental approach for future campaigns
- Conclusion and outlook



## Motivation



- Arc-jet testing is one important application for validation of material response codes
- Current monitoring of ablative heat shield behavior through
  - measurement of temperature distributions (surface and in-depth)
  - total recession and char depth measurements (integral post-test)
  - indirect or/and time integrated characterization of pyrolysis processes
- Direct quantification of pyrolysis chemistry is not provided though more useful for validation of material response codes
- Potential for providing information on shape and composition of the blowing layer with optical methods through
  - optical emission spectroscopy (OES)
  - filtered imaging
  - laser induced fluorescence (LIF/PLIF)
  - absorption spectroscopy (AS), laser absorption spectroscopy (LAS)

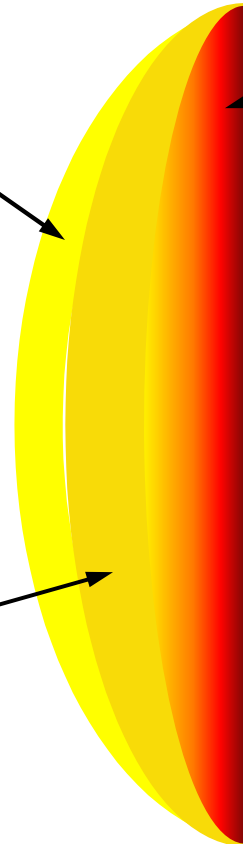
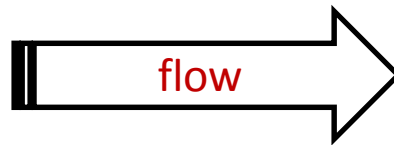


# Post-shock/Boundary Layer system



## Shock

High temperatures  
Non-equilibrium radiation



## Boundary layer

Moderate to low temperatures  
May be in non-equilibrium  
Low radiation levels

## Post shock

High temperatures  
May equilibrate  
Dominates radiation for large vehicles  
Ablation products may diffuse upstream

## Blowing layer

Temperatures close to T surface  
In equilibrium ???  
Low radiation levels



## Characteristics of different methods



### OES:

- Line of sight measurement, spatial resolution only through assumption of rotational symmetry
- Information on excited states, only in equilibrium reliably related to total densities
- Set-up relatively uncomplicated, usually no impact on material testing
- Experimental set-ups in principle available, improvements desirable

### Filtered imaging:

- Same restrictions as OES (LOS, excited states monitored)
- Provides 2-d measurements  
→ boundary layer shape
- Requires detailed knowledge of the spectral distribution
- Experimental set-ups partly available

### LIF/PLIF

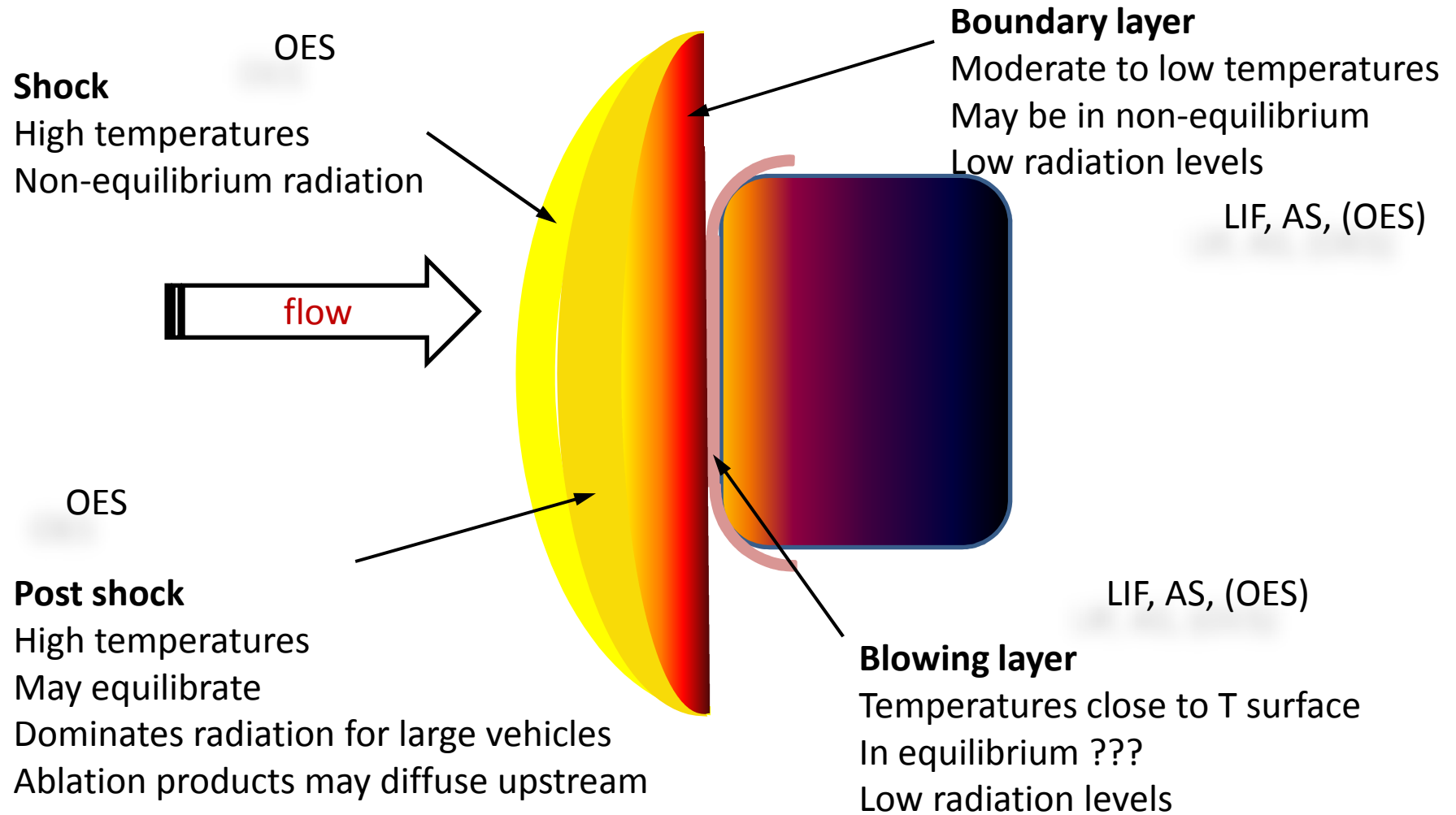
- Spatially resolved measurement
- Only one species at a time
- Set-up fairly complicated
- Usually needs more time than provided during conventional testing  
→ dedicated tests required
- Information on ground state
- Calibration process complicated
- Experimental set-ups partly available, calibration methods need to be developed.

### Absorption spectroscopy:

- Line of sight measurement
- Gives information on the ground state
- Background radiation may (plasma or surface) may have significant impact on accuracy
- Experimental set-ups to be designed



# Post-shock/Boundary Layer system





## Interpretation of OES data



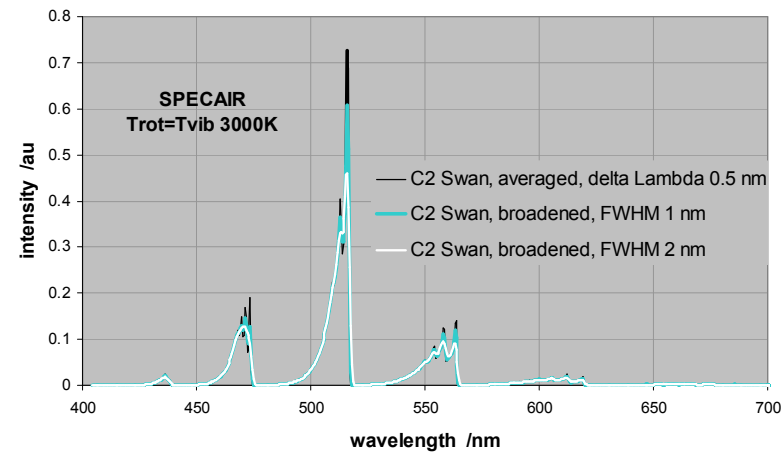
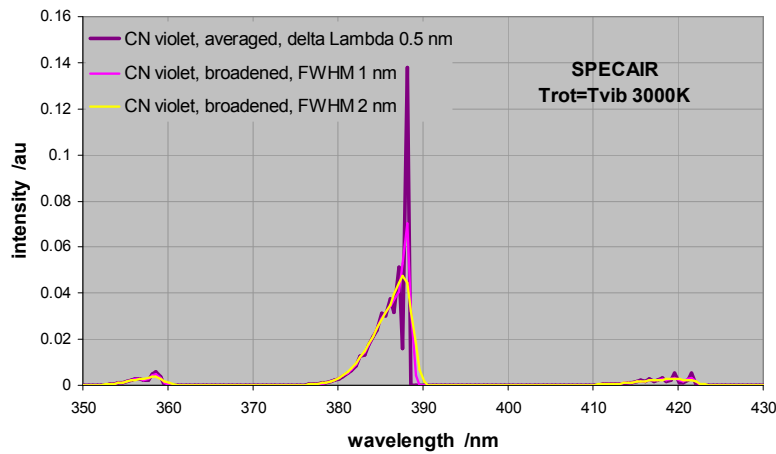
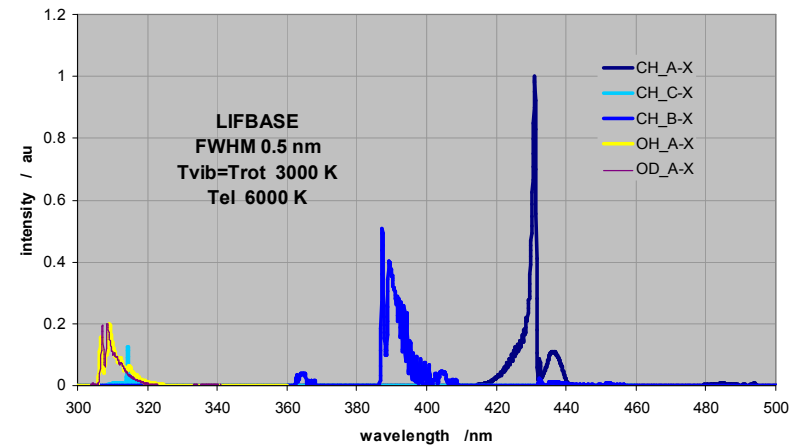
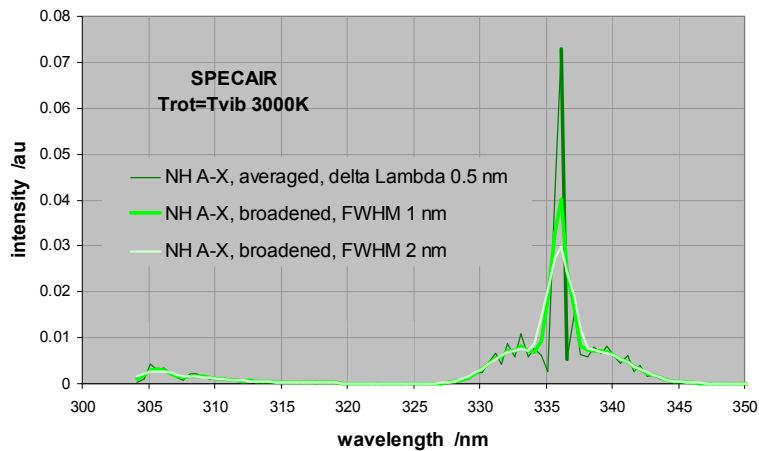
- Theoretical simulation of the spectra must be possible/known
- Diatomic molecules well known
- Larger molecules are far more complicated
  
- Electronic transitions usually in the UV/VIS/NIR range
  - easy detectable
  - electronically excited states only in equilibrium coupled to the ground state
    - less probable if transition between excited states
  - transitions to the ground state (X) preferred
  
- Vibrational bands usually in the IR
  - higher complexity (and price) of the detection system
  - easily disturbed by thermal radiation



# Simulation Capabilities



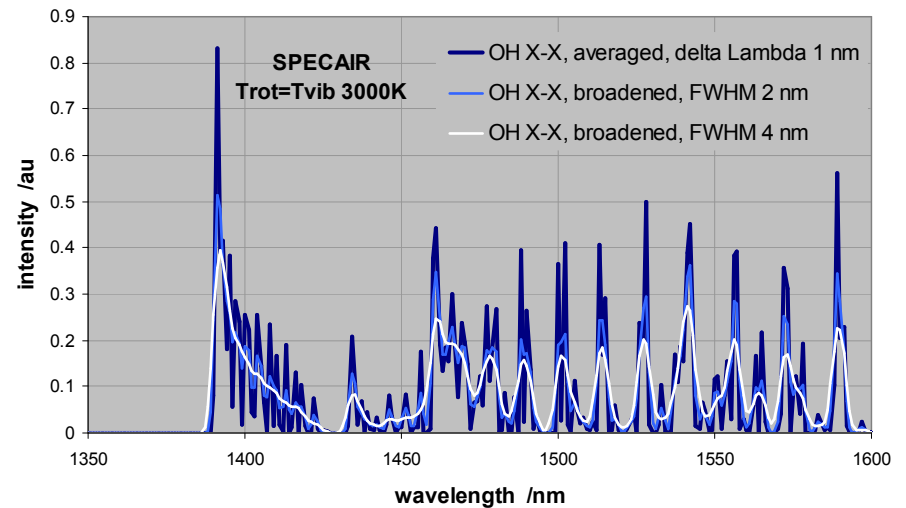
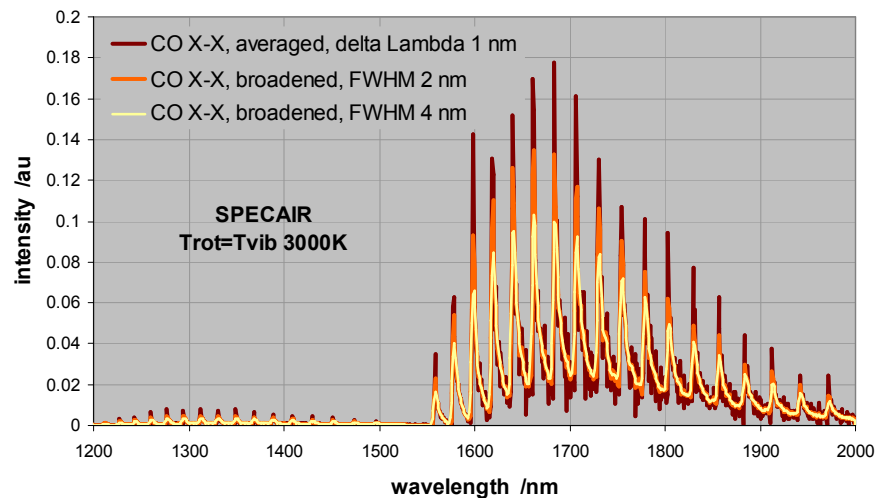
- Molecules containing H must be related to pyrolysis: NH, OH, CH (NH from interaction with plasma, OH maybe, CH not)
- Molecules containing C may be related to pyrolysis or ablation.





# Simulation Capabilities

- CO and OH offer infrared systems of vibrational transitions within the ground state
- Well suitable for determination of ground state densities if detectable with sufficient accuracy
- Radiation is emitted already at low temperatures in comparison to electronic transitions.







## Existing OES data (Langley/Ames)



- Measurements with fibers embedded in PICA and carbon phenolic in the AHF (2010)
  - 300nm to 900nm, no intensity calibration available
  - No spatial resolution from surface through shock

Schott, T., Herring, G.C., Munk, M.M., Grinstead, J. H., Prabhu, D.K., “Fiber-Based Measurement of Bow-Shock Spectra for Reentry Flight Testing”, 48<sup>th</sup> AIAA Aerospace Sciences Meeting, Orlando, FL, 2010.

- Measurements in front of PICA and SiC in the HYMETS arc-jet (NASA Langley, 2012)
  - intensity calibrated, 340nm to 950nm, UV experiments in preparation
  - Main ablation related radiation from CN, Ca lines observed
  - Spatial resolution through shift of the optical axis relative to the recessing material surface

Danehy, P. M., Hires, D. V., Johansen, C. T., Bathel, B. F., Jones, S. B., Gragg, J. G., Splinter, S. C., “Quantitative Spectral Radiance Measurements in the HYMETS Arc Jet”, 49<sup>th</sup> AIAA Aerospace Sciences Meeting, Nashville, TN, 2012.

- Measurements in front of PICA in the IHF (2004)
  - intensity calibrated, 400nm to 950nm, focus on micro spallation through laser attenuation
  - spatial resolution through shift of the optical axis relative to the recessing material surface
  - main ablation related radiation from CN, no Ca, N, or K detected

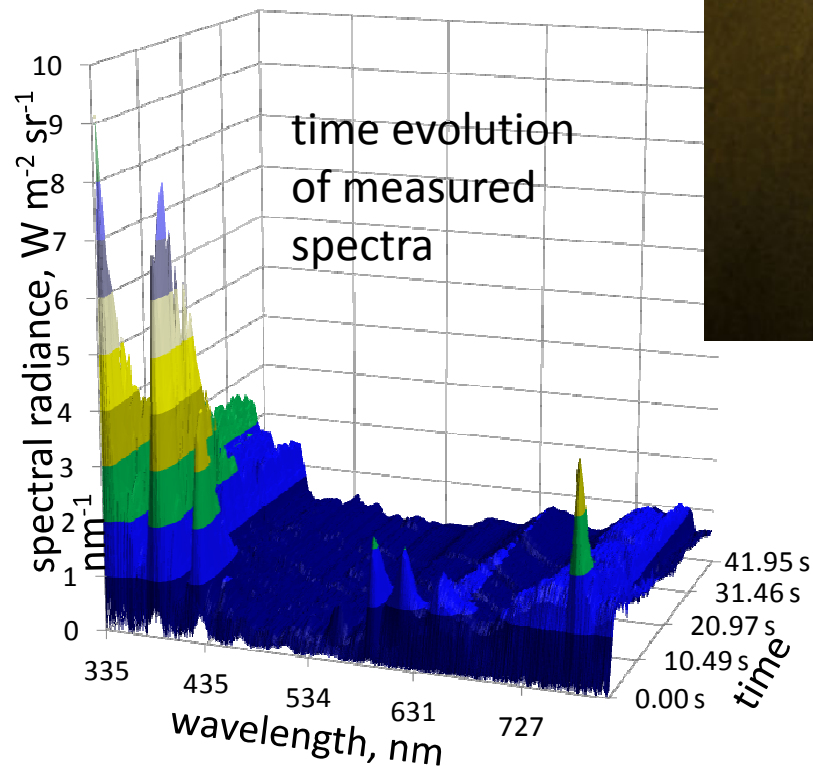
Raiche, G. A., Driver, D. M., “Shock Layer Optical Attenuation and Emission Spectroscopy measurements during Arc-jet Testing with Ablating Models”, AIAA 2004-825, 42<sup>nd</sup> AIAA Aerospace Sciences Meeting, Reno, NV, 2004.



## Recent OES measurements during SPRITE testing

- Emission spectroscopy measurements conducted to identify radiating species
- Two fiber coupled spectrometers used (200nm – 950nm) with focusing mirror
- Before test, optical paths close to each other barely touching the SPRITE nose tip
- Alignment before evacuation through a laser fed to the fibers

- Material recession would change the measurement position relative to the surface
- Continuous data acquisition during SPRITE and calorimeter insertion  
repetition rate  $\sim 2\text{Hz}$   
→ 81 spectra for the PICA model,  
4 spectra for the calorimeter

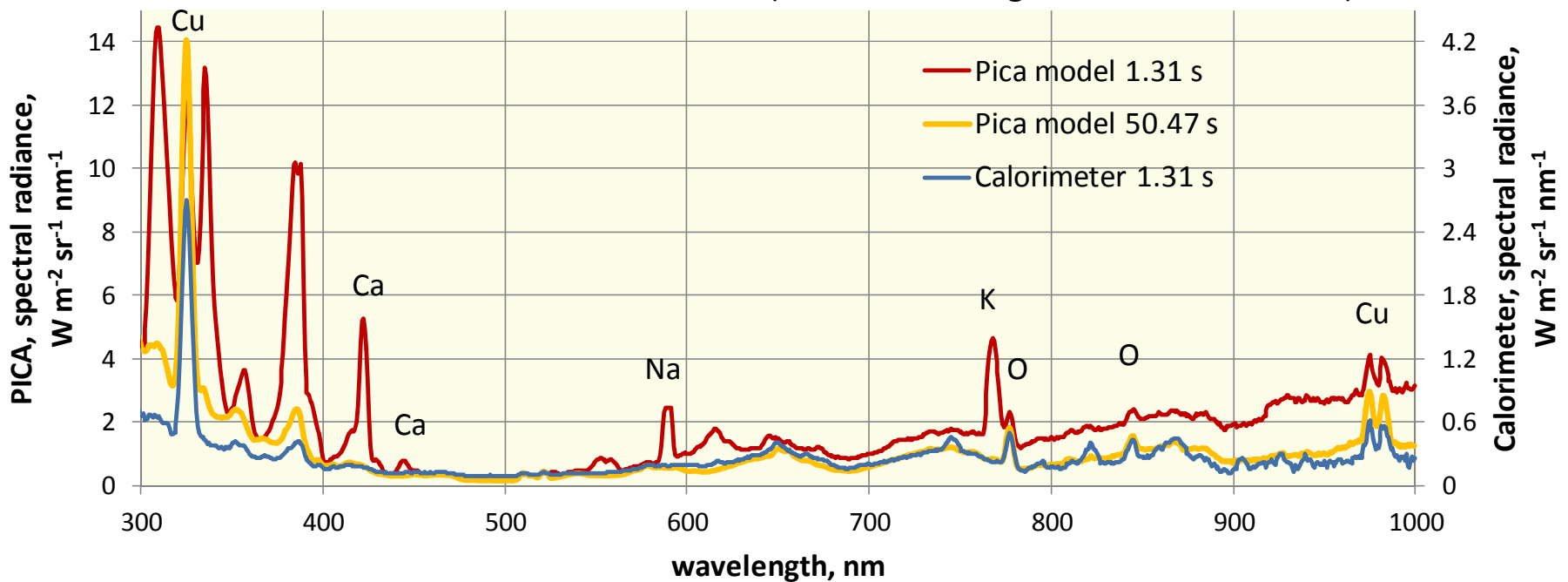




# OES-SPRITE: Spectral Overview



- At the end of the model insertion (50s), spectra are qualitatively similar to the ones in front of the copper calorimeter → no effect from ablation, outside BL
- Atom lines from Cu, O, N, molecular emission from N<sub>2</sub> and N<sub>2</sub><sup>+</sup>
- Shortly after insertion, emission peaks
- Continuum emission seen  
→ surface radiation
- Additional atom emission from Ca, K and Na (also seen during Stardust observation)

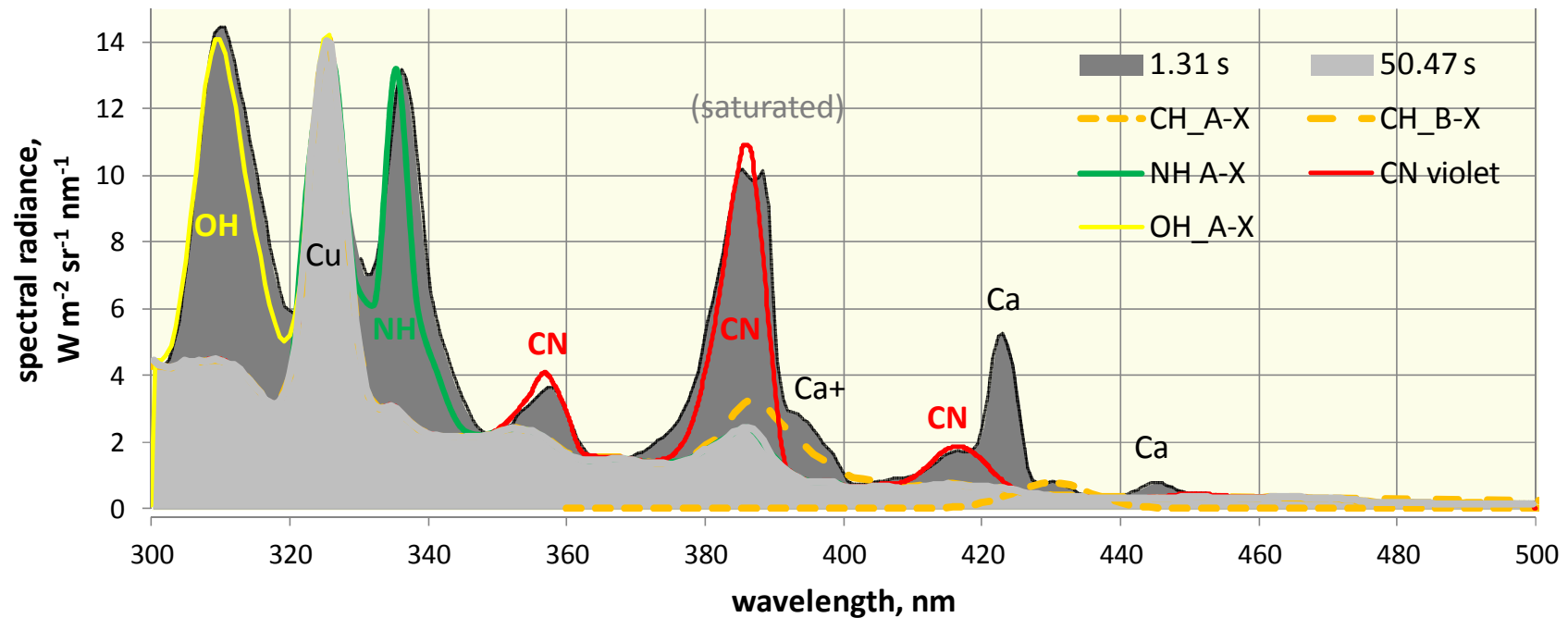




# OES-SPRITE: UV-spectra



- Difference between end and start of insertion should characterize the effect of ablation.
- Identified species: NH and OH, CN (strongly overlapping with  $N_2^+$ )



## Conclusions:

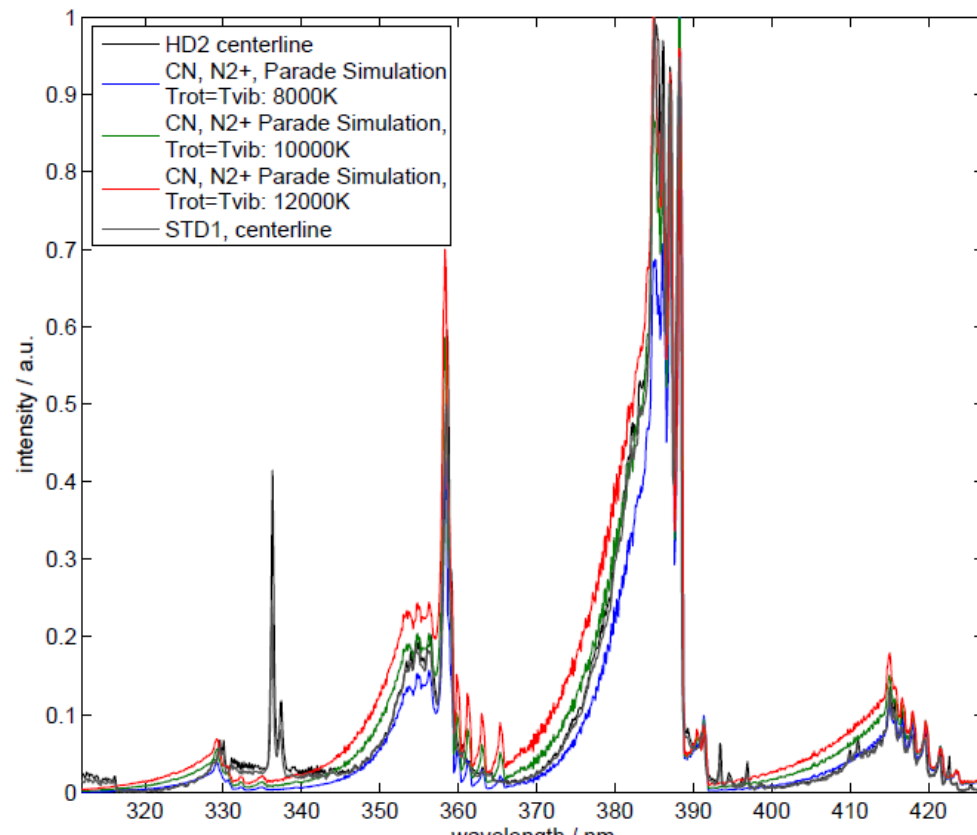
- The ablation products OH, NH, and CN were seen in emission from the post shock/boundary layer
- Further characterization (e.g. for CH) requires experiments in higher spectral resolution.
- The role of K and Na as strong atom emissions is still to be verified, H was not identified.



## European OES data



- Emission spectroscopy measurements in front of a AQ61 material sample
- Strong CN emission and clear emission of NH seen.
- Fit of simulated CN emission indicates temperatures between 8000K and 10000K (no material response model, simulation scaled to fit the CN measurements).

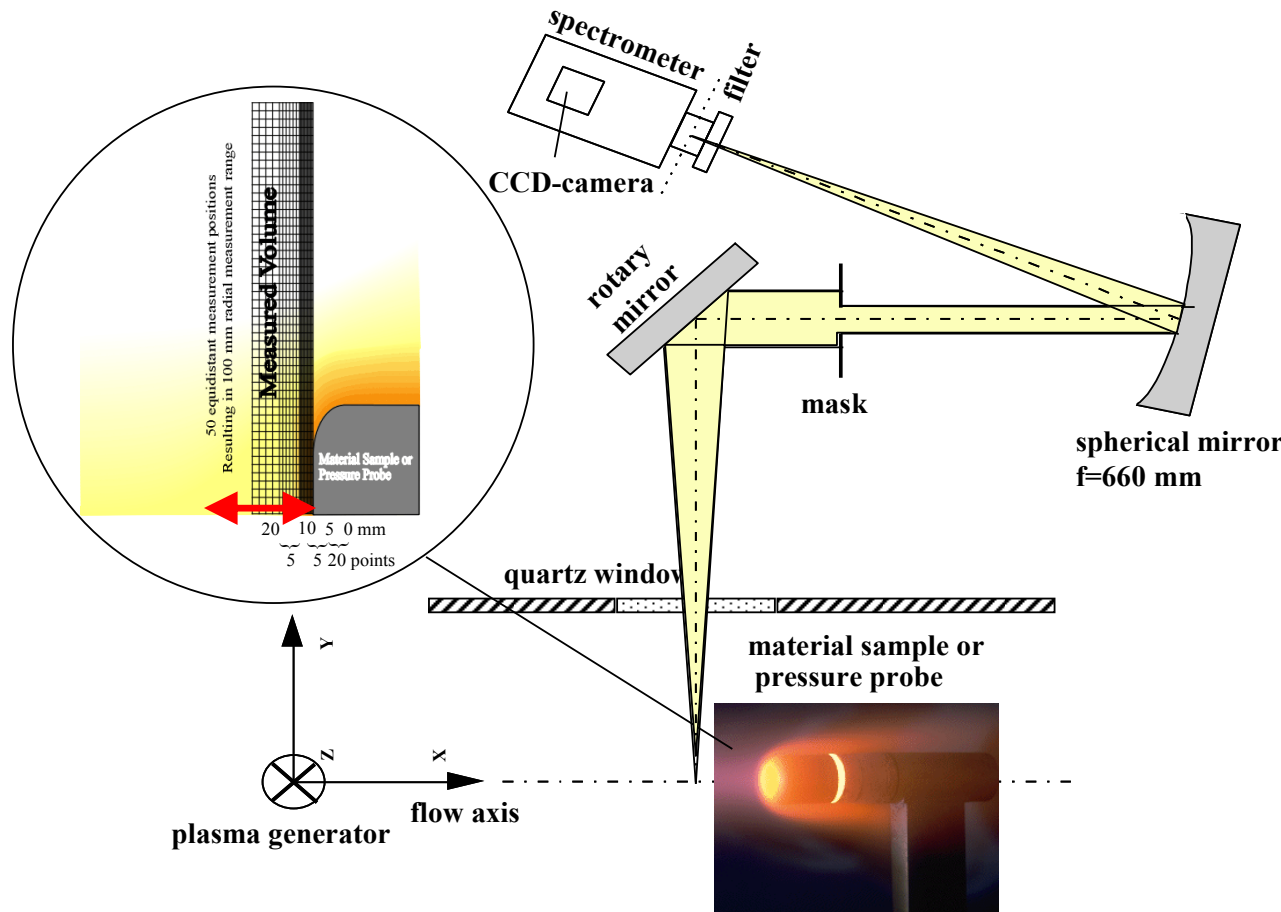


Emission spectra of samples STD1 and HD2 (European Ablator AQ61) at 3.5 MW/m<sup>2</sup> including PARADE simulated spectra.

Wernitz, R., Eichorn, C., Herdrich, G., Loehle, S., Fasoulas, S., Roeser, H.P., "Plasma Wind Tunnel Investigation of European Ablators in Air Using Emission Spectroscopy", AIAA 2011-3761, 42nd AIAA Thermophysics Conference 27 - 30 June 2011, Honolulu, Hawaii.

# Proposed experiments -OES

- Measure complete shock/post-shock/boundary layer system simultaneously  
→ image complete stagnation line on CCD.
- Best results expected from flat face model.



Spatial resolution:

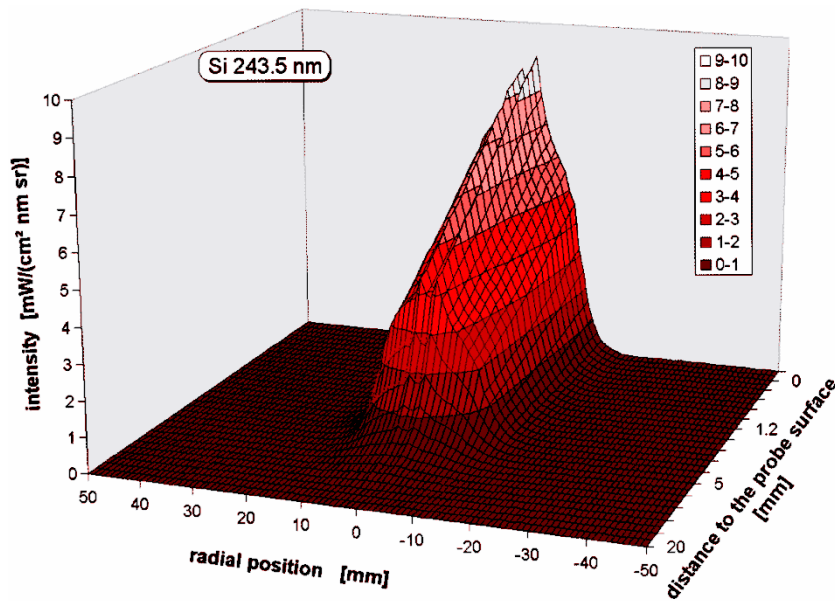
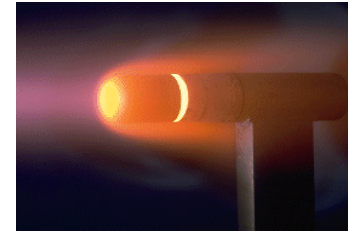
- 30 points in axial direction on the CCD over 20 mm → 0.12 mm max.
- 2x50 points radial, equidistant over 2x100mm → every 2mm with 0.1mm spot size for Abel-inversion → 3000 spectra in the flow field



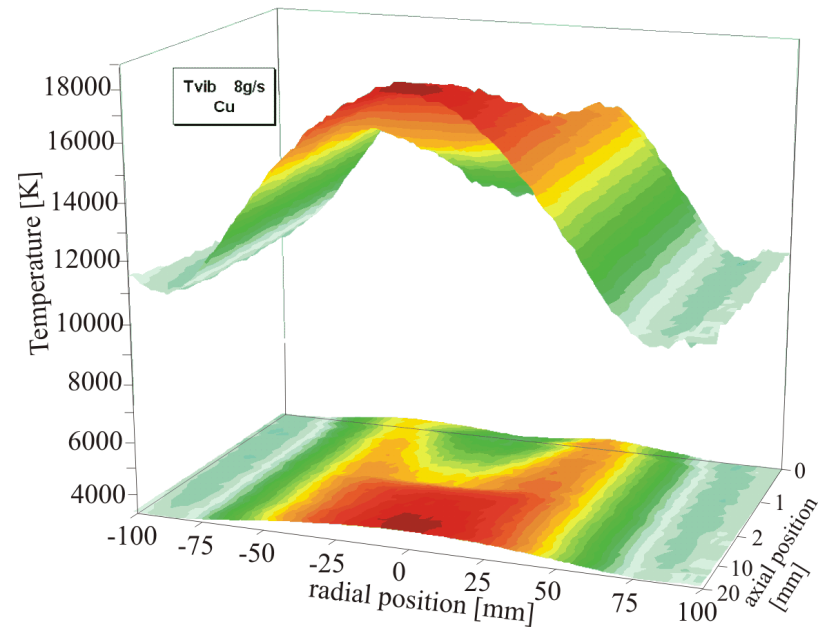
## Proposed experiments -OES



- Measure complete shock/post-shock/boundary layer system simultaneously  
→ image complete stagnation line on CCD.
- Best results expected from flat face model.



Si 243.5nm line emission in front pf SiC



N2+ vibrational temperature (Cu probe)





## Proposed experiments - PLIF

- Measure NH and OH with PLIF in front of the probe surface
  - spatially resolved distribution of NH and OH
  - boundary layer shape
- If appropriate calibration method can be developed, absolute particle densities would be accessible.
- Possibility of accessing CN (through CN violet system) and CH should be investigated
- Filtered imaging might support the boundary layer shape experiments

*I. A. Ben-Yakar and R.K. Hanson. Hypervelocity combustion studies using simultaneous OH-PLIF and Schlieren imaging in an expansion tube.*

*AIAA Paper 99-2453, 35th AIAA Joint Propulsion Conference, Los Angeles, CA, June 1999.*

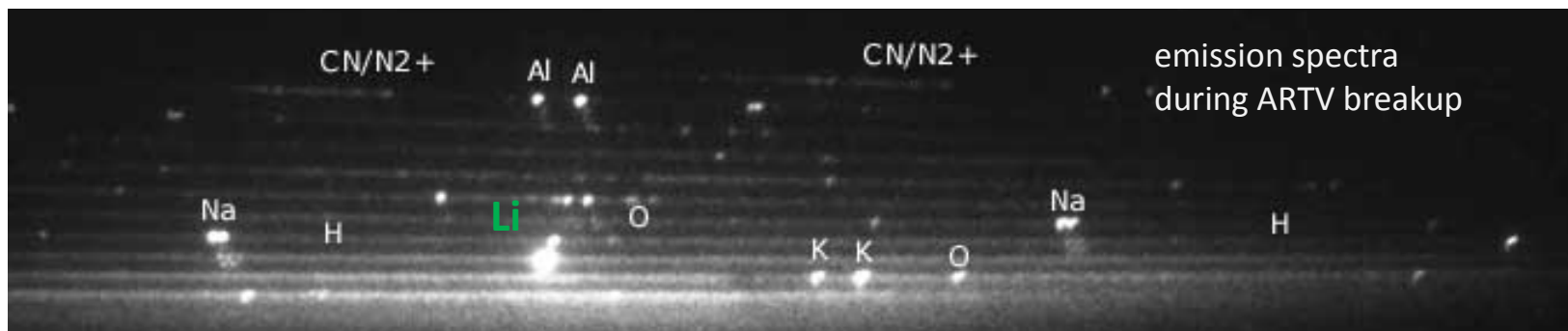
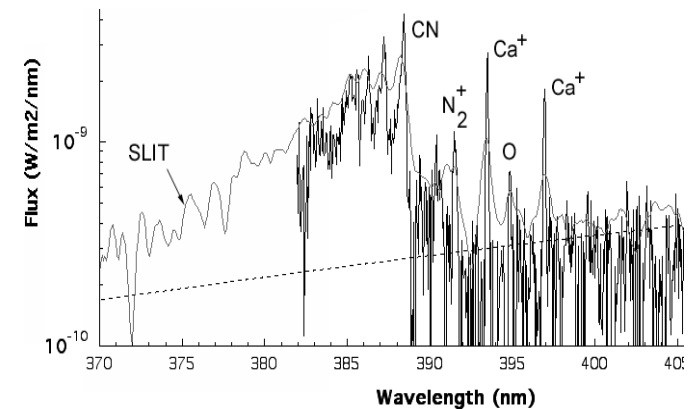




# Proposed experiments – tracer elements



- Both in ground based experiments and during airborne observation missions, tracer elements (Ca, Ca<sup>+</sup>, Na, K) were clearly seen in emission spectra, although present only in low concentrations.
- During the observation of the ATV Jules Verne break-up, spectral signatures were used to identify the fragments (e.g. lithium for the batteries).





## Proposed experiments – tracer elements



- Both in ground based experiments and during airborne observation missions, tracer elements (Ca, CA+, Na, K) were clearly seen in emission spectra, although present only in low concentrations.
- During the observation of the ATV Jules Verne break-up, spectral signatures were used to identify the fragments (e.g. lithium for the batteries).
- Seeding of the ablator with specific tracer elements at a defined depth would provide information when this particular depth is reached in recession or charring (depending on the seeding method).
- If different tracer elements are used, different depths or/and locations can be monitored with the same set-up.
- Spatial information can be extracted from non resolving measurements (e.g. airborne or ground based re-entry observations).
- A simple design could be realized by plugs (e.g. painted on the back side) which could be inserted in blind holes from the front surface (minimum invasive since not penetrating the heat shield material).
- In contrast to current recession sensors, no internal data acquisition would be required.



## Conclusions



- Recent emission spectroscopy data shows CN, NH, and OH inside the boundary layer in front of a PICA heat shield during arc-jet testing.
- For a validation of material response codes, in particular with respect to pyrolysis products, emission spectroscopic monitoring of the whole stagnation line is proposed for future experiments.
- The identification of NH and OH indicates the feasibility of PLIF experiments to determine the distribution of these products inside the boundary layer, even in form of particle densities if appropriate calibration methods can be designed.
- Measurements of recession and or char depth through seeding of the heat shield material with tracer elements are anticipated to yield valuable data on material response both during arc-jet testing and re-entry observations.
- Applicability of absorption techniques and filtered imaging are under investigation.



5<sup>th</sup> Ablation Workshop

Lexington, KY, Feb. 28 – March 1, 2012

# Backup Slides

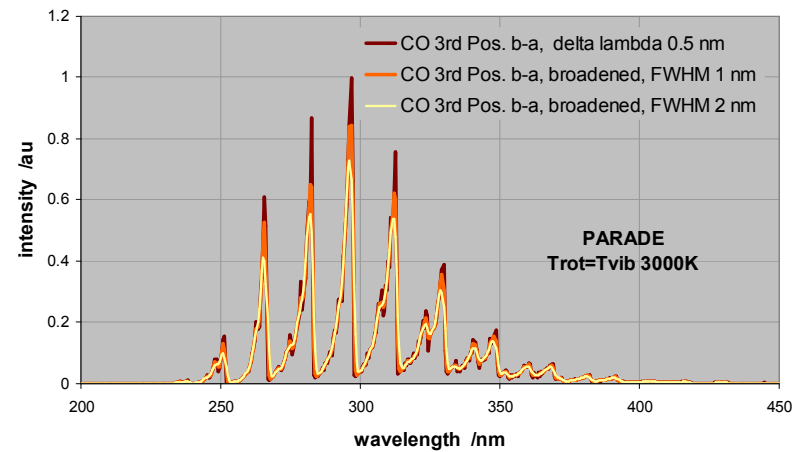
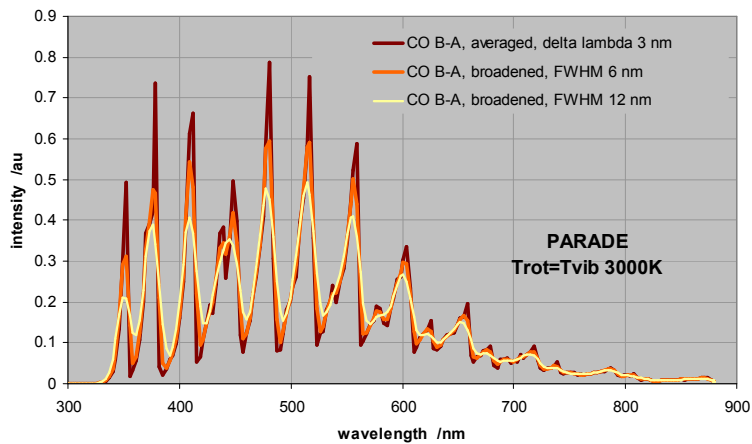
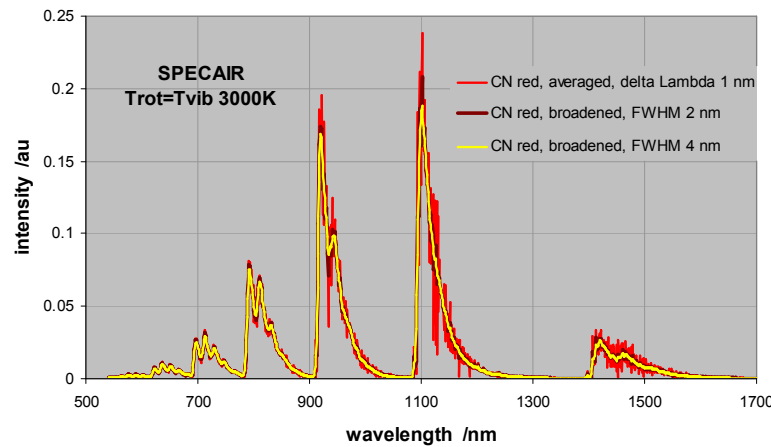




# Simulation Capabilities



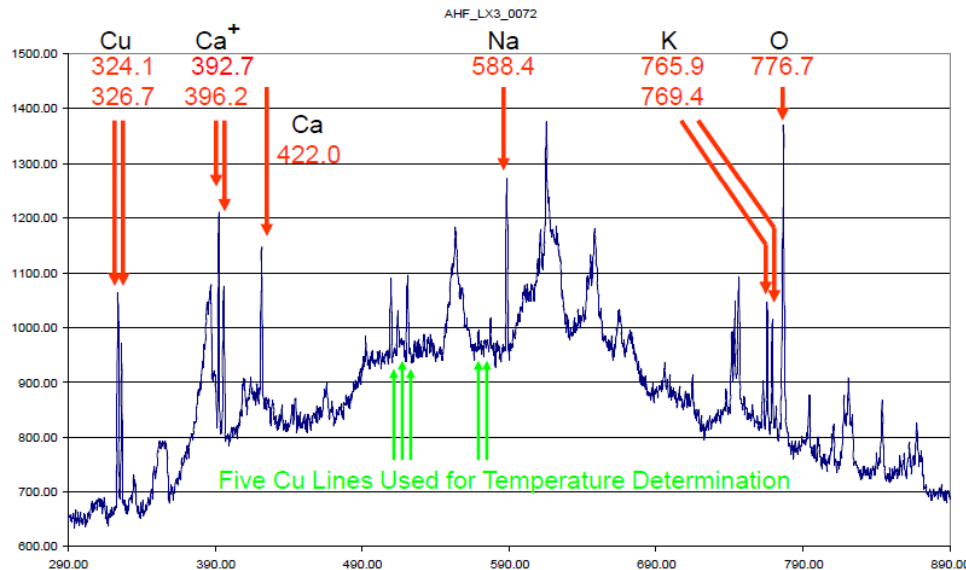
- Systems between electronic states: CN red, div CO systems



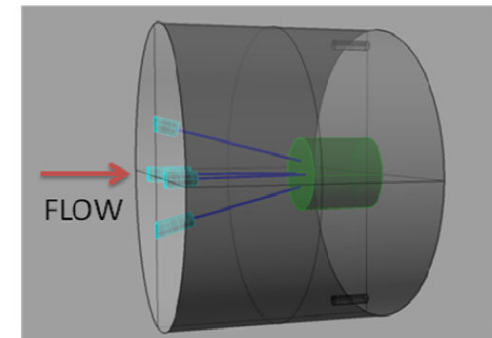
## Existing OES data (Langley/Ames)



- Investigation of PICA and carbon phenolic in flow of the AHF facility.
- No intensity calibration available
- No spatial resolution from surface through shock



**Figure 10** Visible-band spectra of hot gas cap identifying several atomic lines, including the Cu lines (green arrows) used for the path-averaged temperature measurement. Red numbers = raw measured wavelengths (nm) that give good agreement with known wavelengths for all 5 elements – if corrected upward by ~ 0.6 nm (based on our laboratory calibration with Hg and H lamps).



**Figure 2a.** Ghosted view of a typical PICA puck.



**Figure 2b.** Photograph of a PICA model.

Schott, T., Herring, G.C., Munk, M.M., Grinstead, J. H., Prabhu, D.K., “Fiber-Based Measurement of Bow-Shock Spectra for Reentry Flight Testing”, 48<sup>th</sup> AIAA Aerospace Sciences Meeting, Orlando, FL, 2010.



## Existing OES data (Ames)

- intensity calibrated emission spectroscopy
- wavelength range 400nm to 950nm
  - no comparison data in the UV
- focus on micro spallation through laser attenuation
- spatial resolution through shift of the optical axis relative to the recessing material surface
- Main ablation related radiation from CN

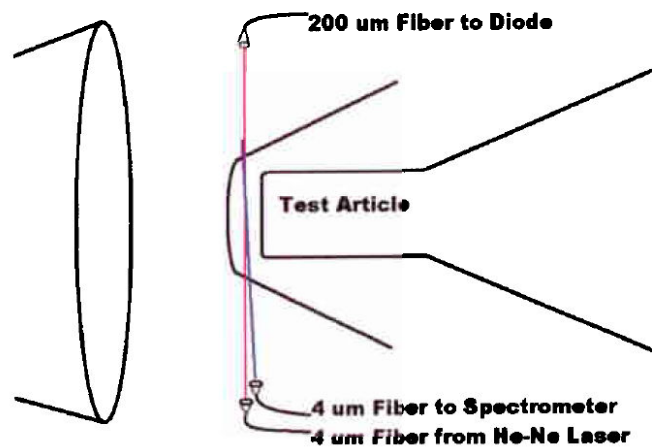


Figure 1. Spectrometer and laser attenuation lines of sight

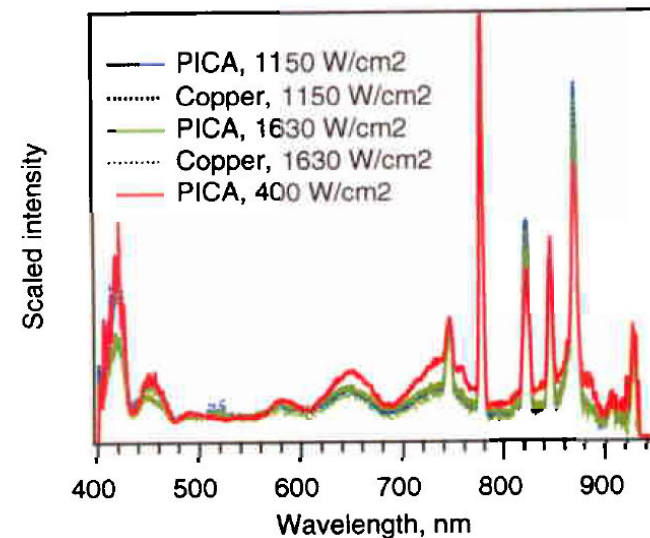


Figure 10: Shock layer spectra over PICA and copper, scaled to the peak at 777 nm for different heating rates.

Raiche, G. A., Driver, D. M., "Shock Layer Optical Attenuation and Emission Spectroscopy measurements during Arc-jet Testing with Ablating Models", AIAA 2004-825, 42<sup>nd</sup> AIAA Aerospace Sciences Meeting, Reno, NV, 2004.

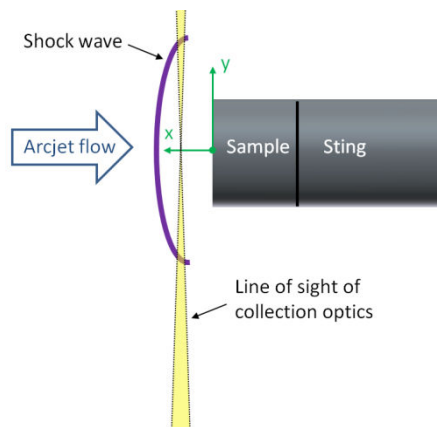




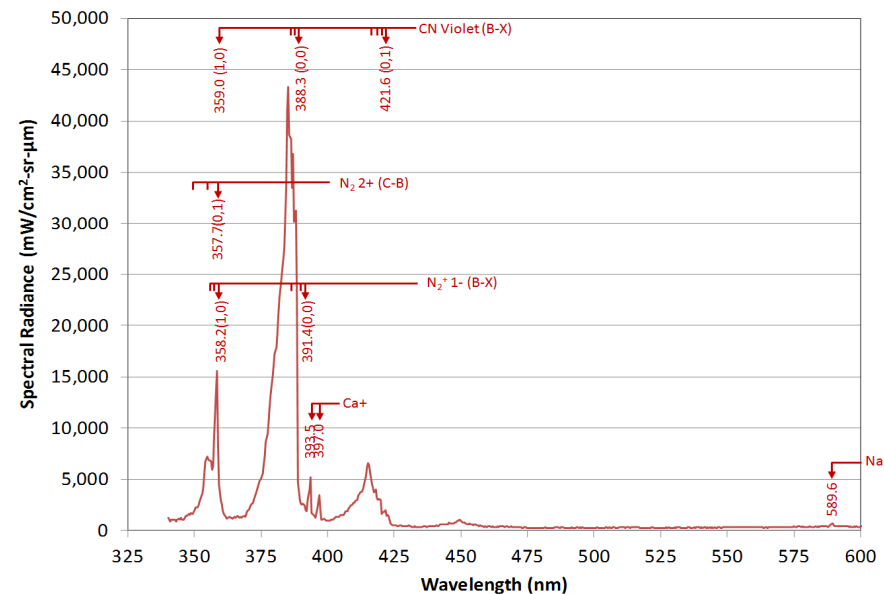
## Existing OES data (Langley)



- Intensity calibrated emission spectroscopy between 340nm to 950nm
- PICA and SiC material probes investigated
- Main ablation related radiation from CN, Ca lines observed
- UV experiments in preparation



Detailed schematic of setup in the test section, as viewed from the top. The sample is an end-on cylinder.



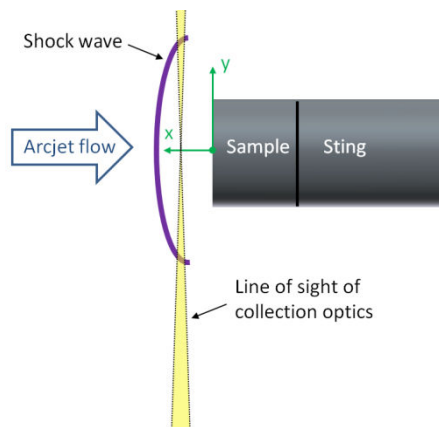
Detailed spectral radiance observed in Run 6 with the Air simulant gas flowing over a PICA test sample. Selected atomic and molecular lines are shown. This is an average of 6 spectra obtained within  $2.2 \pm 0.3$  mm in front of the sample.



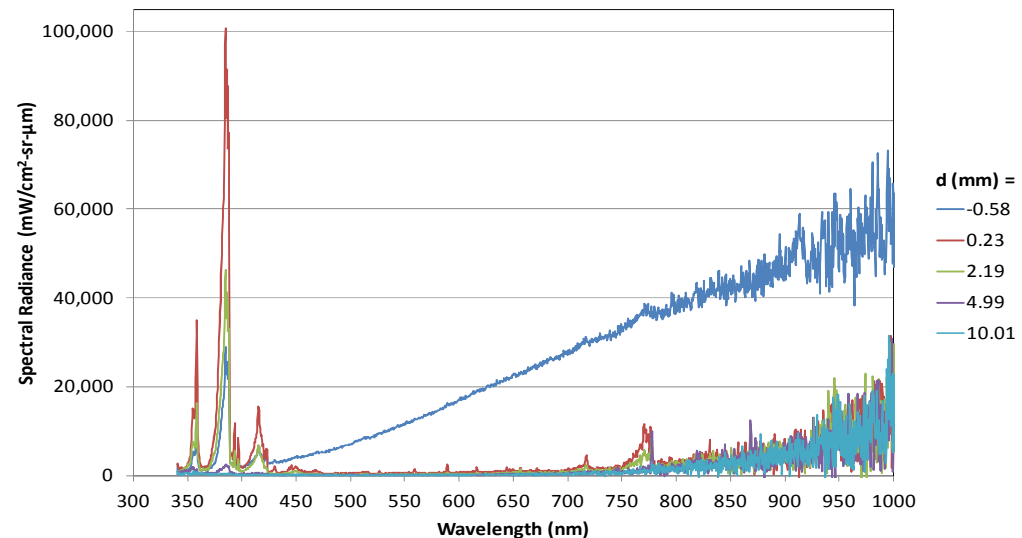


## Existing OES data (Langley)

- Intensity calibrated emission spectroscopy between 340nm to 950nm
- PICA and SiC material probes investigated
- Main ablation related radiation from CN, Ca lines observed
- UV experiments in preparation
- Spatial resolution through shift of the optical axis relative to the recessing material surface



Detailed schematic of setup in the test section, as viewed from the top. The sample is an end-on cylinder.



Emission spectra obtained with Air flow over the PICA material in Run 6 showing measurements at different spatial locations in front of the material. These spectra were obtained during the first stepper-motor scan of Run 6 (first 6 seconds of the run).



B.J. Kirby and R.K. Hanson. “Imaging of CO and CO<sub>2</sub> using infrared planar laser-induced fluorescence,” *Proceedings of the Combustion Institute*, 28, 253–259, (2000).

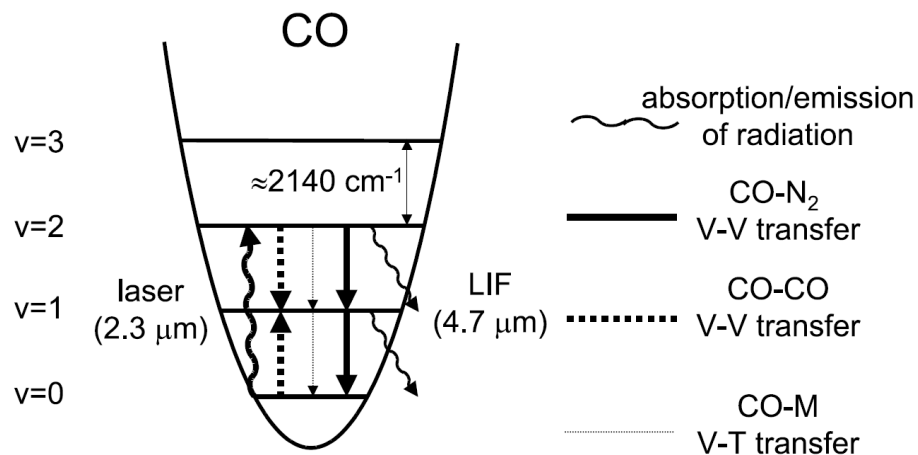


Figure 5: CO excitation/collection scheme.

M.D. Di Rosa and R.L. Farrow, “Two-photon excitation cross section of the *B–X* (0,0) band of CO measured by direct absorption,” *J. Opt. Soc. Am. B* **16**, (11), 1988-1994 (1999).