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Thin Films Technical Group

Fundamental Properties and Real-World Breakdown of Low Absorption Thin Films from the Visible to Infrared

Joey Talghader, University of Minnesota

12 October 2021

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About the Thin Films Technical Group

We serve the thin film community with 1000+ members

Our mission:

- Connect our community through webinars, technical events, and social media
- Bridge the fundamentals, the know-hows and the new developments in the field
- Promote networking and career development through continuous learning

Our recent webinars:

- Conformal Optical Coatings by Atomic Layer Deposition
- Environmental Stability of Electron-Beam Deposited Coatings
- Surface Coatings that Inhibit Infection by SARS-CoV-2
- Metasurfaces: New Generation Building Blocks for Emerging Optics

Connect with our Technical Group

Join our online community to stay up to date on our group's activities. You also can share your ideas for technical group events or let us know if you're interested in presenting your research.

Ways to connect with us:

- Our website at www.optica.org/ThinFilmsTG
- On LinkedIn at www.linkedin.com/groups/4783616
- Join us in-person at the Optical Interference Coatings Conference in Whistler, Canada on 19 – 24 June 2022

Attendees of OIC 2019, New Mexico, USA

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Today's Speaker

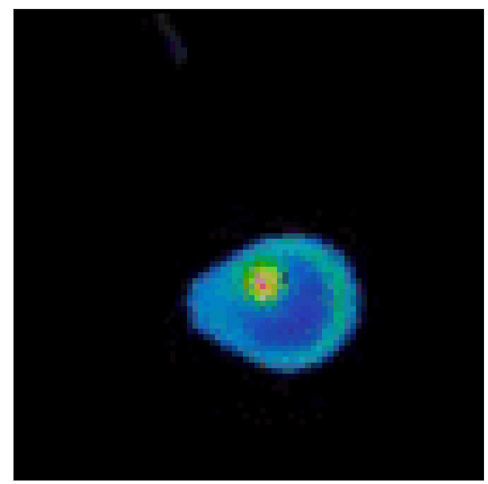
Joey Talghader

University of Minnesota

- Received his BSEE from Rice University and his M.S. and Ph.D. the University of California at Berkeley.
- After working in industry in flash and other nonvolatile memory, he joined the faculty at the University of Minnesota where he is now a Professor.
- Extensively involved in thermal infrared and radiation heat transfer devices, the mechanical design of optical coatings and their materials science, and the miniaturization of micro-opto-mechanical systems.

Fundamental Properties and Real-World Breakdown of Low Absorption Thin Films

J. J. Talghader
University of Minnesota



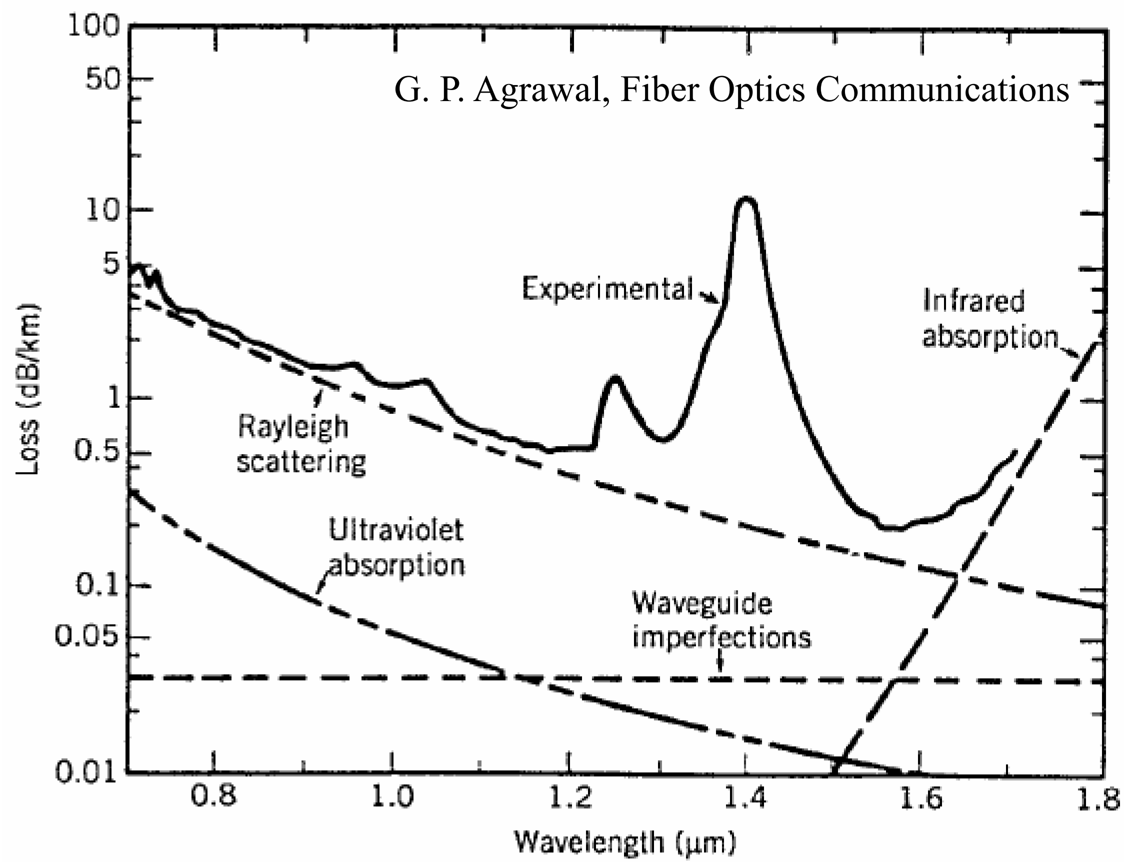
OSA Thin Film Webinar, October 12, 2021 joey@umn.edu

Outline

- Losses and Surface Heating
  - material loss
  - heat conduction
- Low Loss Measurement
- Contamination-Induced Breakdown
- Laser Accelerated Particles

Primary Material Losses

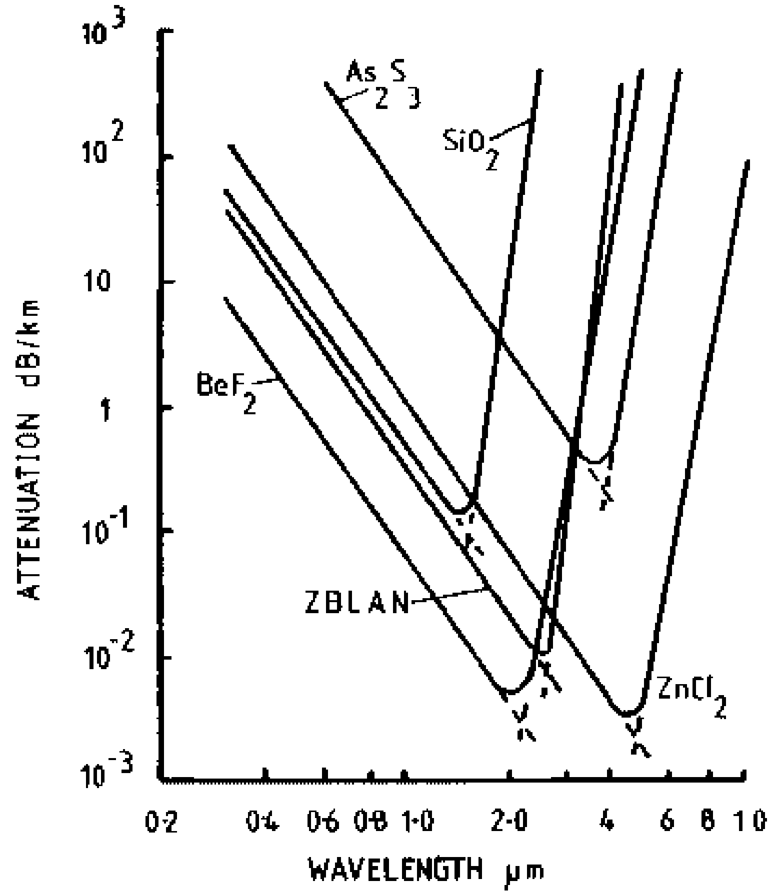
- Infrared Absorption
- Scattering
  - Rayleigh
  - Interface
  - Defects
  - Other
- UV Absorption
- Impurities



In silica and most other optical coating materials, the dominant fundamental losses are the IR absorption tail and Rayleigh scattering

Fundamental Absorption

- Consider a high-reflectivity optical coating with a loss of 1ppm with a penetration depth of 1.5µm (3µm total weighted travel in coating).
- Roughly an equivalent absorption coefficient of $\alpha = 3.3 \times 10^{-2}\ cm^{-1}$

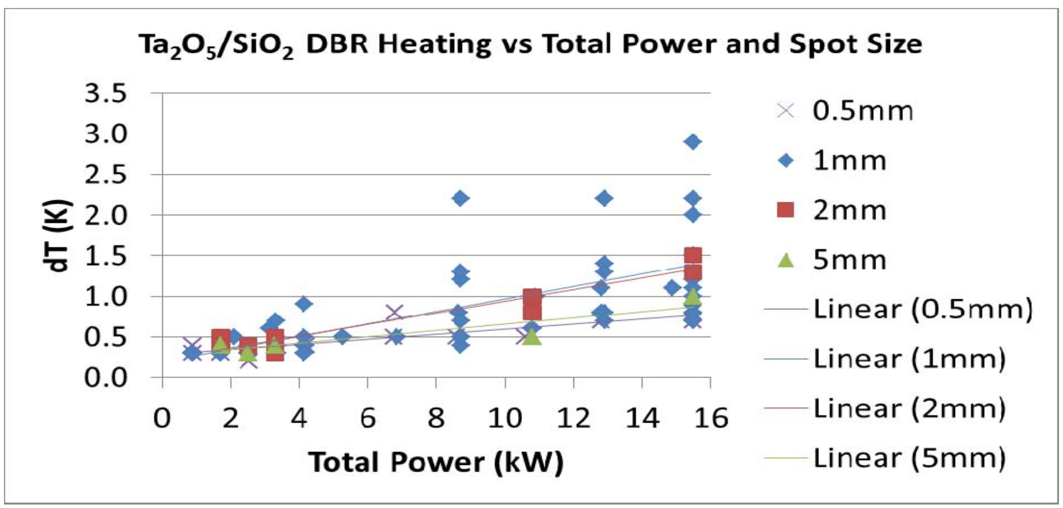


J. A. Savage, 1987
Materials for Infrared Fiber Optics

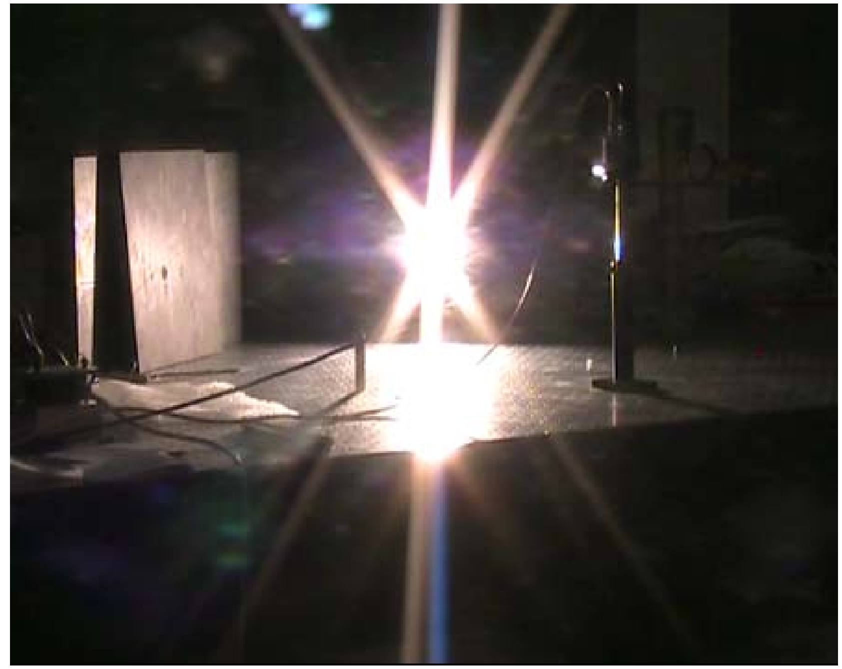
The fundamental absorption loss of silica at 1.55µm corresponds to an absorption coefficient of about $\alpha = 4.6 \times 10^{-7}\ cm^{-1}$

Coating Loss and CW Laser Optics

- In CW laser damage testing of high quality optics (i.e. absorption a few ppm or less) nothing usually happens
- Surface heating is minimal
- Many MW/cm^2 of CW power can be applied
- Failures are rare and random and typically do not correlate well with the coating absorption



Surface Images During Testing



These images were captured at the same moment. On the left is the high reflectivity optic under test. On the right is the reflected beam hitting a carbon block and lighting up the room.

Coating Loss and Surface Temperature

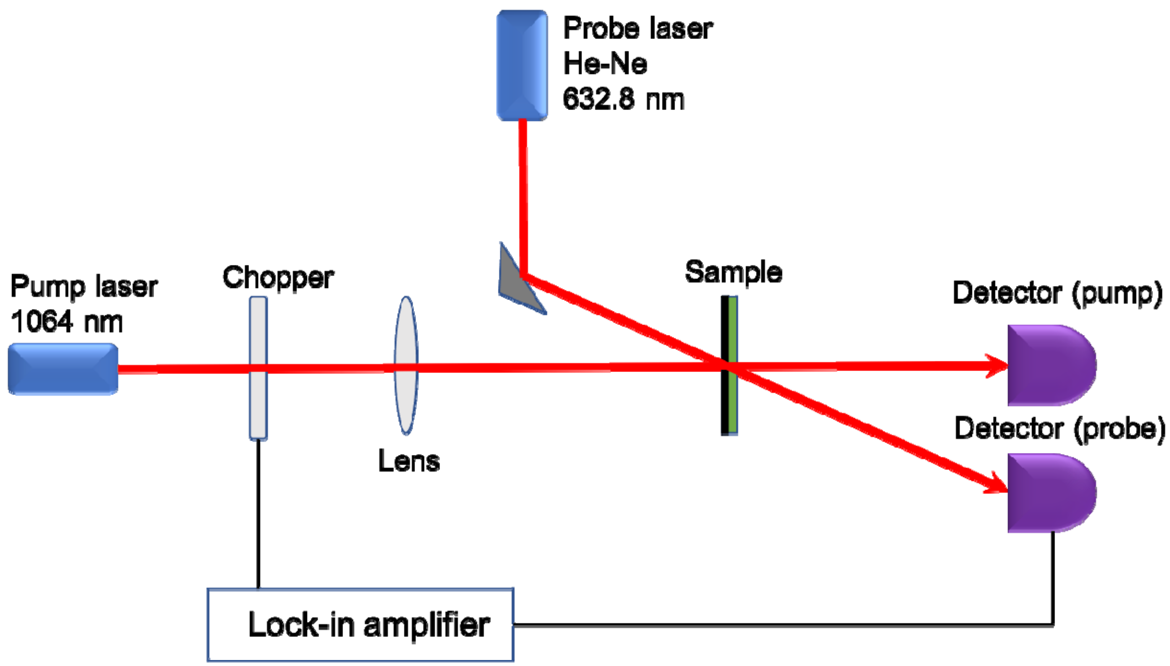
Is such a low rise in surface temperature reasonable?

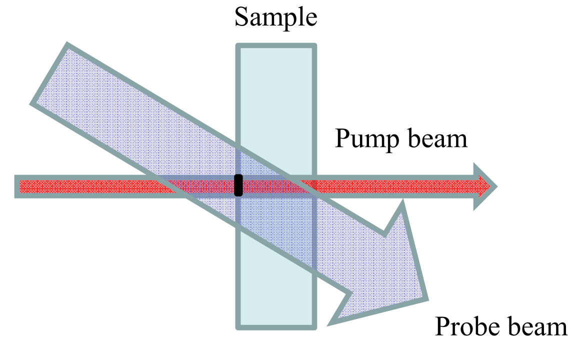
$$q = k\, dT/dx$$

- Thermal Conductivity of Fused Silica, k = 1.38W/(m-K)
- Assume we have a 3” fused silica wafer 1mm thick uniformly illuminated with 3MW CW laser
- 1ppm absorption HR coating absorbs about 0.106 W/cm^2

- ΔT across wafer (front to back) would be ~ 0.8K

Photothermal Common Path Interferometry (PCI) Concepts





- 1064 nm pumped beam is focused on the surface of the sample.
- A lower-powered (He-Ne) probe beam is illuminated over the same area.
- The pump beam heats the sample slightly and changes the surface shape and refractive index.
- This causes the phase shift between center and outer ring of probe beam

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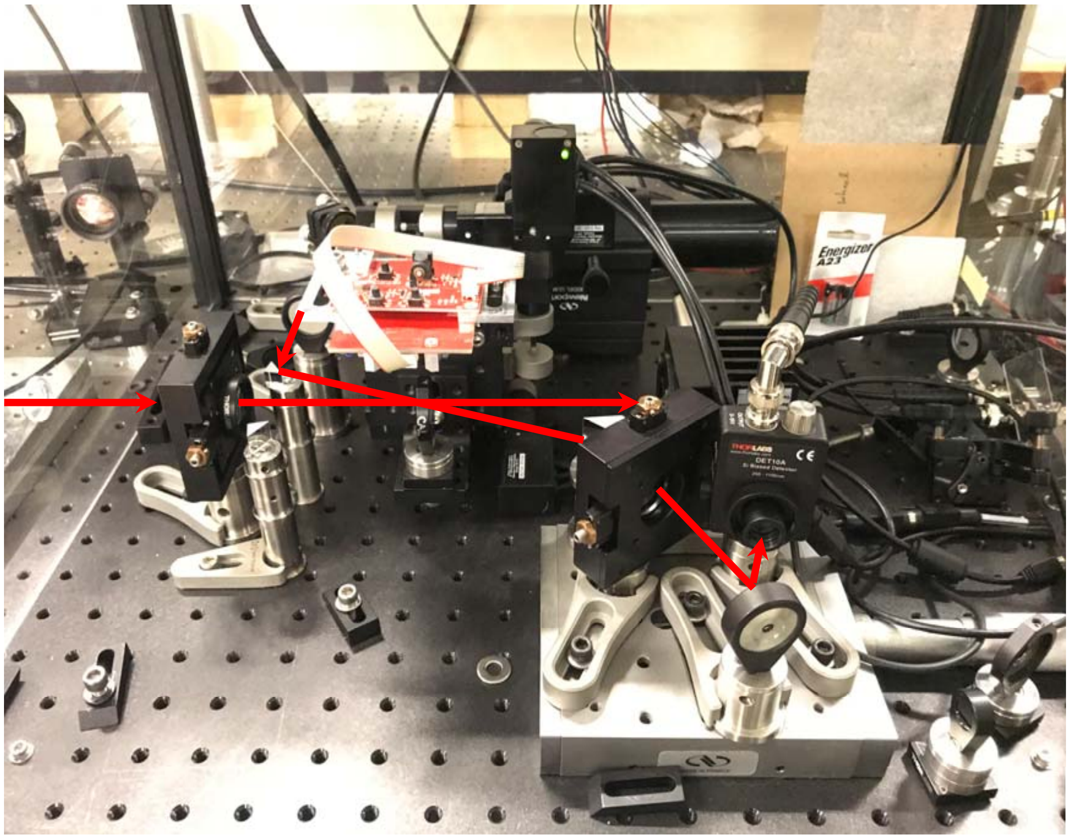
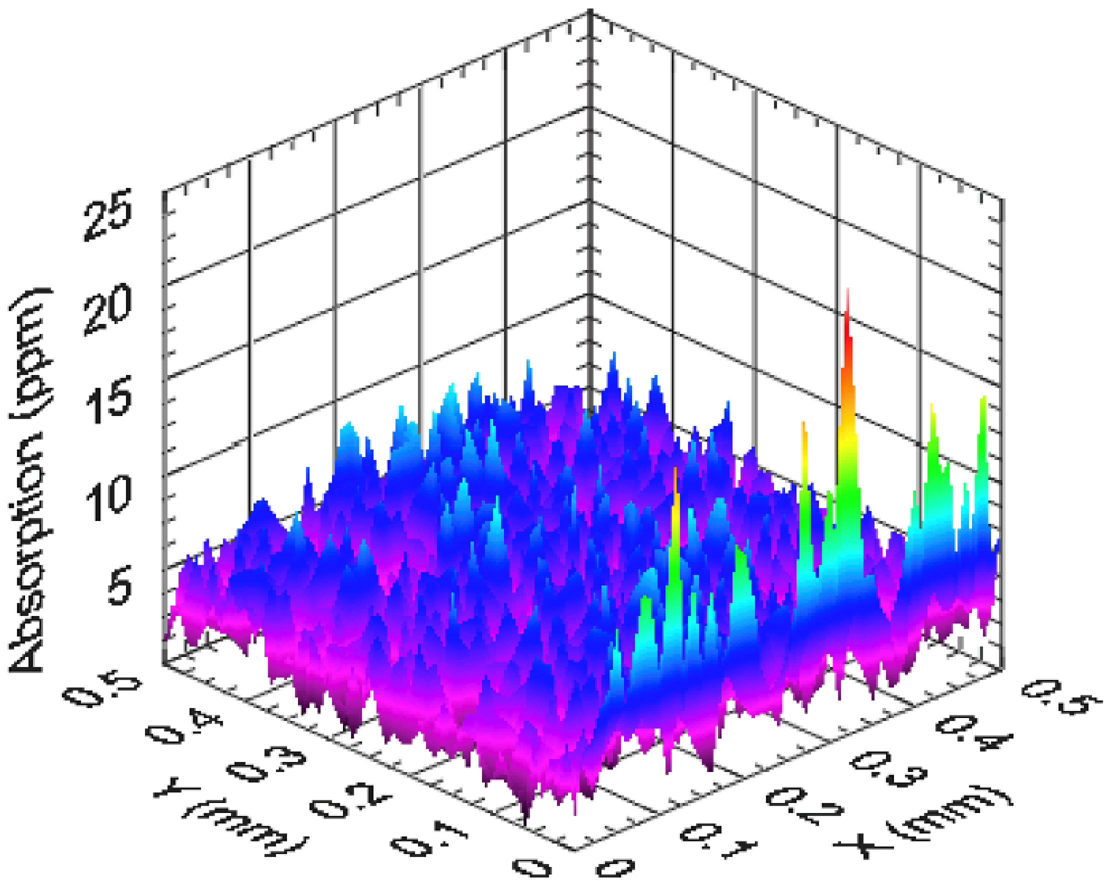
Experimental Setup: PCI



- Originally developed by A. Alexandrovski et. al.
- PCI – Photothermal Commonpath Interferometry system
- PCI is one of the best systems to measure absorption loss in highly transparent optics
- Near-IR region measurements (with 1064 or 1070 nm pump laser source)
- Also modified so we can measure SWIR, MWIR, and LWIR

Typical Absorption Data



All-Silica

- X-Y surface scan
- Absorption spikes almost always seen
- Absorption of films of most optical coating materials are low
- This sample contains low density silica
- Quarter wave thicknesses

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Contamination-Induced Breakdown

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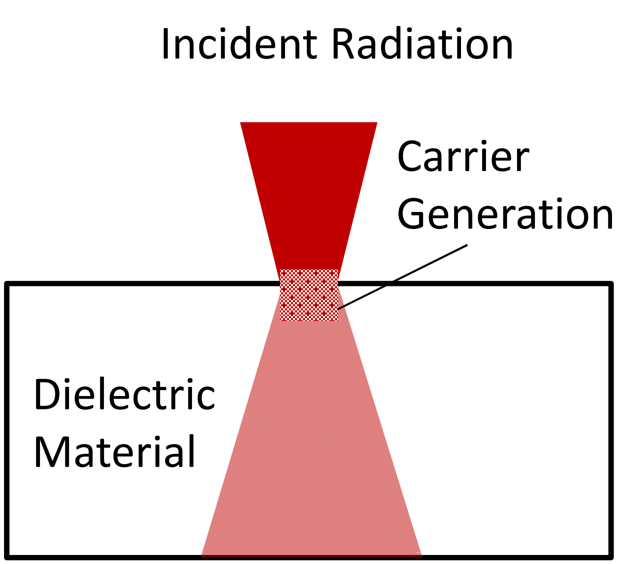
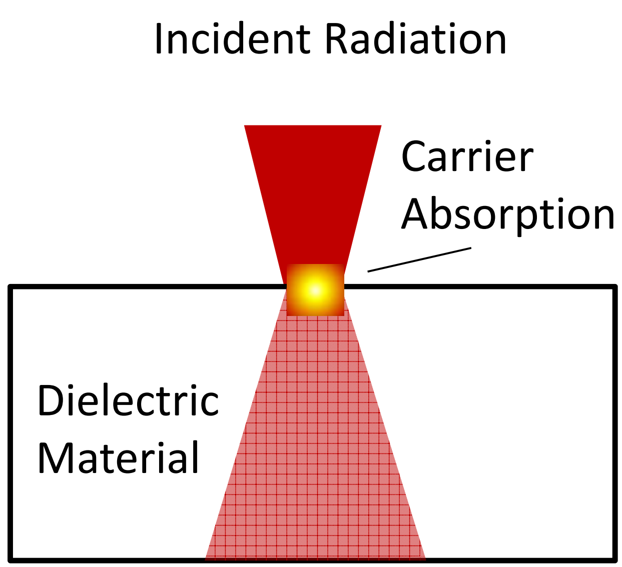
Short pulse damage: Clean Samples

Step 1: Carrier Generation



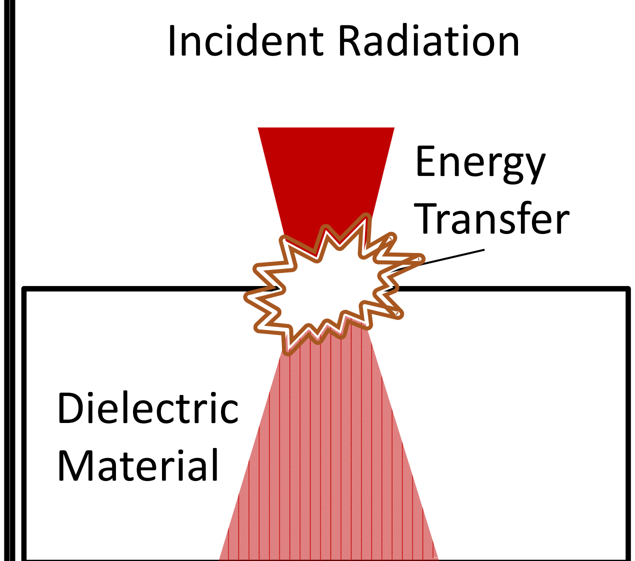
Carriers generated by non-linear high-field effects

Step 2: Carrier Absorption



Free-carriers strongly absorb and transfer energy

Step 3: Energy Transfer



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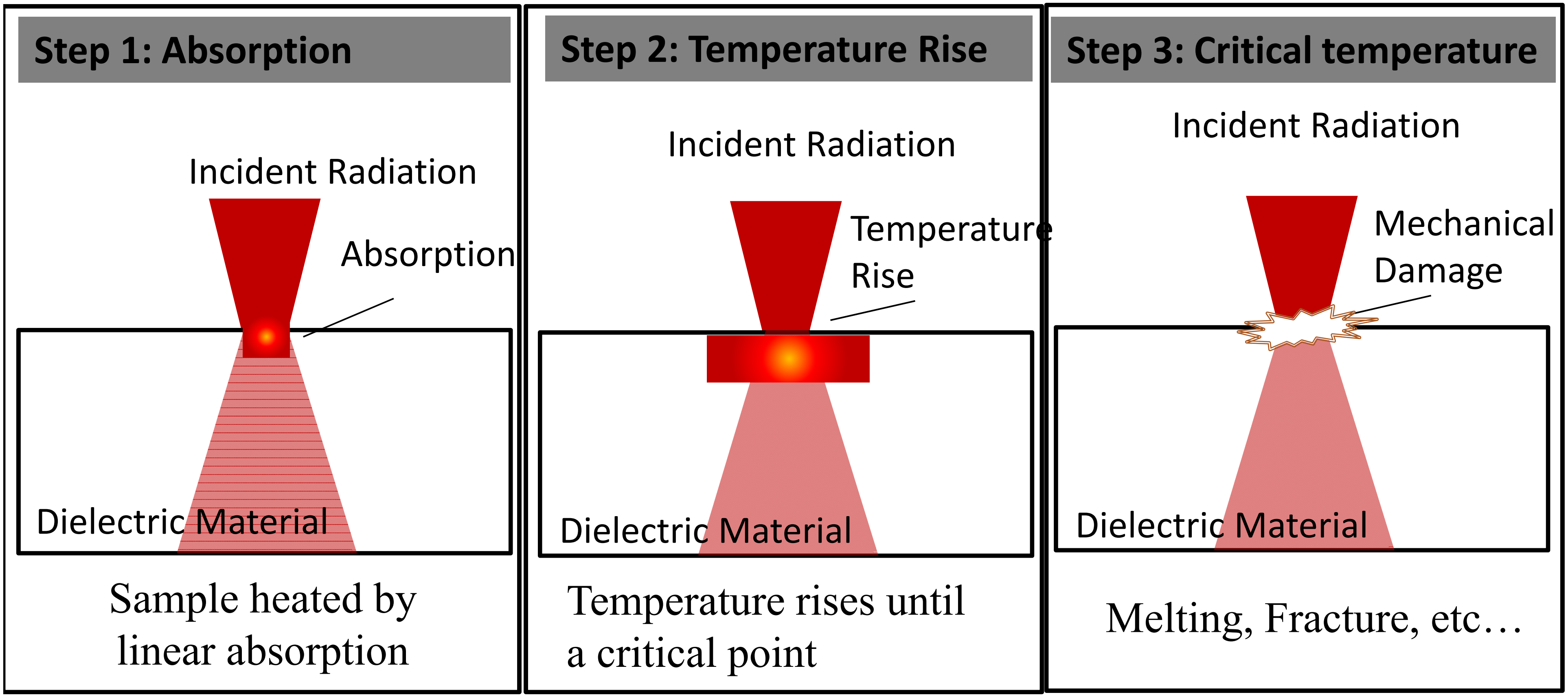
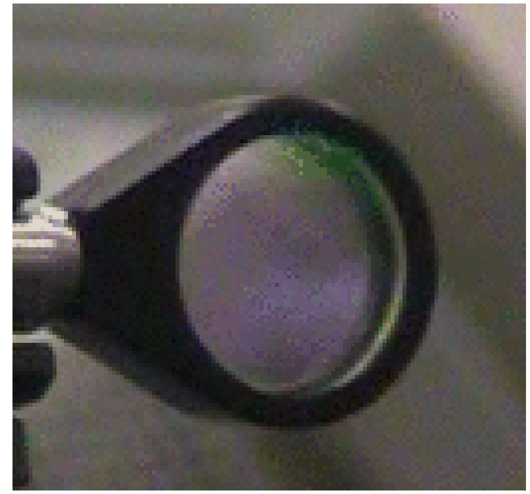
Long pulse damage: Clean Samples



Contaminated Optics under High-Power CW Fluence

- Short burst of light is emitted from the surface
  - Surface flash broad but not quite thermal
- Particles rapidly heat to evaporation
  - Migrate over and evaporate from surface
  - Surviving particles tend to coalesce
  - Process takes less than a millisecond
  - Surface left with spots of flat residue
  - Conditioned residue higher in hydrocarbon content
  - Volume of the material on the surface decreases
- Optical surface itself heats substantially
  - Surface temperature can decrease as material evaporated
  - Surviving optics have higher laser damage thresholds
  - Temperature of conditioned surfaces reduced
- Subsequent tests induce little change



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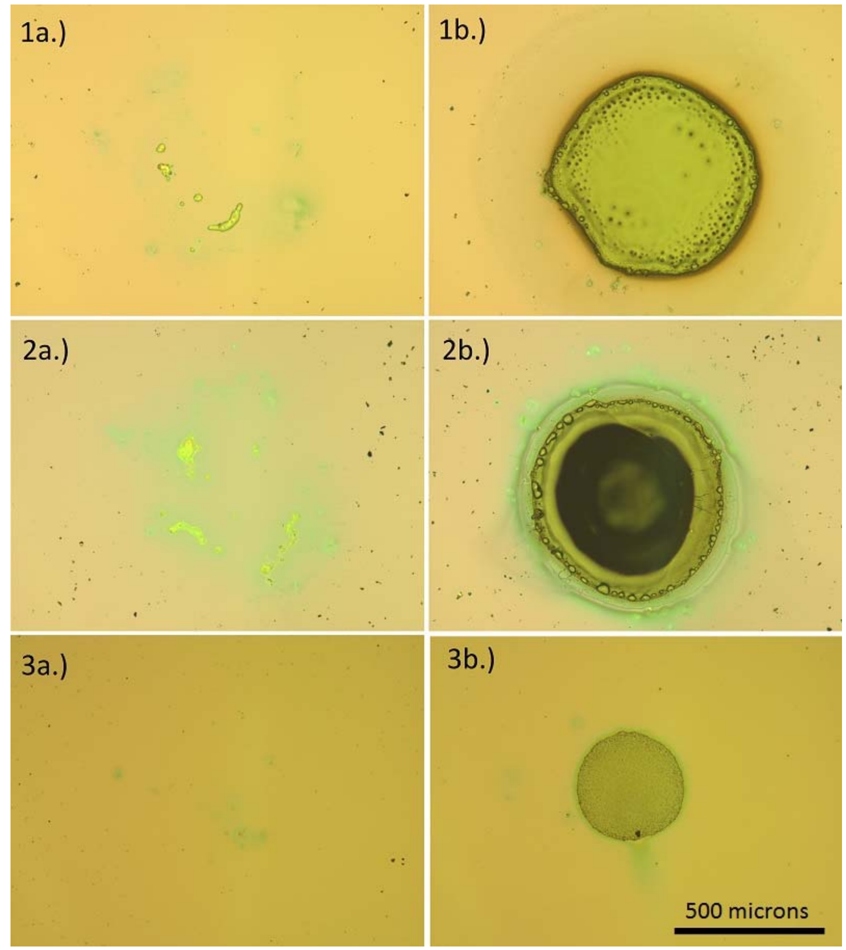
Half-Wave Coating Damage: Low vs. High Band Gap Materials

- During testing of <u>half-wave coatings</u>, low band gap materials failed with complete film delamination.
- Higher band gap materials often exhibited cracking but not compete film removal unless far above threshold
- Neither case led to significant substrate damage like that of the <u>high reflectivity mirrors</u>



Carbon-contaminated λ/2 coatings after testing.
1a) titania at 128 $kWcm^{-2}$, 1b) titania at 155 $kWcm^{-2}$,
2a) tantala at 1 $MWcm^{-2}$, 2b) tantala at 1.5 $MWcm^{-2}$,
3a) hafnia at 3.6 $MWcm^{-2}$, 3b) hafnia at 8.3 $MWcm^{-2}$

University Of Minnesota

Potentially Relevant Materials Properties

Film	Absorption	Thermal Conductivity
Titania	3.5 ppm $\lambda/2$	$3\ \mathrm{W\ m^{-1}\ K^{-1}}$
Niobia	2 ppm DBR	$1.5\ \mathrm{W\ m^{-1}\ K^{-1}}$
Tantala	12 ppm $\lambda/2$, 0.7 ppm DBR	$3\ \mathrm{W\ m^{-1}\ K^{-1}}$
Hafnia	35 ppm $\lambda/2$, 20 ppm DBR	$1.2\ \mathrm{W\ m^{-1}\ K^{-1}}$
Silica	1.5 ppm $\lambda/2$	$1.1\ \mathrm{W\ m^{-1}\ K^{-1}}$

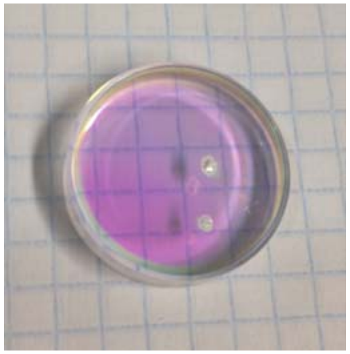
Film	Melting Point	Bandgap
Titania	1775 ^{o}C	3 eV
Niobia	1520 ^{o}C	3.85 eV
Tantala	1918 ^{o}C	4.3 eV
Hafnia	2758 ^{o}C	5.7 eV
Silica	1723 ^{o}C	9 eV

Only the bandgap fits the observed trends

Other Hints of a LIDT Band Gap Dependence

- Despite significantly lower intrinsic absorption, contaminated tantala-silica DBRs failed near 180 kWcm^{-2} while contaminated hafnia-silica DBRs survived upwards of 1 MWcm^{-2}
- Thermal behavior opposite of that when pristine
  - 2 ^{o}C vs. 43 ^{o}C at 3MWcm^{-2}
- For similar irradiances the substrate damage under tantala DBRs was far more extensive

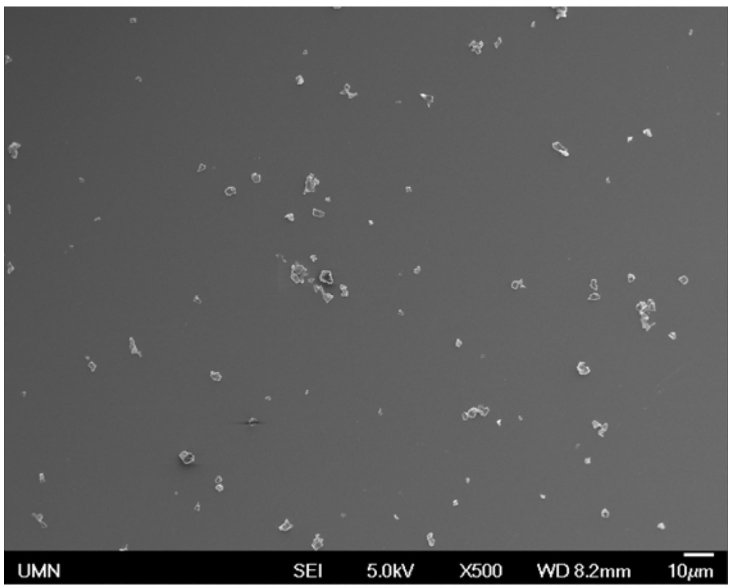
Hafnia-silica damage at 1 and 3 MWcm^{-2}

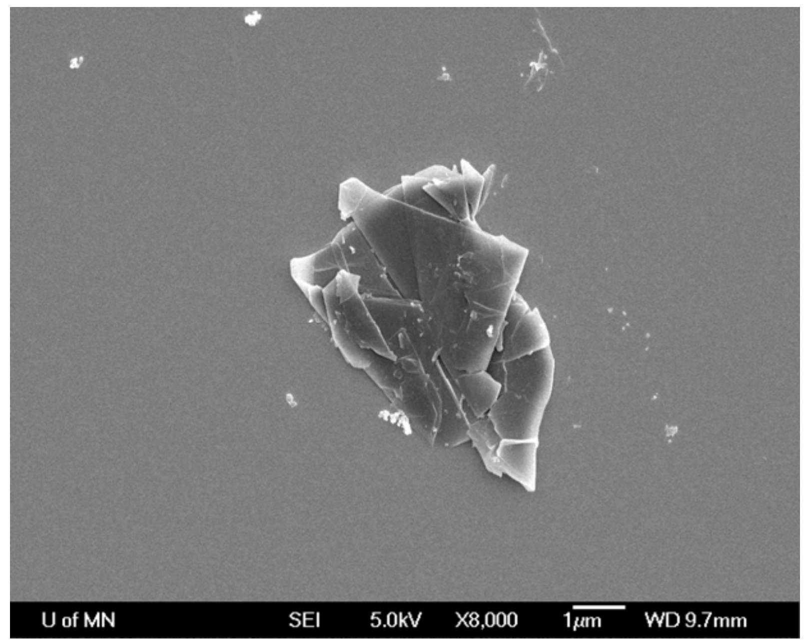


Tantala-silica damage at 2.5 MWcm^{-2}

Sample Contamination

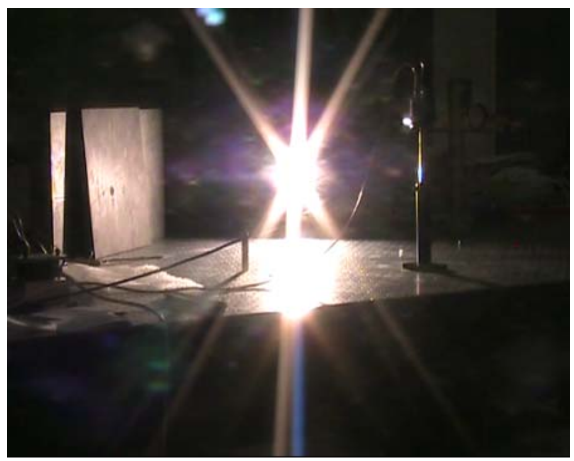
- Samples are intentionally contaminated with carbon-based particles with a size distribution centered around 8μm (but sold as 20-50μm)
- Particles in water suspension, which is applied to sample and evaporated
- Varying particle and mass densities used per unit area but kept constant in materials comparisons

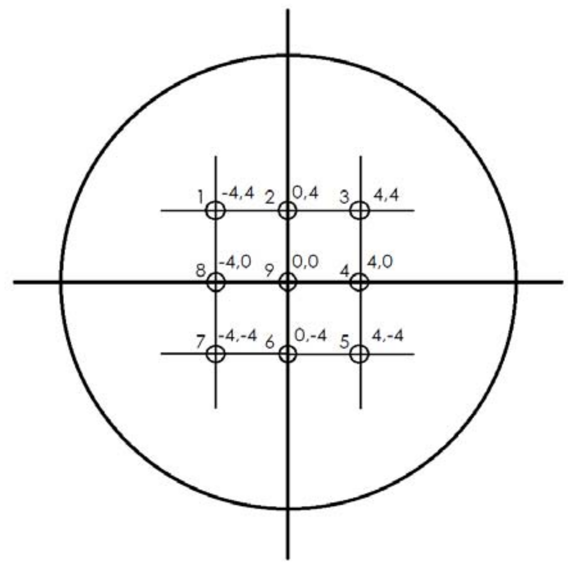




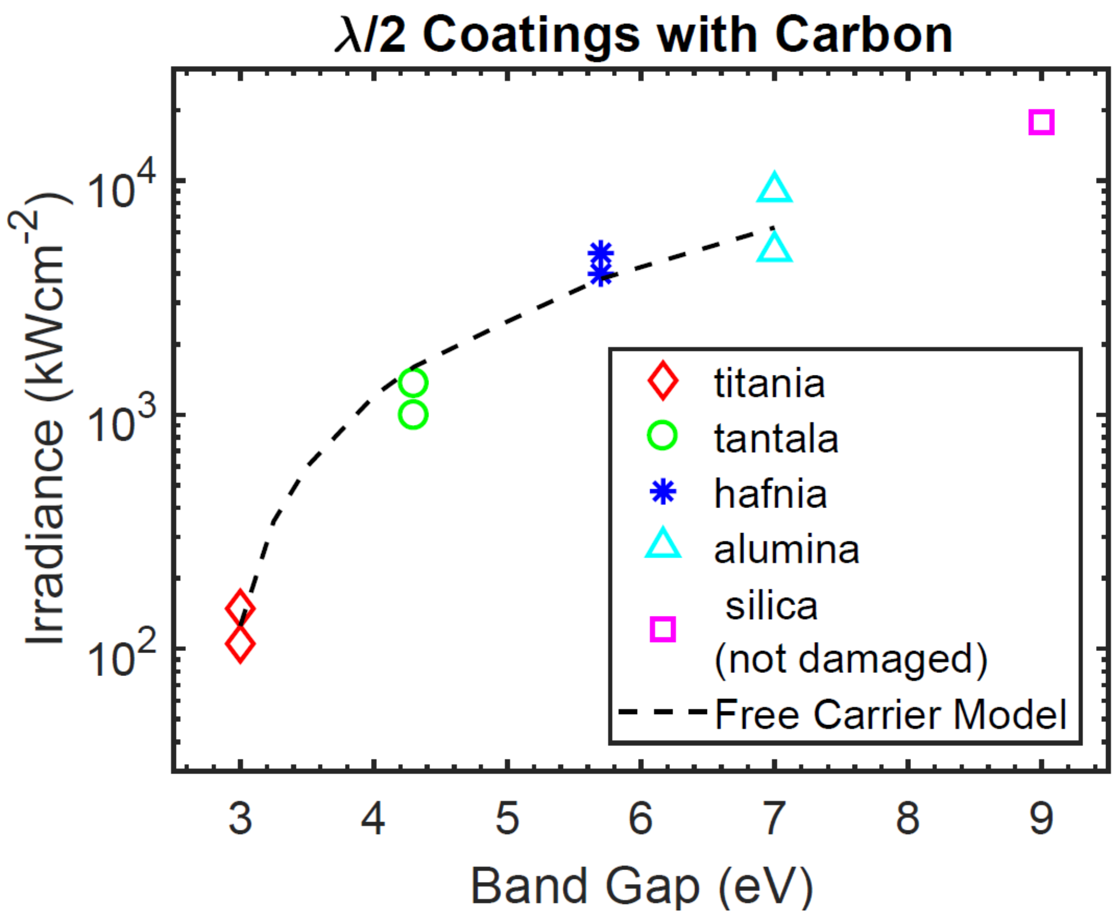
Laser Damage Testing

- A 17kW IPG Yb-doped fiber laser at the Penn State Electro-Optics Center illuminates samples for 30 seconds
- Each sample has 9 test points
- Each point is tested only at one power then the power is stepped up at a new site
- Upon catastrophic failure, a new sample is used



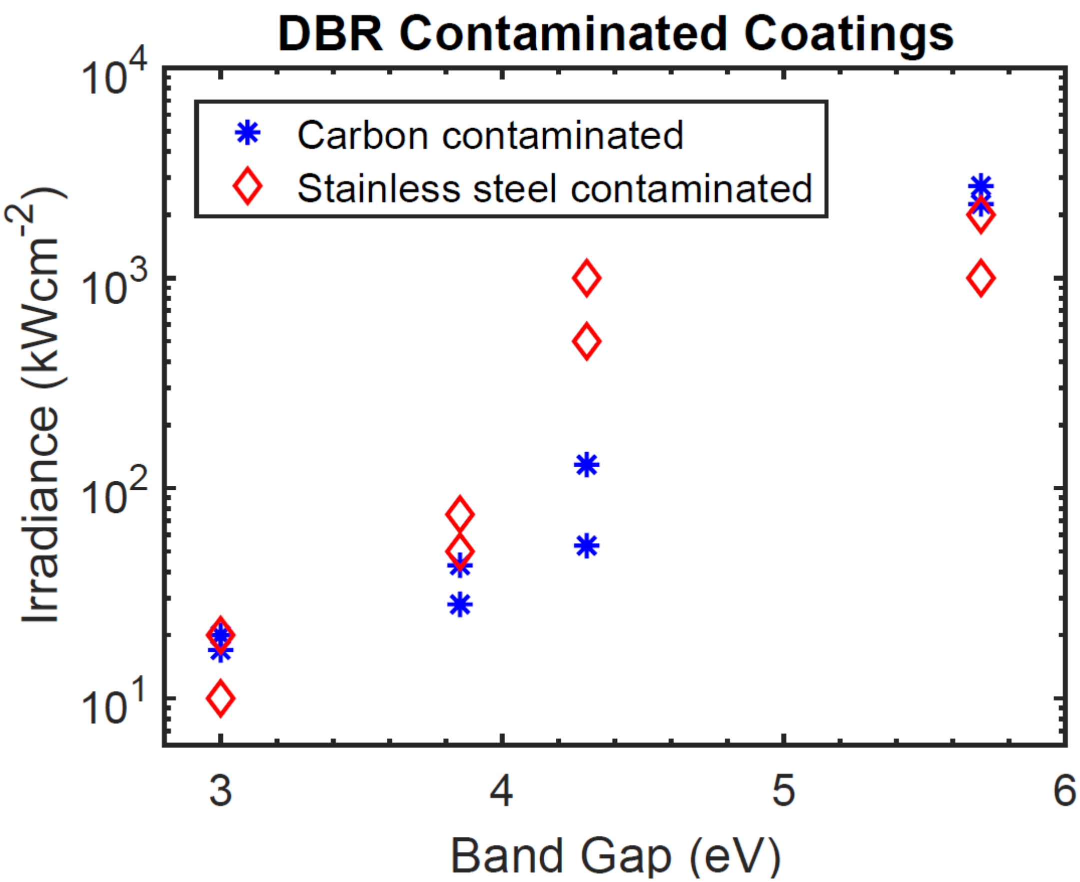


Laser Damage Thresholds vs Bandgap I



- Half-wave coating failures are not catastrophic
- Many measurements made for each material:
- Lower point represents lowest measured damage level
- Upper point represents highest survived irradiance

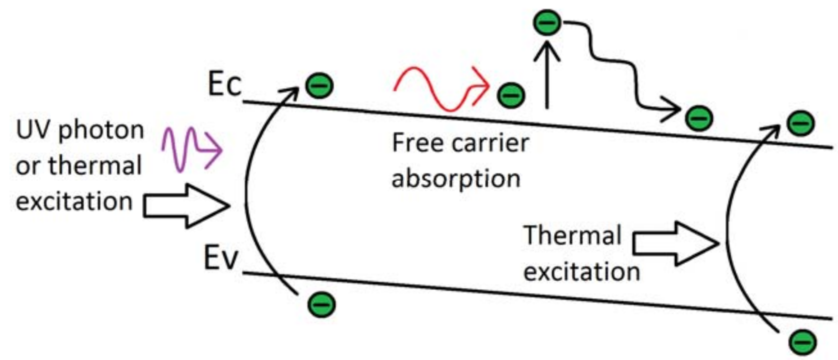
Laser Damage Thresholds vs Bandgap II



- DBR failures are catastrophic
- Many measurements made for each material:
- Lower point represents lowest measured damage level
- Upper point represents highest survived irradiance

Free Carrier Initiated Laser Breakdown

- Contamination creates very strong local absorption and heating.
- Particles may superheat beyond their sublimation temperatures
  - $T_{subl} \sim 3900K$ for carbon particles
- Two of the possible methods of exciting carriers
  - Thermal excitation $$n \propto T^{\frac{3}{2}} \exp\left(\frac{-E_G}{2k_B T}\right)$$
  - UV blackbody radiation $$B(\lambda, T) = \frac{2hc^2}{\lambda^5} \frac{1}{e^{hc/(\lambda k_B T)} - 1}$$

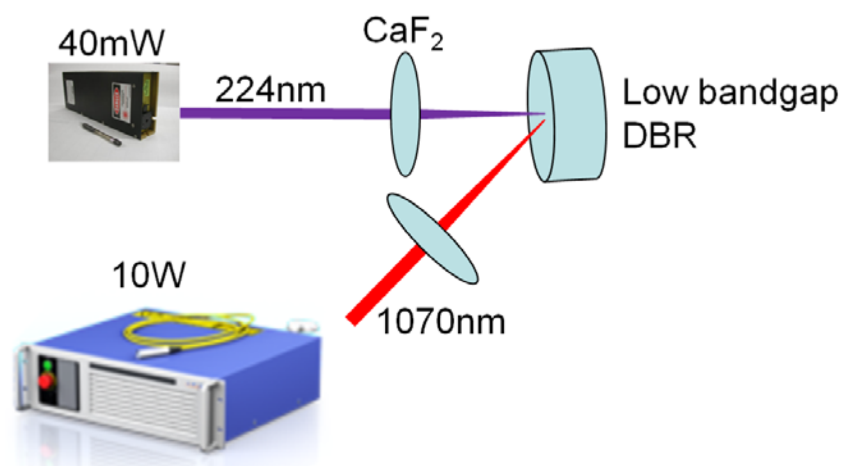


UV Seems to Play no Role in Breakdown

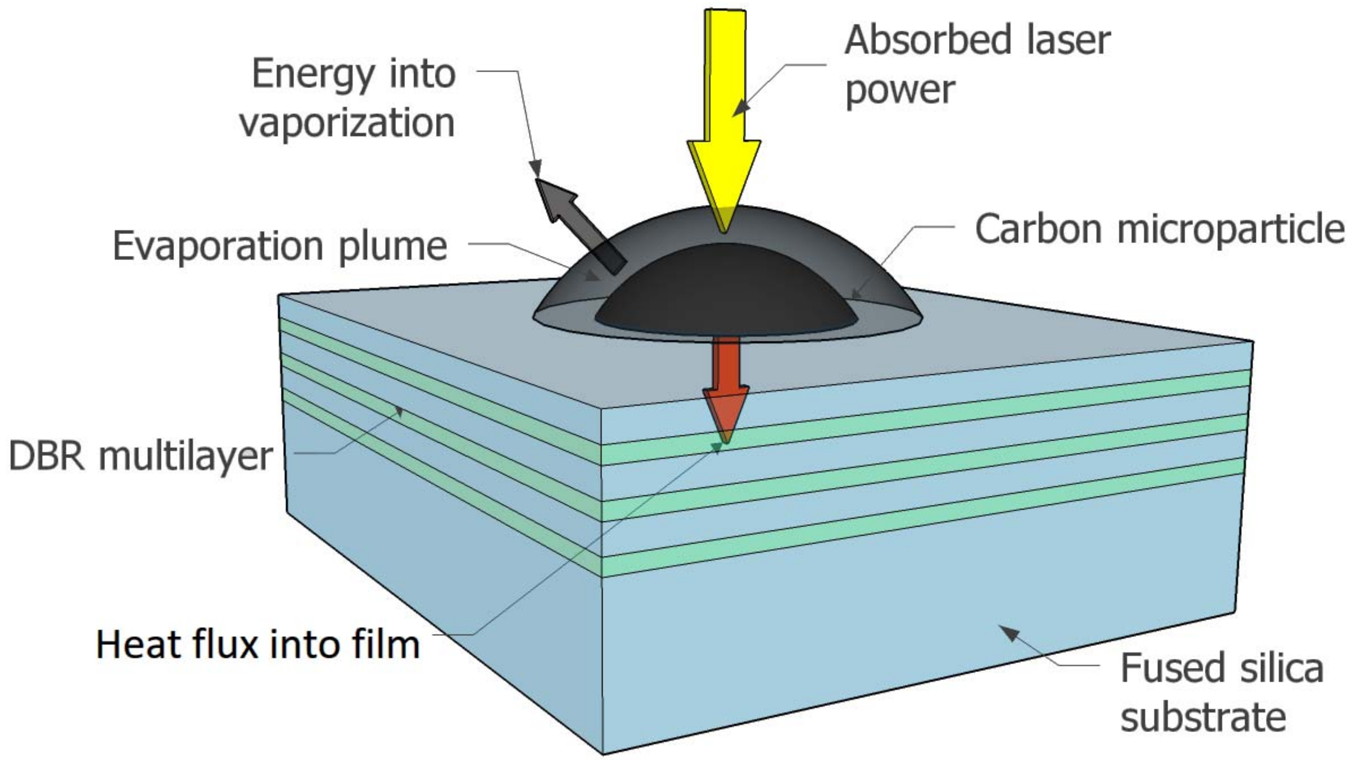
- To test UV generated free carriers as a possible breakdown mechanism, pristine low bandgap DBRs were exposed to simultaneous UV and high power IR illumination
  – UV light should generate free carriers causing the IR laser to damage the optic
- Unable to damage pristine optics despite exposure of over 17 $MWcm^{-2}$
  – Direct UV light generation is unlikely a source of free carriers

Photon Systems HeAg
224nm deep UV laser

Initial tests using 10W laser with tight focus
Follow up tests using 17kW laser



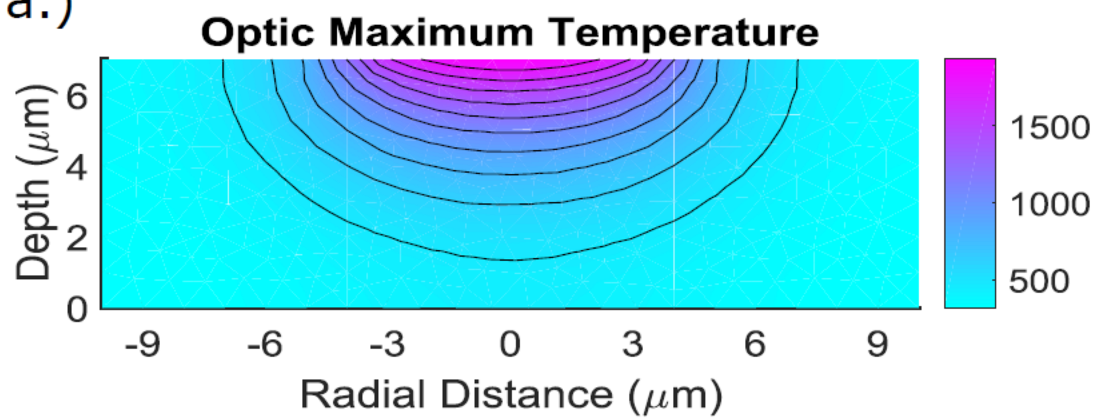
Surface-Particle-Laser Interaction Model



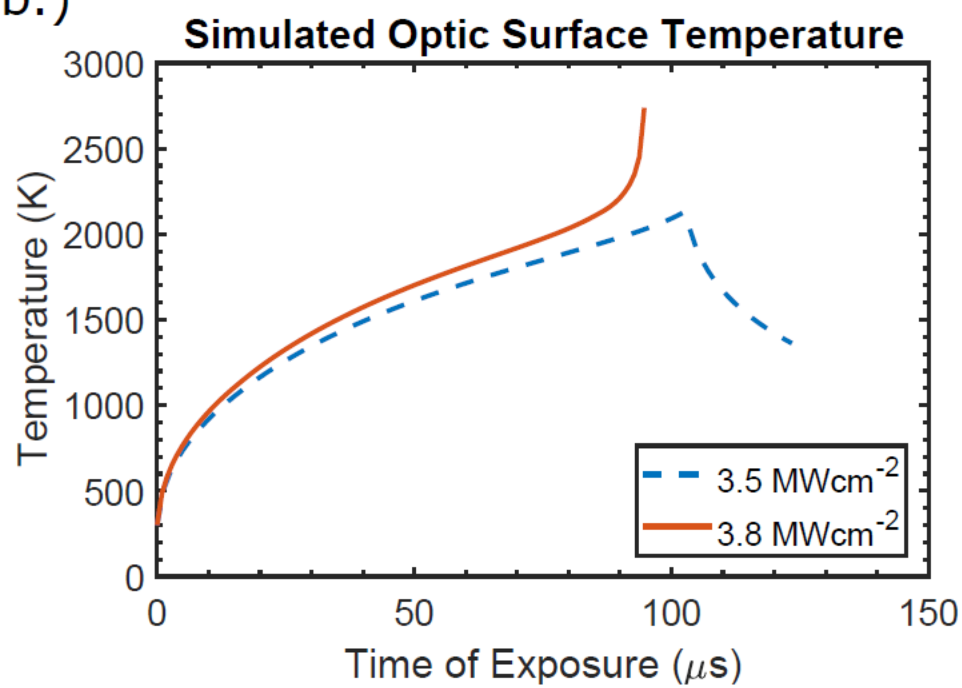
Thermal Model and Predictions

- $Ta_2O_5 – SiO_2$ high reflectivity coatings
- Model includes both heat transfer and substrate absorption
- Model quantitatively predicts onset of failure
- Failure is closely related to speed of evaporation of a particle

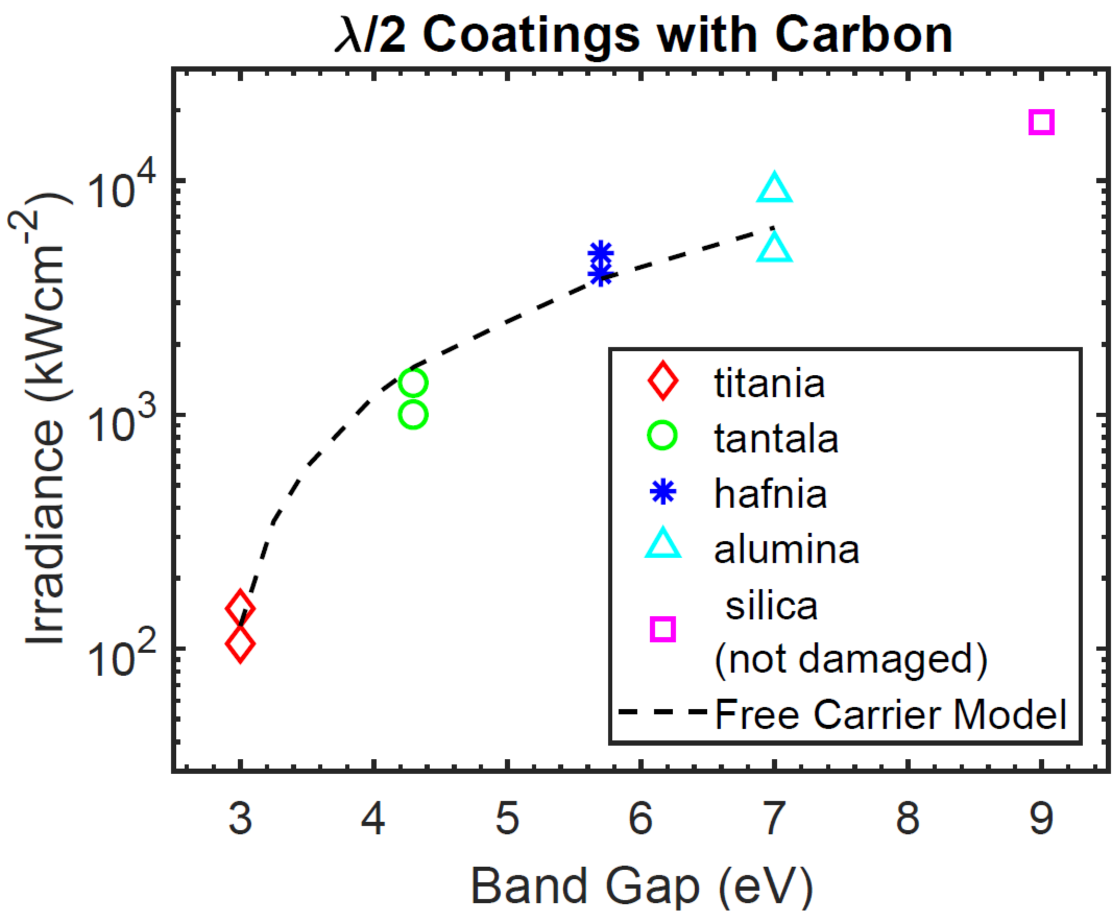
a.)



b.)



Laser Damage Thresholds vs Bandgap I



- Half-wave coating failures are not catastrophic
- Many measurements made for each material:
- Lower point represents lowest measured damage level
- Upper point represents highest survived irradiance

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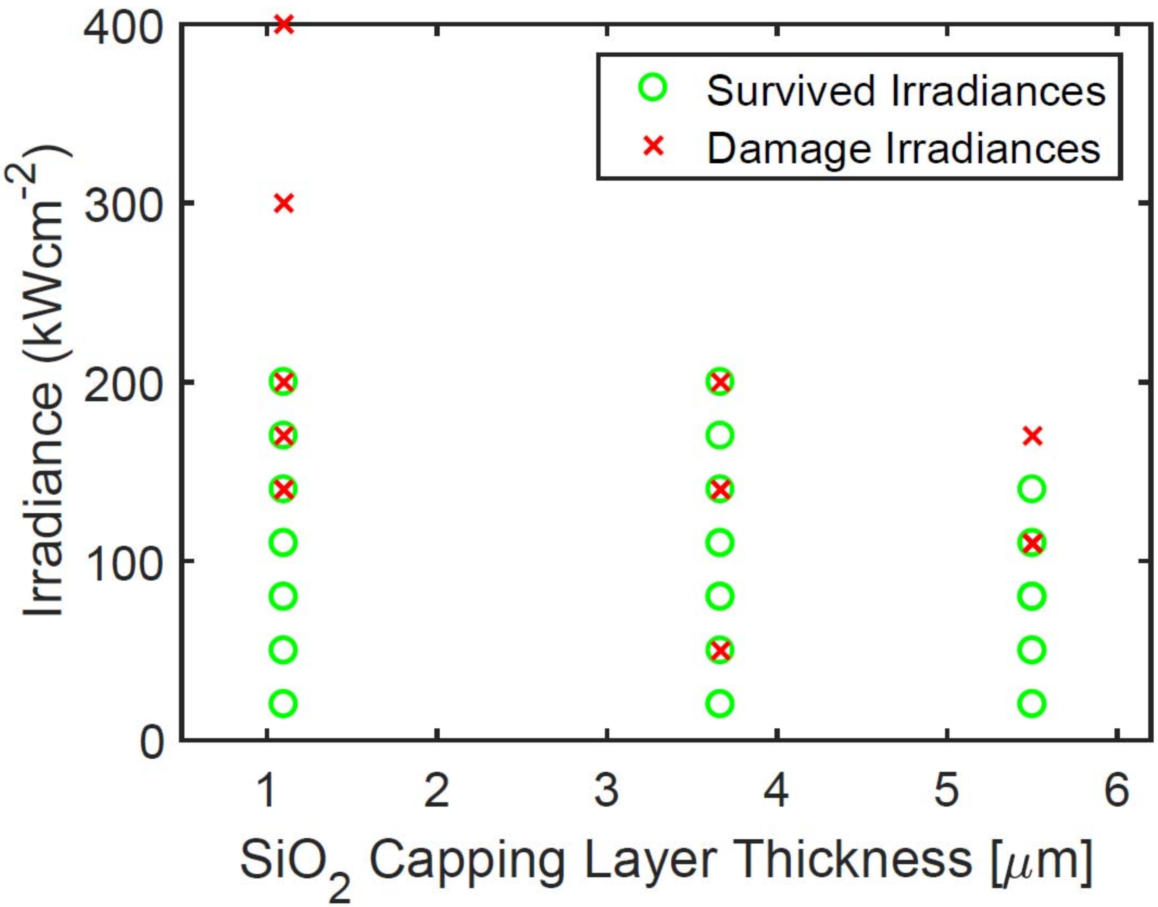
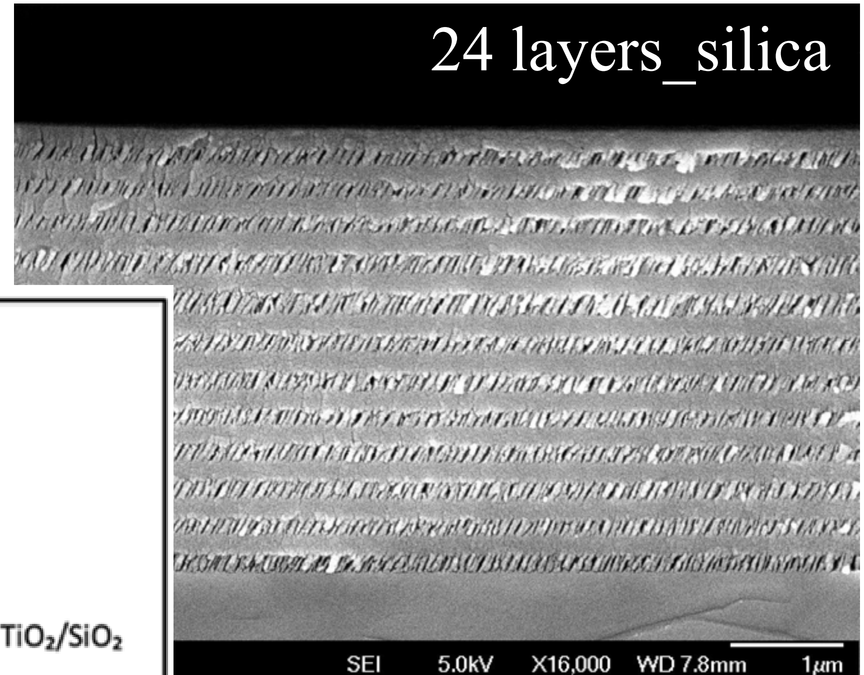
LDT Independent of Protective Layers

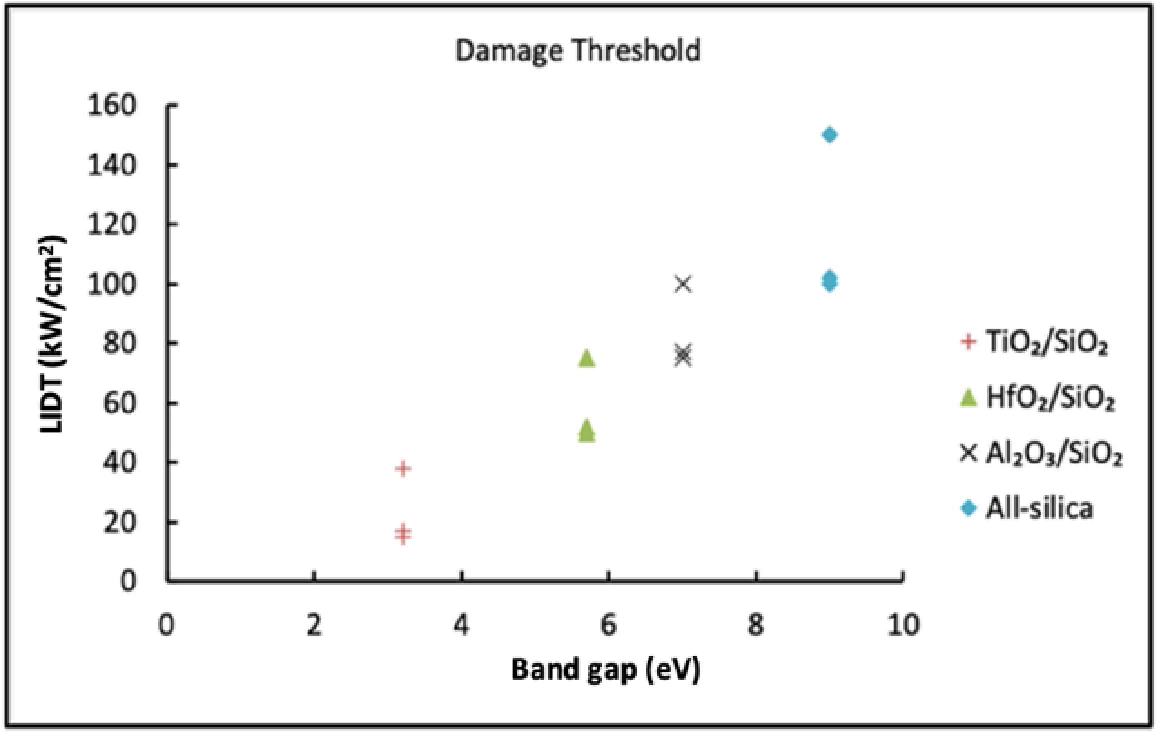


Plot of irradiances survived for Ta_2O_5 optics coated with different thickness SiO_2 cap layers. Since there is no clear dependence on protective cap thickness, this data initially seemed to indicate that direct thermal transfer was not the appropriate mechanism. **Theory indicates that 10+µm caps are necessary.**

LDT All-Silica

- Suggests that all silica (low density/high density) HR coatings should have even higher laser damage thresholds than standard HfO_2/SiO_2 pairs





- This behavior has been seen in our tests with high-angle evaporated silica

Laser Acceleration of Particles

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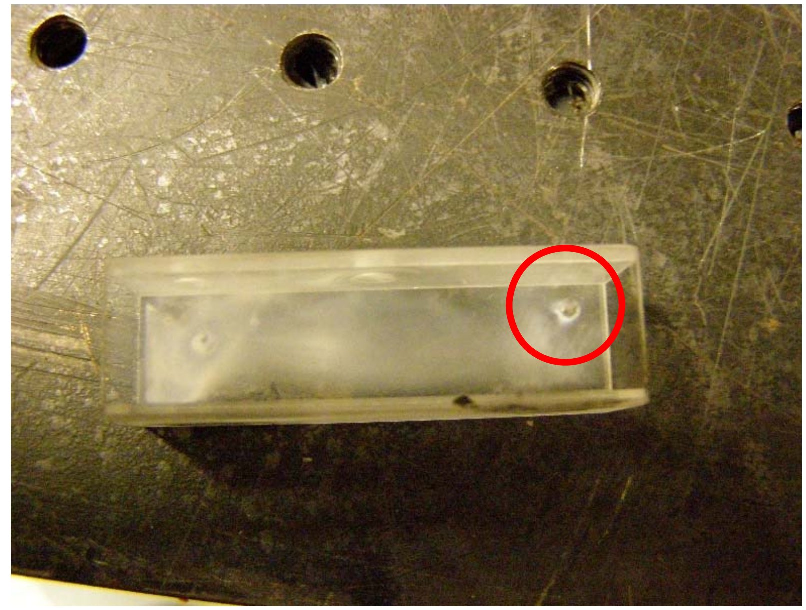
Motivation: Laser Damage Induced by Random Particles Entering Beam

- In an early experiment, we noticed a failure due to an atmospheric particle entering our laser beam
- Further testing indicated a radical change in laser damage thresholds
- This presentation describes a study of the laser acceleration process



Damaged spot on the cuvette surface. Laser intensity was 1 MW/cm^2.

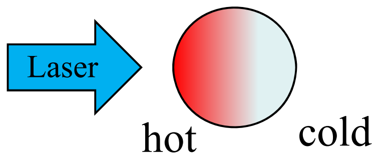
The cuvette surface survived without any damage when shot with no particle bombardment. We used 35-41 µm stainless steel particles.

Substrate Breakdown due to Laser-Induced Particle Acceleration

Substrate	Particle	Summary
Fused Silica	Stainless Steel	Drilled a hole at higher intensity (~1 MW/cm^2) Created divots at lower intensity (~250-500 kW/cm^2)
	PMMA and Fused Silica	No holes or divots even at higher intensity. Occasional tiny smudges at extremely high intensity.
Sapphire	Stainless Steel	Shattered pieces with big hole at the bombardment location even at low intensity (~100 kW/cm^2).
	Fused Silica	Shattered pieces with big hole at the bombardment location at higher intensity (~2 MW/cm^2).
	PMMA	Drilled a hole at highest intensity (~3.4 MW/cm^2).
Spinel	Stainless Steel	Shattered pieces with holes at particle bombardment locations even at low intensity (~75 kW/cm^2).

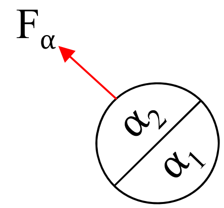
Laser - Particle Forces

Photophoretic Force



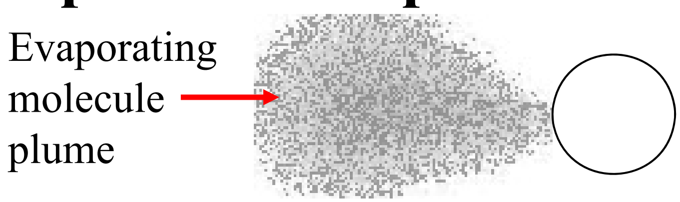
Temperature gradient due to one sided heating.

Thermal Accommodation Force



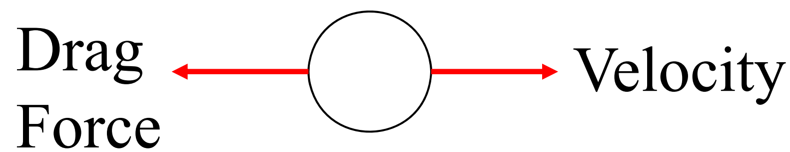
A body fixed force that arises from the diffused reflection of the gas molecules impinging on the particle.

Evaporative Propulsion Force



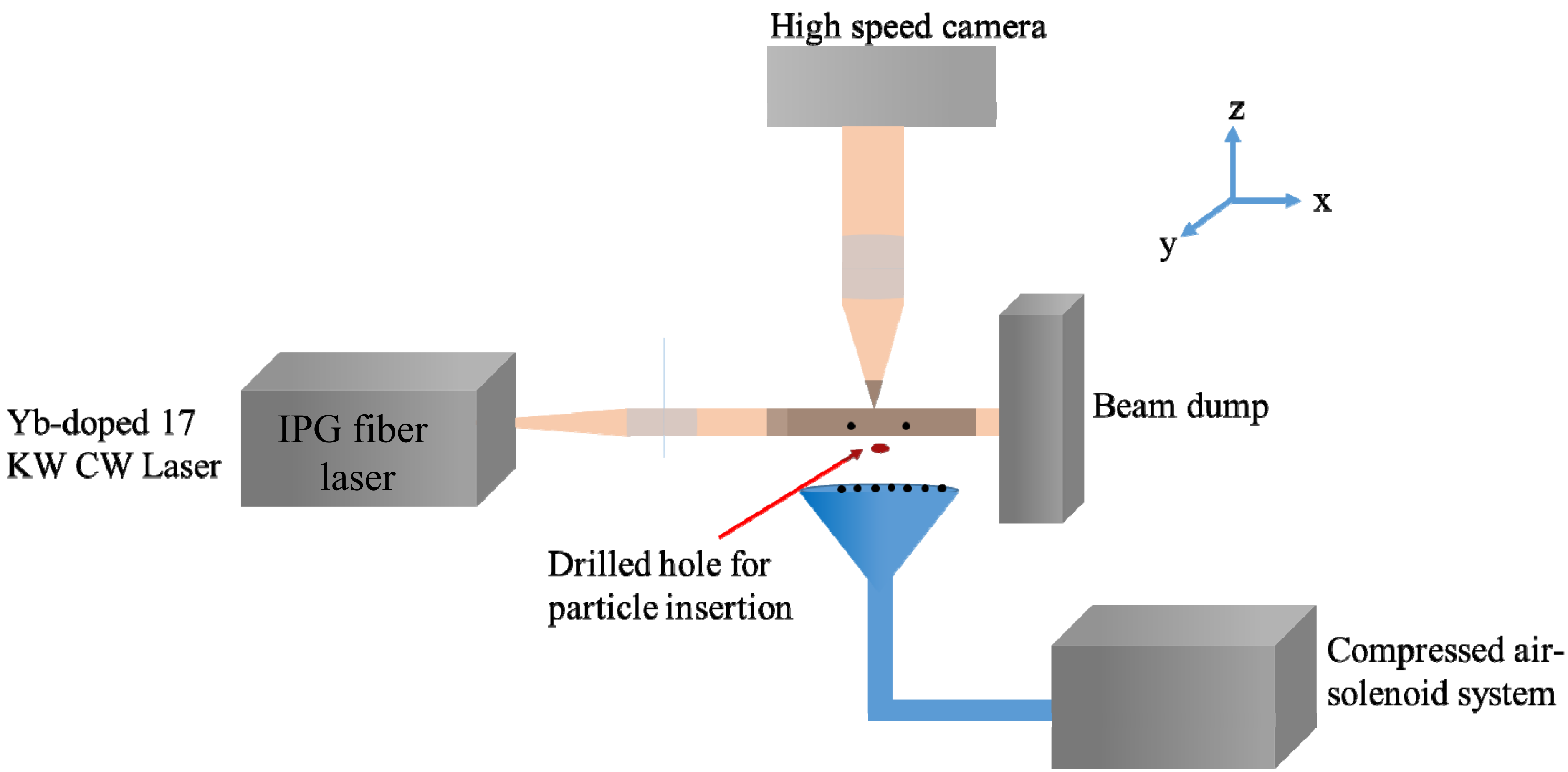
Evaporating molecules impart a 'thrust' force to the bulk particle.

Stokes' Drag Force

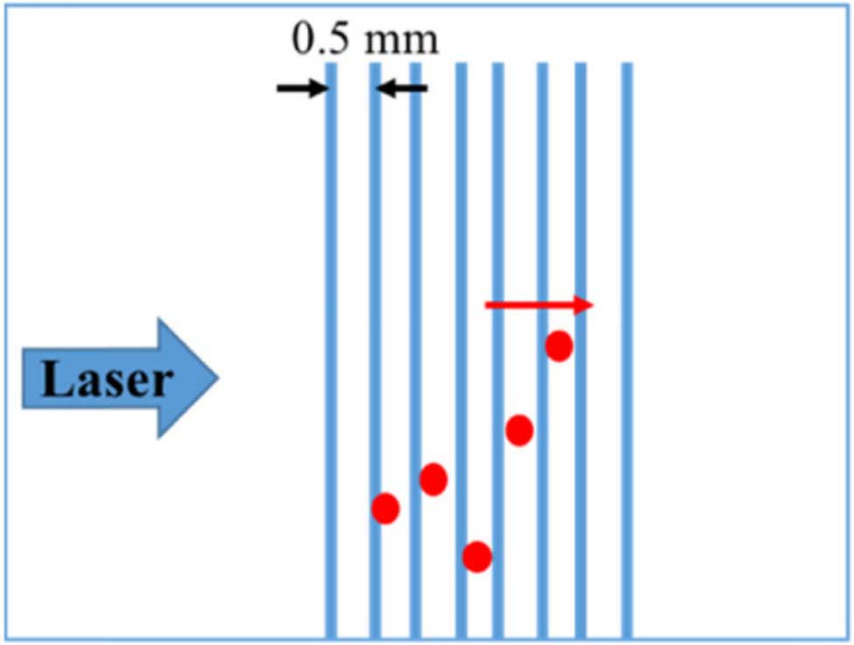


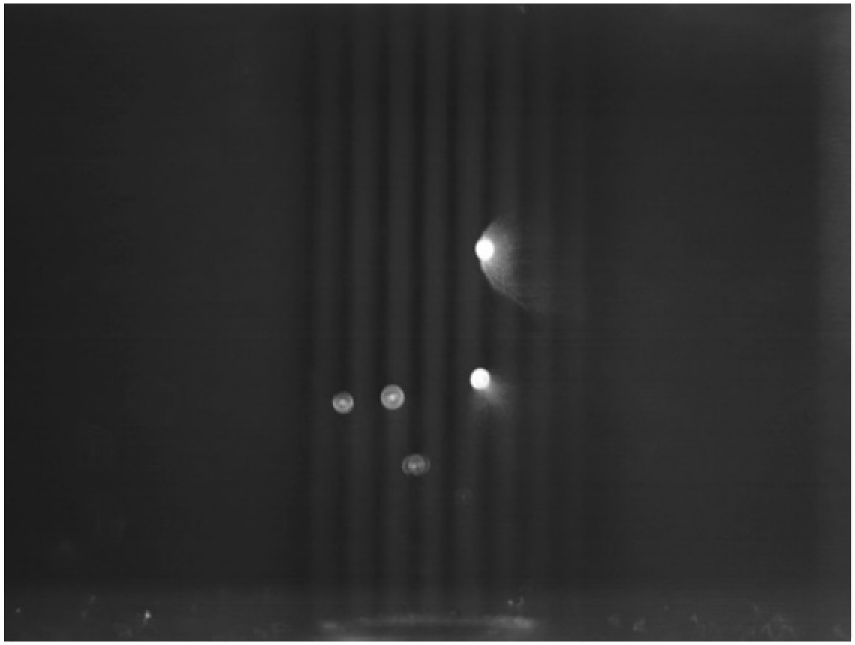
Air resistance inducing a drag force against the particle motion.

Experimental Setup - Diagram



Measurement of Particle Travel

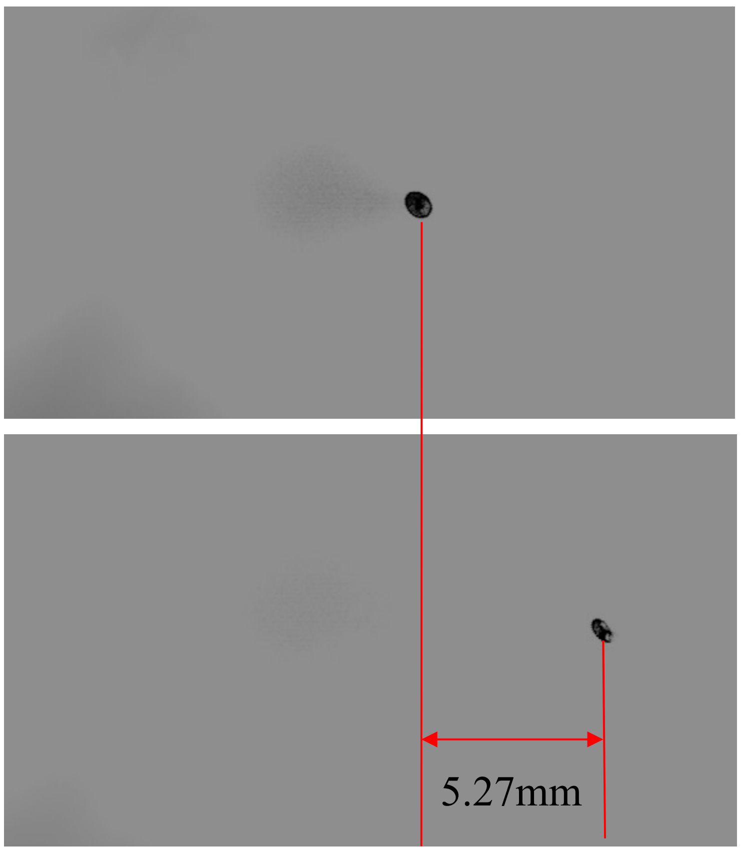


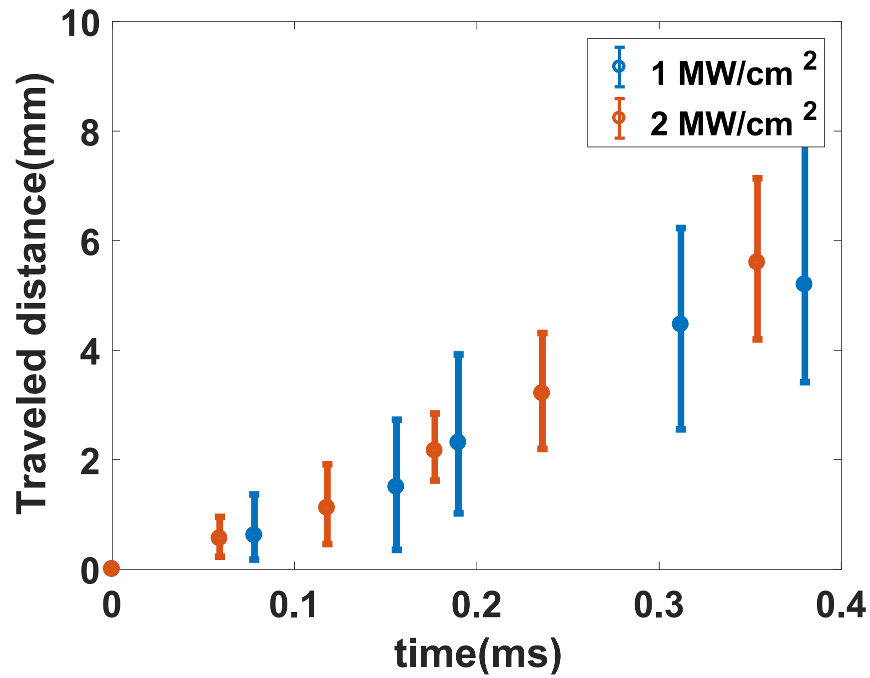


- Measurement ruler etched in one wall of cuvette
- The distance between the vertical lines is 0.5 mm
- The laser propagates from left to right
- Particles enter the cuvette through the hole drilled in the bottom of the cuvette.

Stainless Steel Particle Motion

Successive frames (~0.1ms)





Travelled distance of 35-41 μm particles vs. time for 1 and 2 MW/cm^2 intensities. Each data point represents average from approximately 20-25 particle trajectories.

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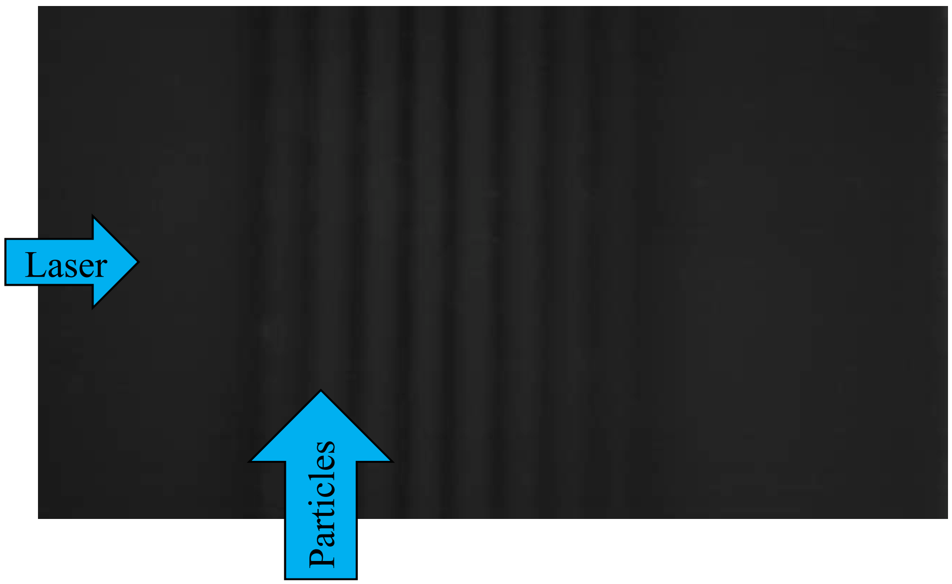
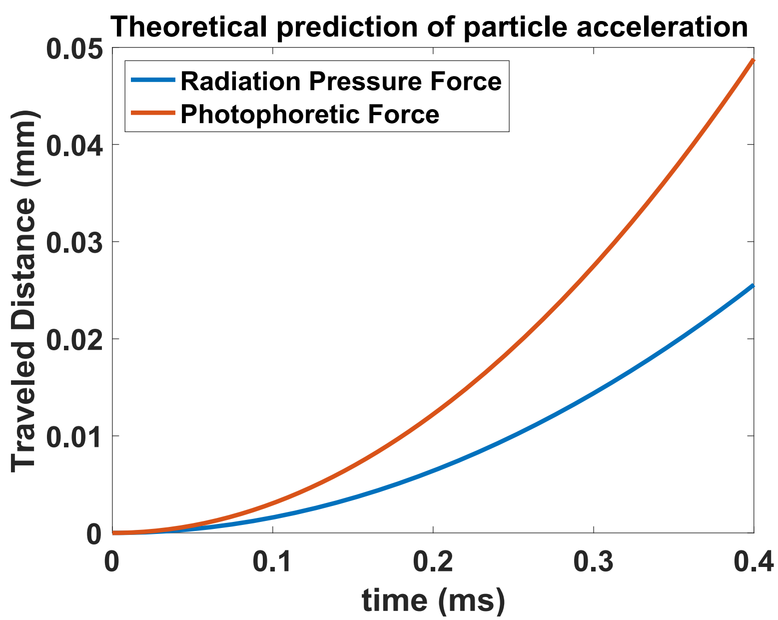
Video of Typical Laser-Particle Interaction

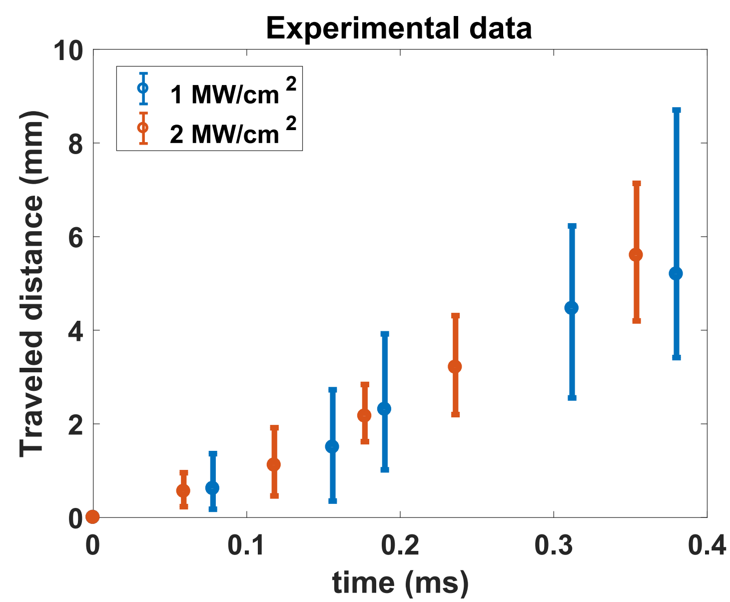


17kW IPG Photonics
Yb-doped fiber laser

Camera video at 0.1ms
frame rate

Comparison of Reality with Radiation Pressure and Photophoretic Forces





- Magnitude off by factor of ~100
- Shape differs as well
- Experimental measurements show the average, minimum, and maximum values from approximately 20-25 particle trajectories per data point

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Evaporative Equations

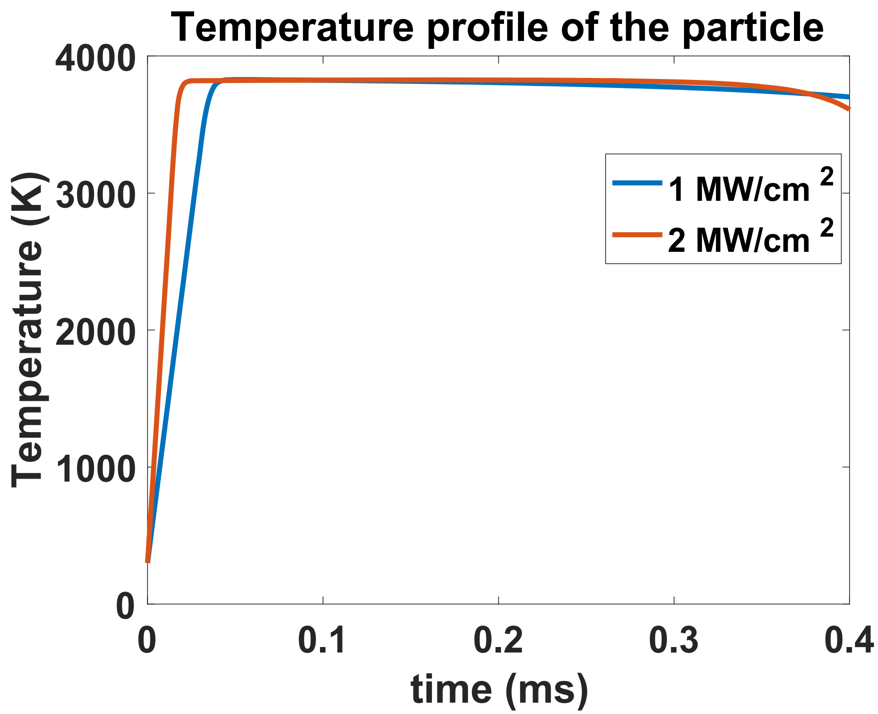
$$\frac{dM}{dt} = \frac{4\pi a P_p D W_v 10^{-6}}{RT} \ln\left(1 - \frac{P_p - P_{atm}}{P_p + P_{atm}}\right)^{-1}$$

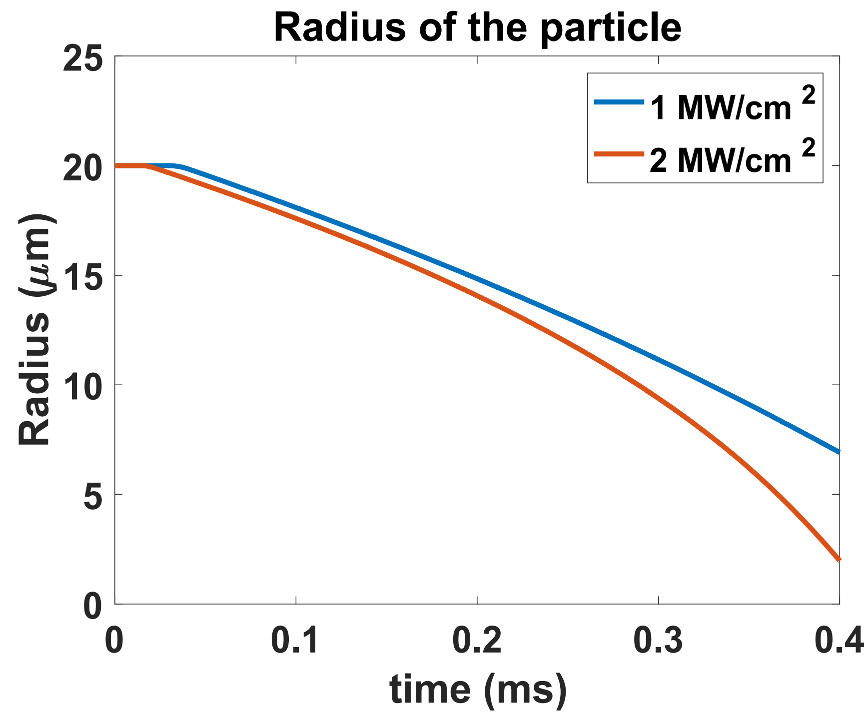
Rate of change of mass of particle

$$P_p = P^* \exp\left[\frac{H_v(T - T_b)}{RTT_b}\right]$$

Exponential change in vapor pressure with temperature

Particle Mass Loss Model





- Very rapid heating of particle from laser beam
- Near the particle sublimation/boiling point, evaporative cooling locks the temperature
- Particle mass decreases steadily – fractional rate increasing due to S/V ratio

Temperature Gradient Across Particle

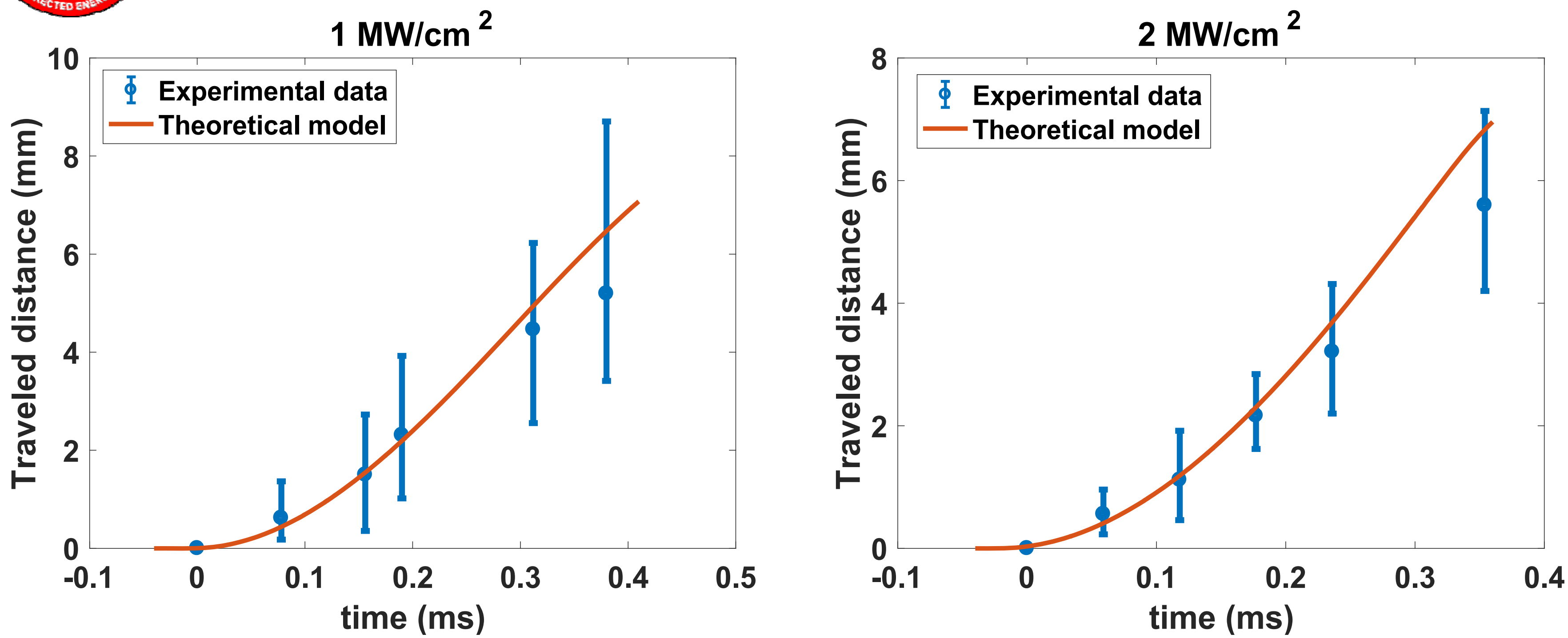
- Evaporation rate will vary across particle (front to back) due to differential heating
- Standard conduction/convection/radiation predicts velocities far too high to match data
- Heat transfer within particle appears to be dominated by radiative diffusion
  - normally dominates in the photospheres of stars

$$\mathrm{F}^{R} = -K^{R}\frac{dT}{dr} = -\frac{16\sigma T^{3}}{3\kappa}\frac{dT}{da}$$

κ - opacity
a - particle radius
σ - Stefan-Boltzmann constant

Heat transfer rate for radiative diffusion

Model vs Experiment



- Data matches well at both intensities tested – magnitude and curve shape
- 40µm particles simulated (actually a distribution ~ 35-41µm)
- Drag calculated using particle size measured by visual size of particle plume
- Laser beam is partially attenuated by evaporated plume
- Typical camera frame offset ~ 0.04ms

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Conclusions

- Contamination-induced optical breakdown is highly bandgap dependent
- Protective cap layers of higher bandgap must be thick
- UV illumination appears to have no effect on contamination-induced laser damage
- **Particle-induced substrate absorption + heat transfer across particle-optic interface creates free-carriers that cause failure**
- Particle acceleration and evaporation observed in high power laser illumination
  - Max velocities several tens of meters per second
  - Optic damage **strongly** initiated by accelerated particles
  - Acceleration mechanism is evaporative acceleration
  - Heat transfer within particle dominated by photon diffusion – a process normally dominant in stars

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