

# NIF



## Surface interactions on glass optics during fabrication, post processing & laser operation

***Functional Glasses: Properties and Applications for Energy & Information  
Siracusa, Sicily, Italy***

***Friday, January 11, 2013  
Tayyab Suratwala***

**LLNL-PRES-608293**

**Lawrence Livermore National Laboratory • National Ignition Facility & Photon Science**

This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344

# High-peak-power and high-average-power lasers demand laser damage resistant optics

## Fusion Energy



- National Ignition Facility (NIF)
- Mercury Laser
- Laser Inertial Fusion Energy (LIFE)
- Laser MegaJoule (LMJ)
- Laboratory Laser Energetics (LLE)
- Etc....

## Directed Energy



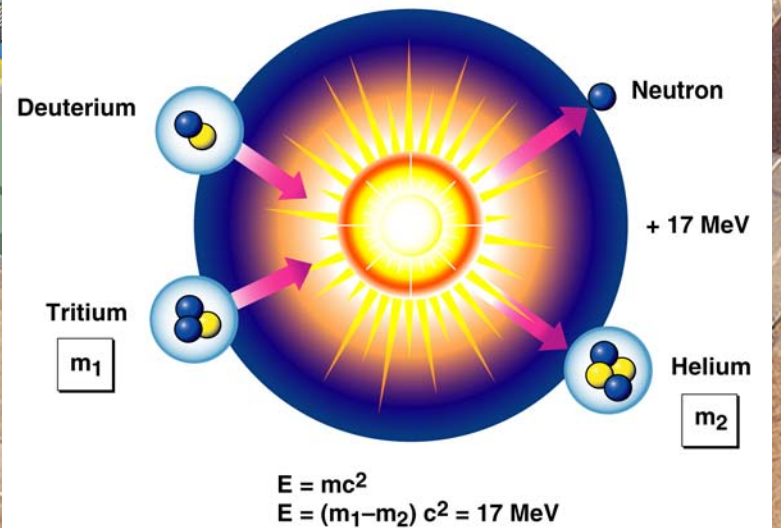
- High-Average-Power Laser (HAPL)
- Diode-pumped, solid-state heat-capacity laser (SSHCL)
- Tailored-aperture ceramic laser (TACL)

## Commercial Lasers



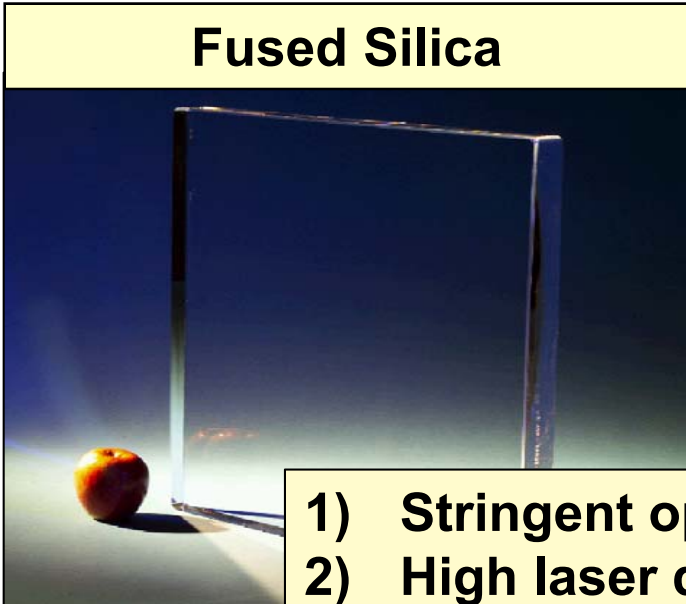
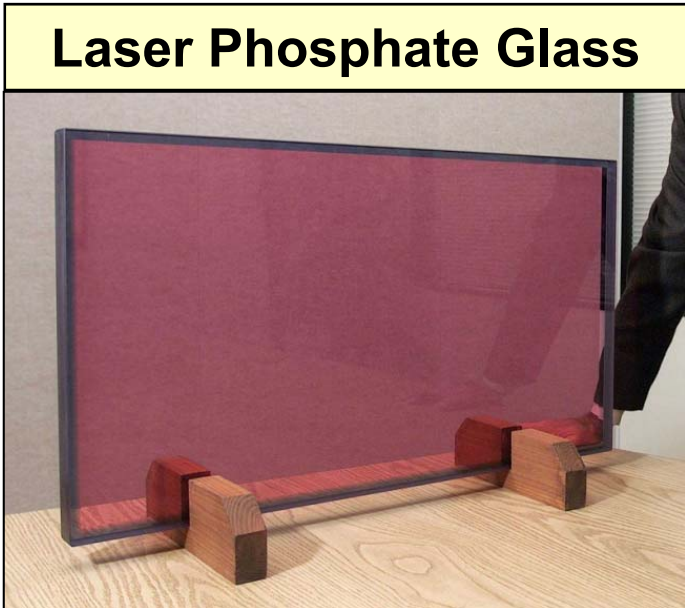
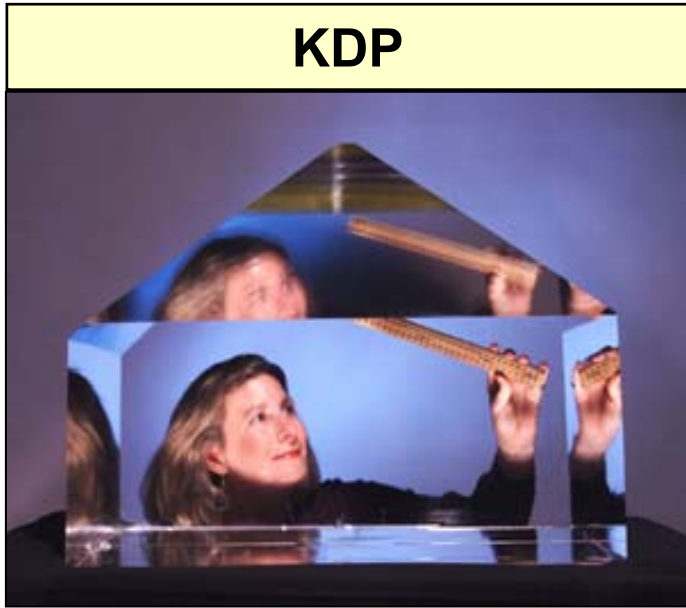
NIF concentrates all the energy in a football stadium-sized facility into a  $\text{mm}^3$

Matter Temperature  $> 10^8 \text{ K}$   
Radiation Temperature  $> 3.5 \times 10^6 \text{ K}$   
Densities  $> 10^3 \text{ g/cm}^3$   
Pressures  $> 10^{11} \text{ atm}$



NIF-0506-12065  
19EIM/dj

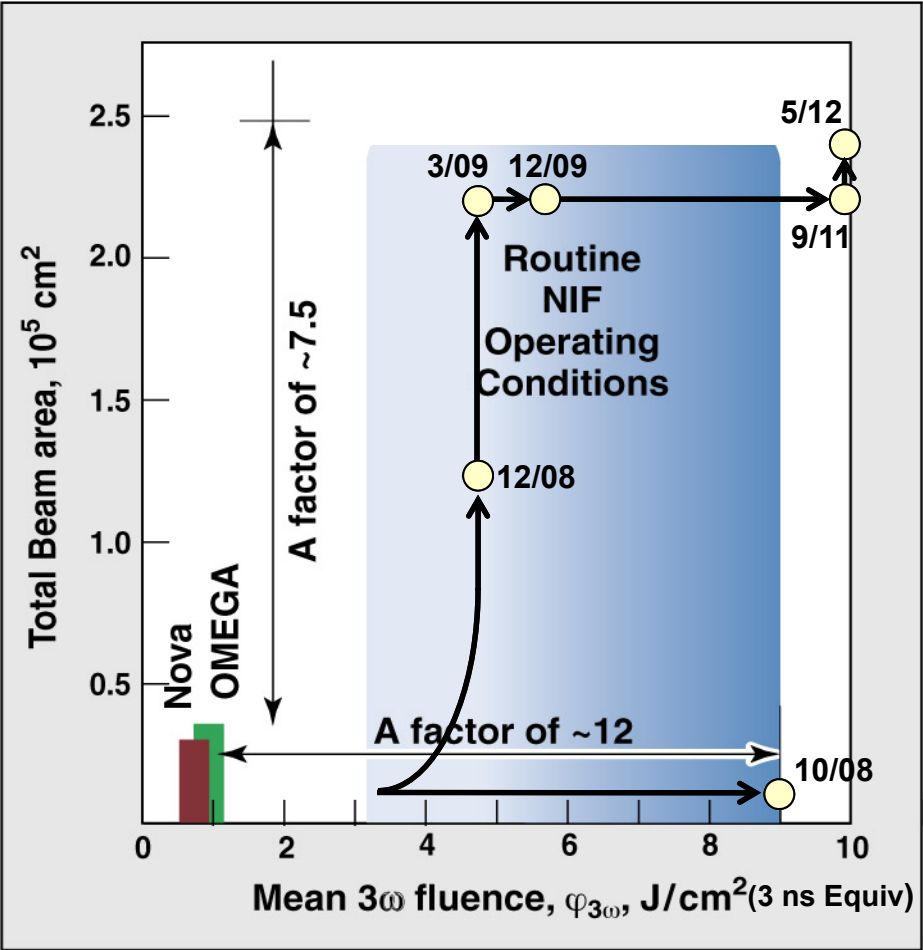
# Materials for NIF large optics are limited only to four different glasses or single crystals



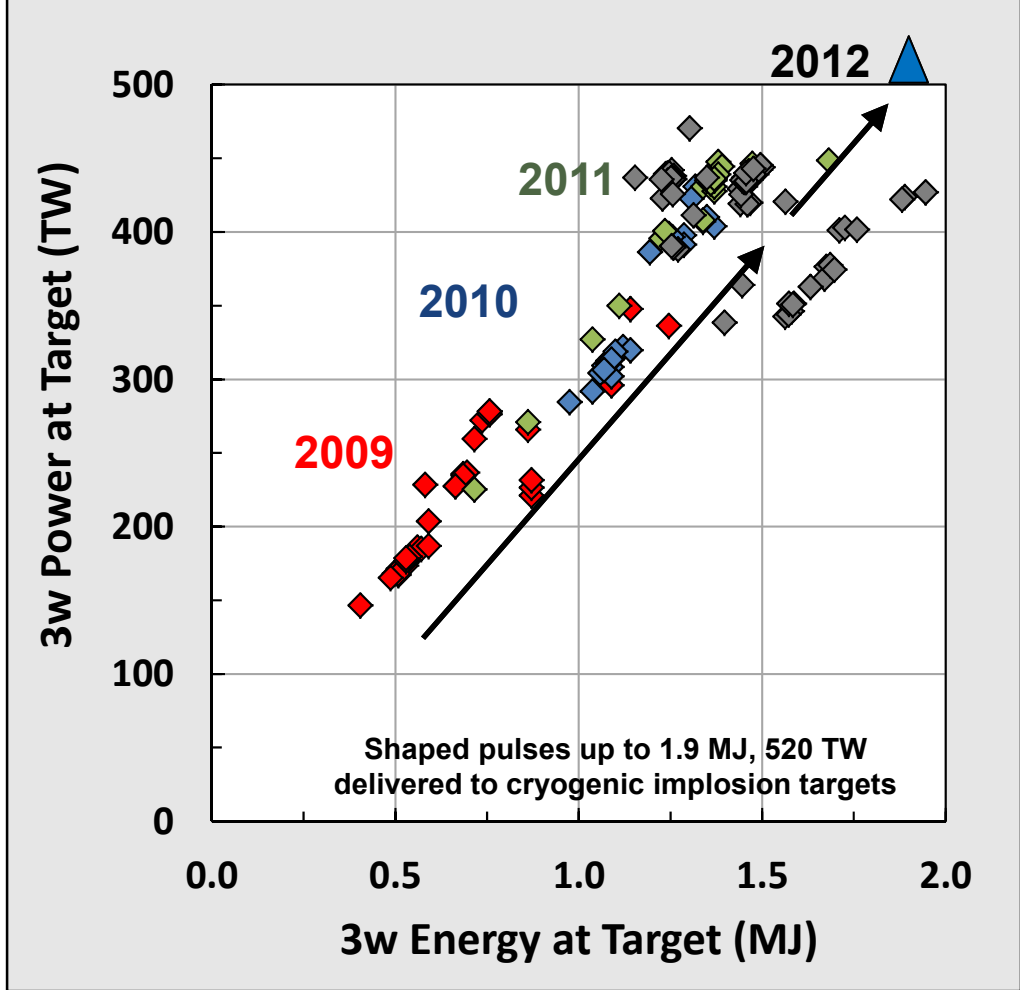
- 1) Stringent optical requirements
- 2) High laser damage resistance
- 3) Manufacturability to 0.5 m size scale

# NIF's operational fluence & power have increased dramatically, strongly supported by more damage resistant optics

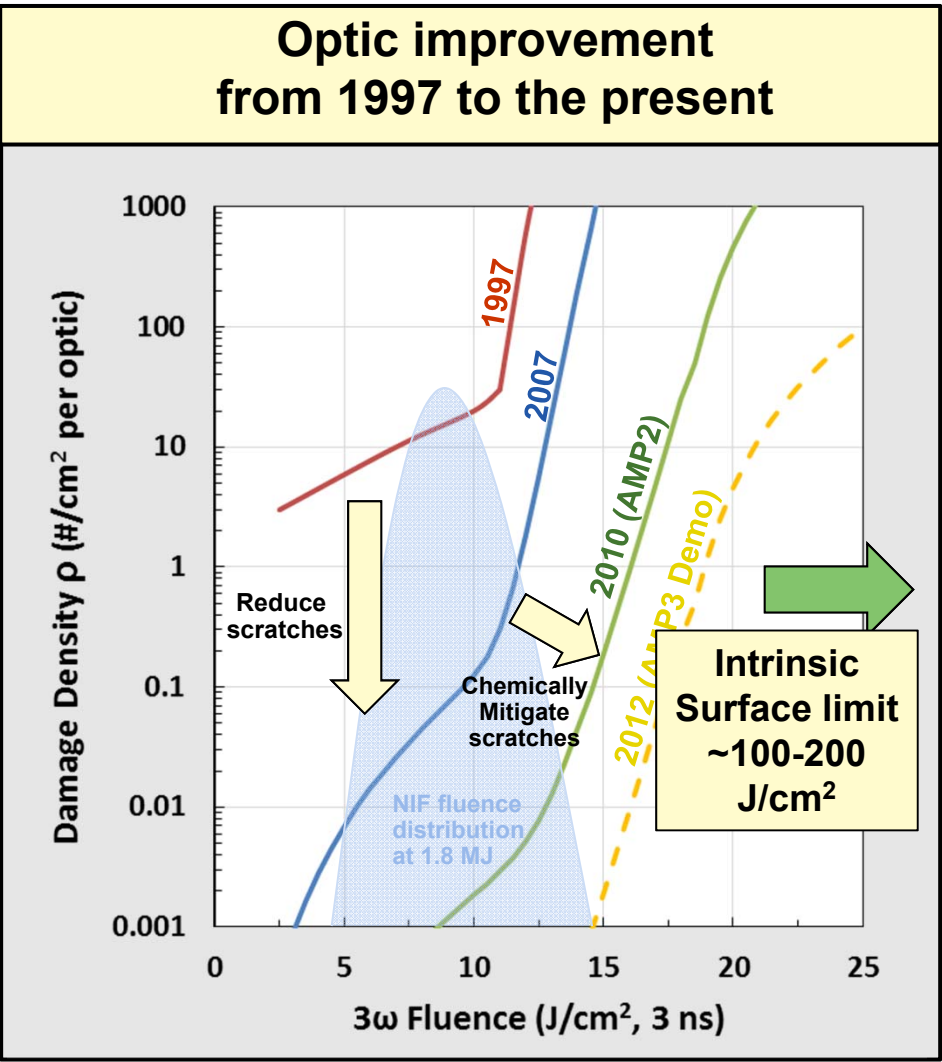
NIF can operate ~10x higher in fluence than previous lasers



NIF's  $3\omega$  power has been increasing at a rate of 100TW/year



# Greater understanding glass surface interactions has led to greatly improved high fluence glass optics



- $\rho(\varphi)$  is the expected density of initiated sites as a function of  $3\omega$  illuminating fluence
- $\rho(\varphi)$  is the metric used to describe the quality of the surface finish
  - Better optics have a lower  $\rho(\varphi)$
- Greater than 4 orders of magnitude improvement from 1997 to present
  - Fracture reduction in conventional polishing
  - Chemical treatment to make residual fractures benign

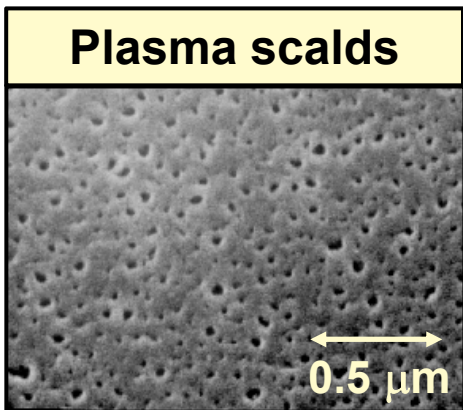
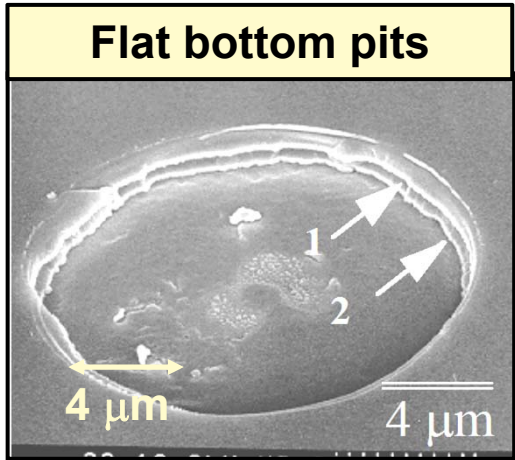
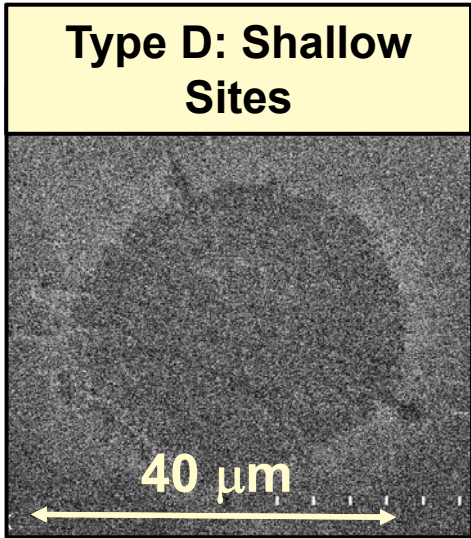
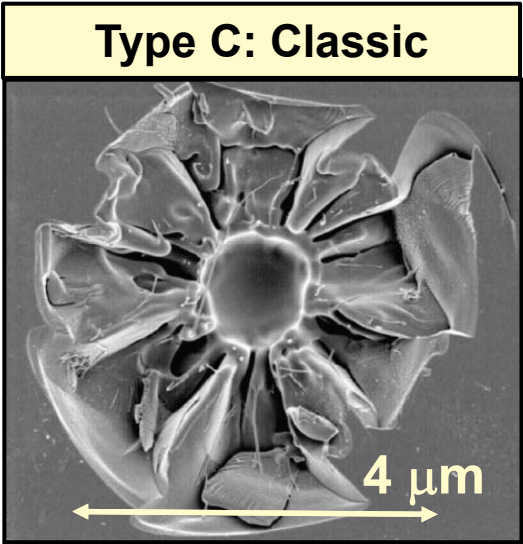
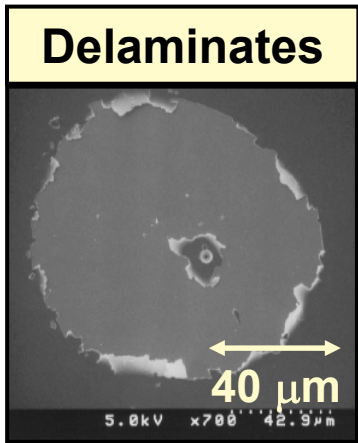
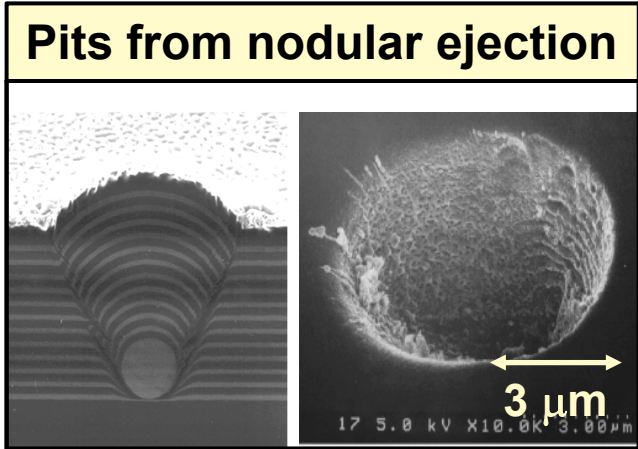
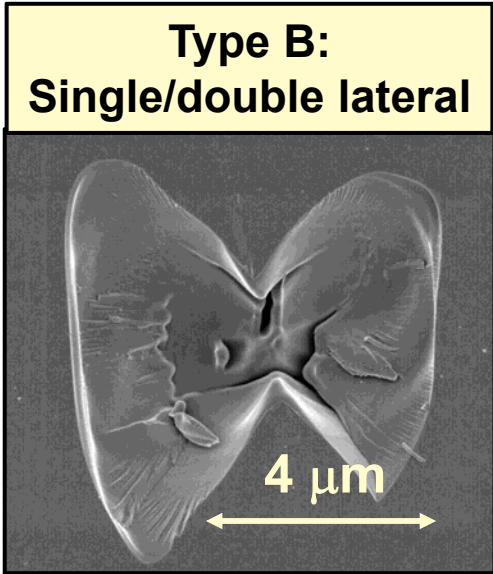
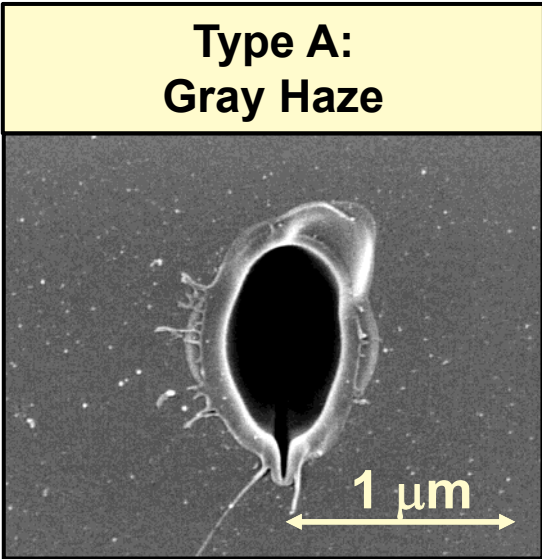
**Even today, there is much opportunity to increase surface damage threshold of glass surfaces**

# Our S&T has focused on understanding surface interactions on glass surfaces during fabrication, post processing and laser operation

**Current Efforts**

1. Optical Fabrication	2. Post Processing & Coatings	3. Laser Operation
<ul style="list-style-type: none"> <li>• Sub-surface damage management</li> <li>• Forensics of surface fractures</li> <li>• Fundamentals of material removal</li> <li>• Technology of full aperture &amp; small tool optical finishing</li> <li>• Low cost, precursor-free finishing techniques</li> </ul>	<ul style="list-style-type: none"> <li>• Development of chemical/thermal-based flaw/damage mitigation</li> <li>• Development of laser-based flaw/damage mitigation</li> <li>• Laser interference gratings development</li> </ul>	<ul style="list-style-type: none"> <li>• Mechanism of initiation &amp; growth (precursors &amp; modulation)</li> <li>• Precursor isolation &amp; identification</li> <li>• Quantitative understanding initiation &amp; growth behavior</li> <li>• Understanding solarization effects</li> <li>• Understanding modulation effects</li> </ul>

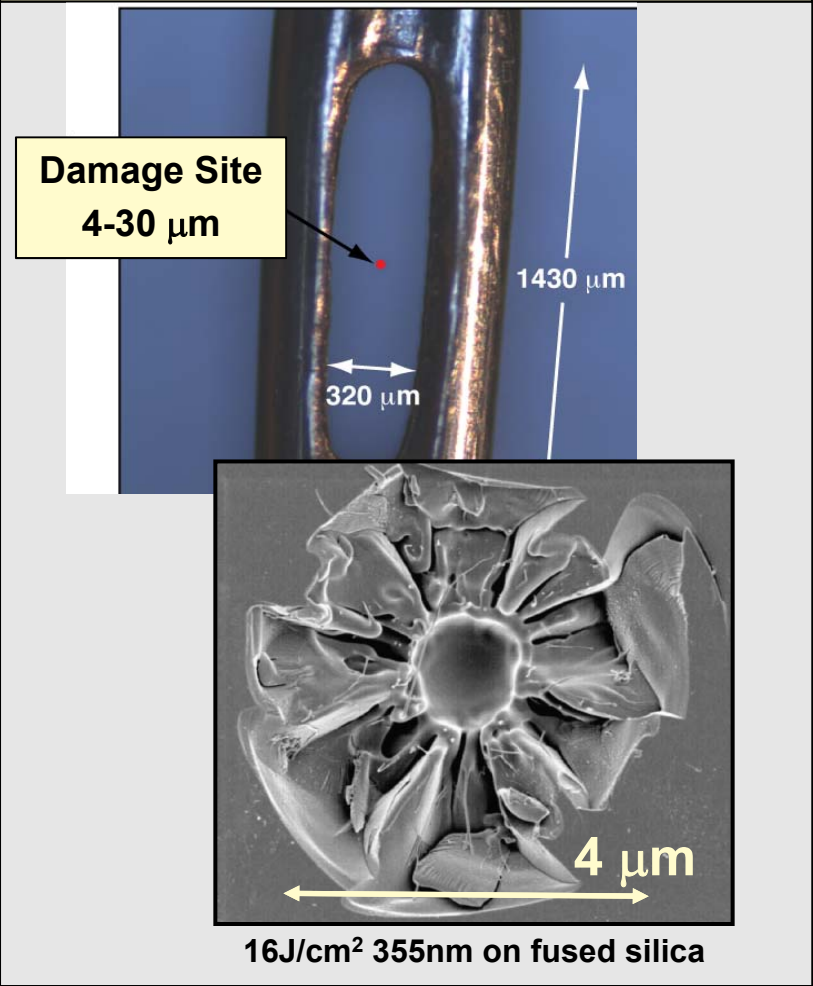
# Various types of microscopic laser damage are observed on high fluence glass optics





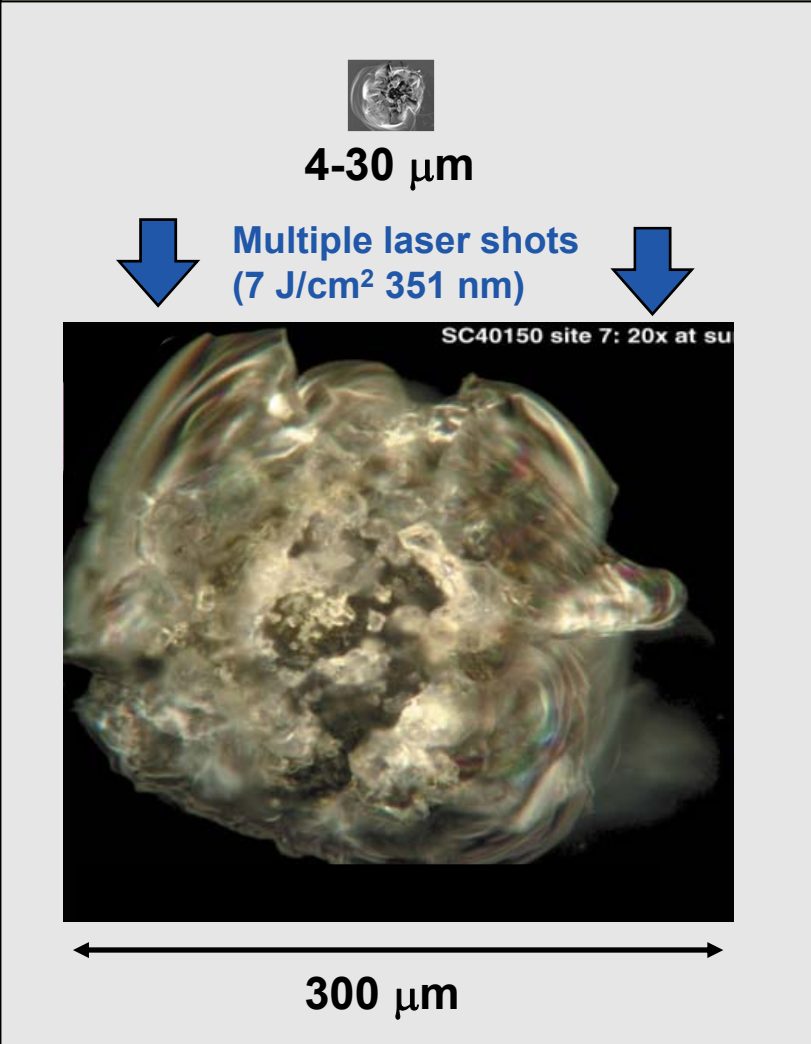
# Many of these damage sites can grow larger with subsequent laser shots

## Surface **initiation** of small damage sites



Damage initiates from sub-band gap absorbing precursors

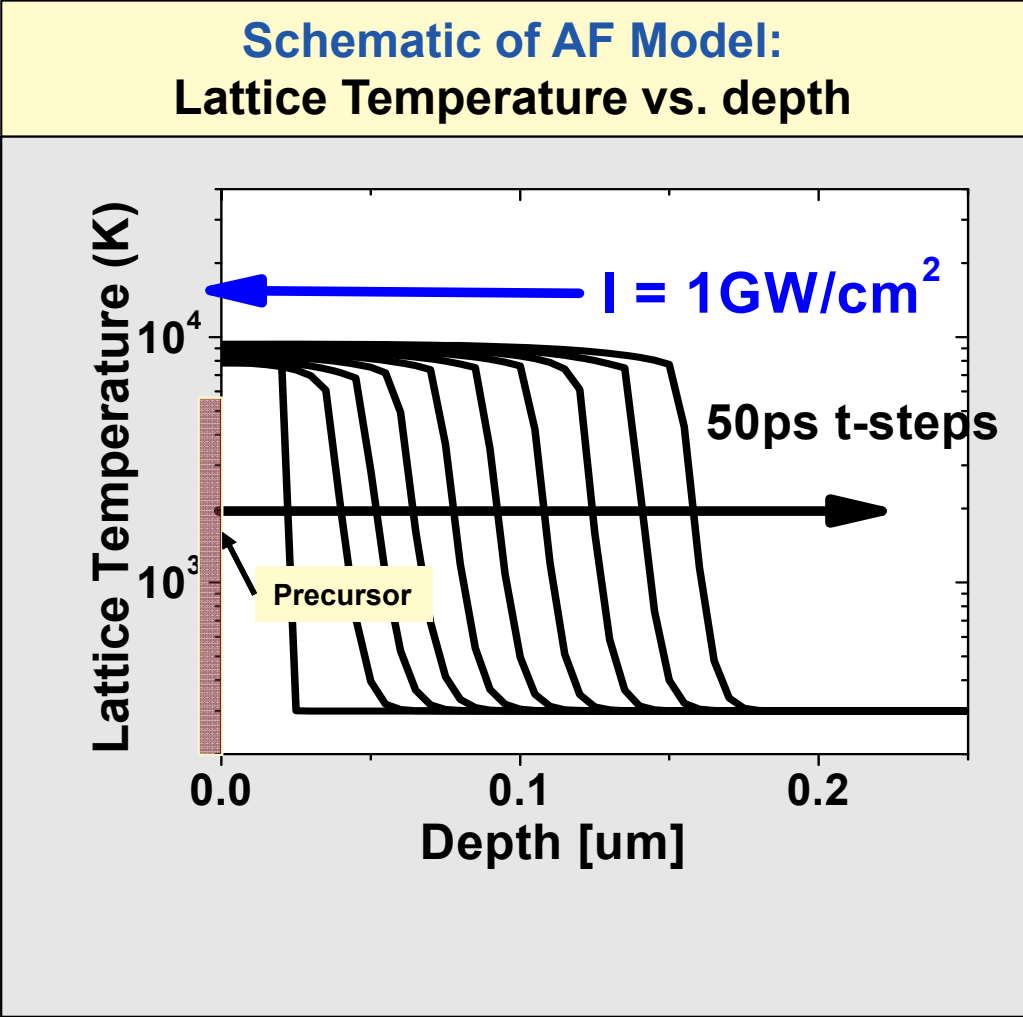
## **Growth** occurs at low fluence



Growth ultimately limits optic's lifetime

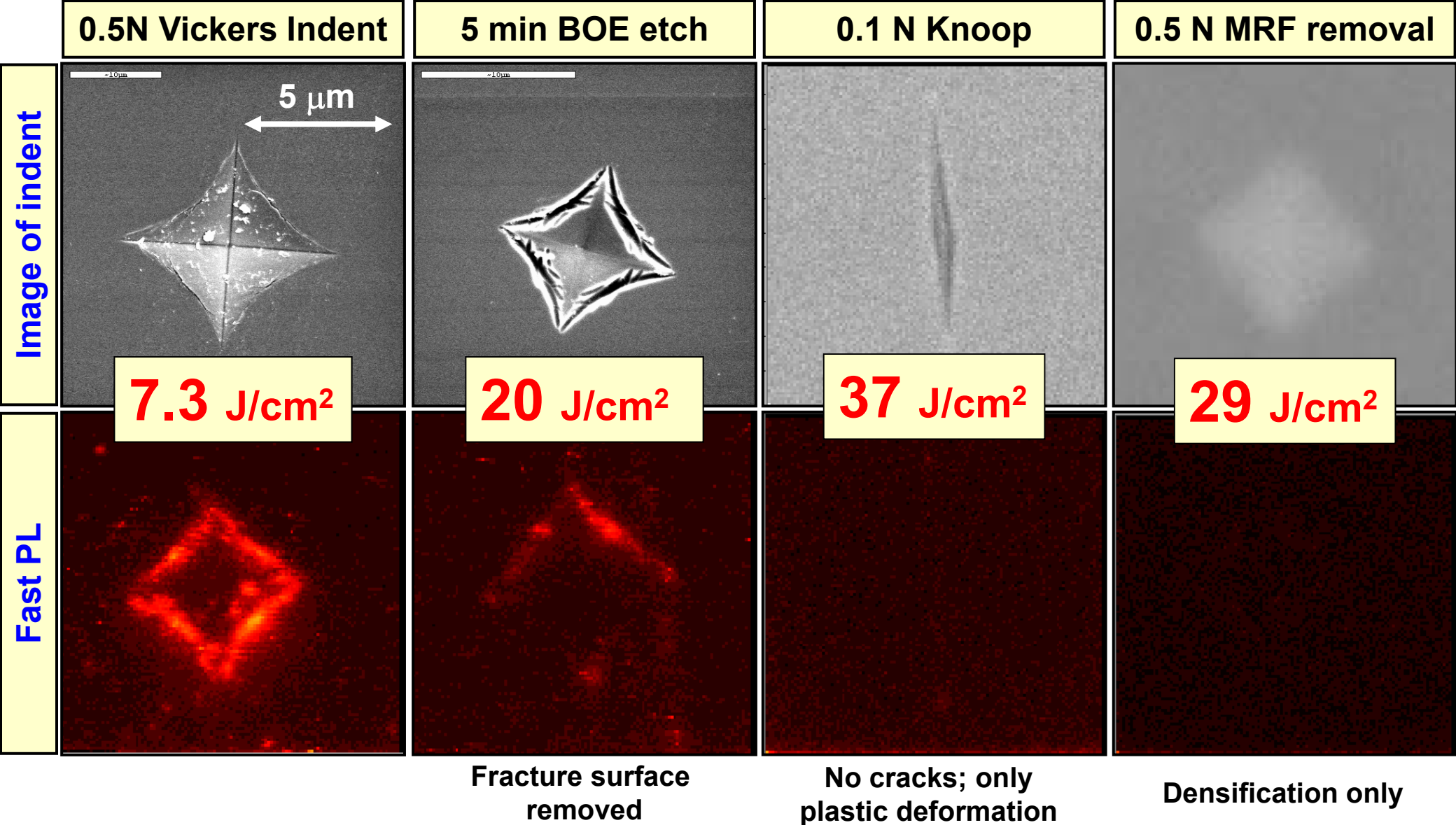
Laser damage mechanism:

T-activated absorption results in the formation of a laser-driven solid-state absorption front (AF)

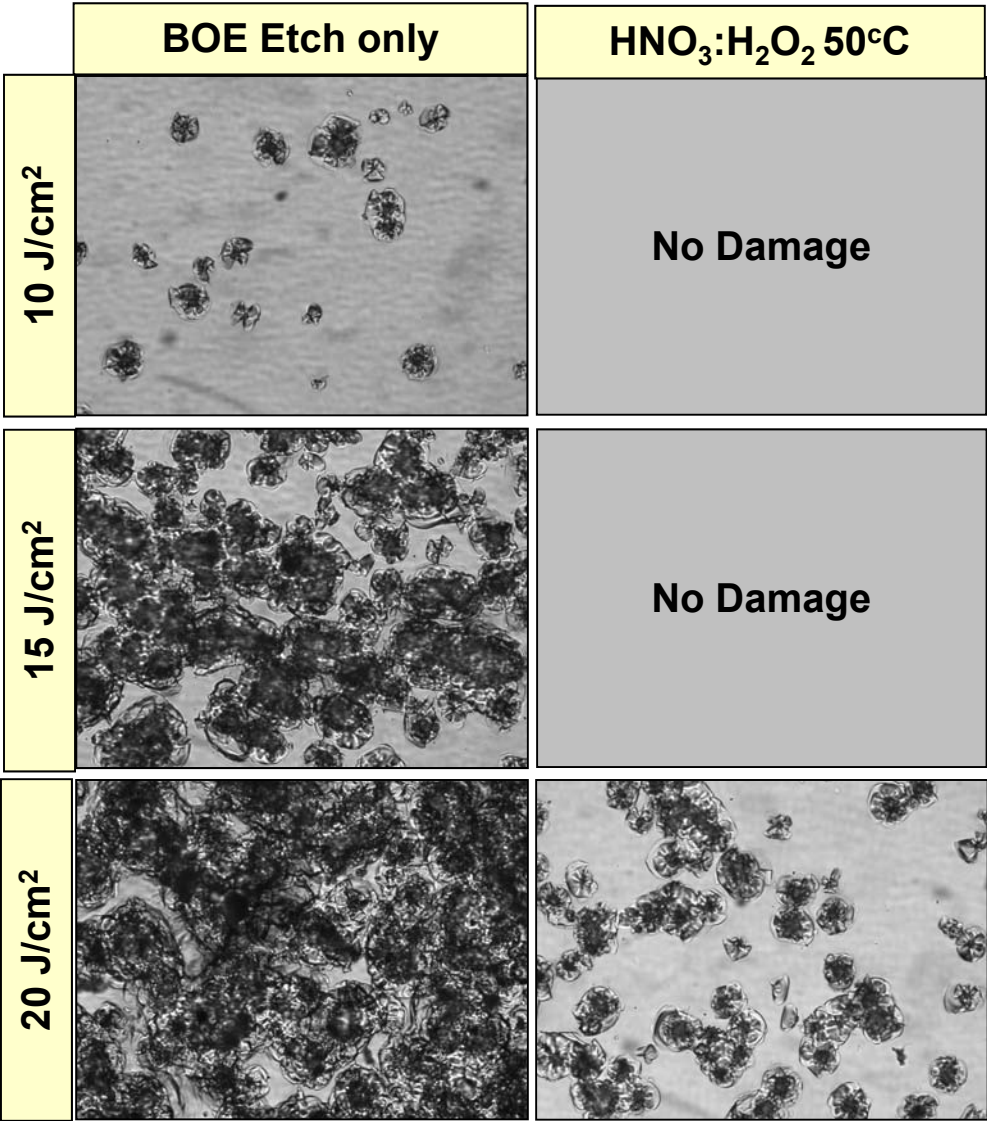
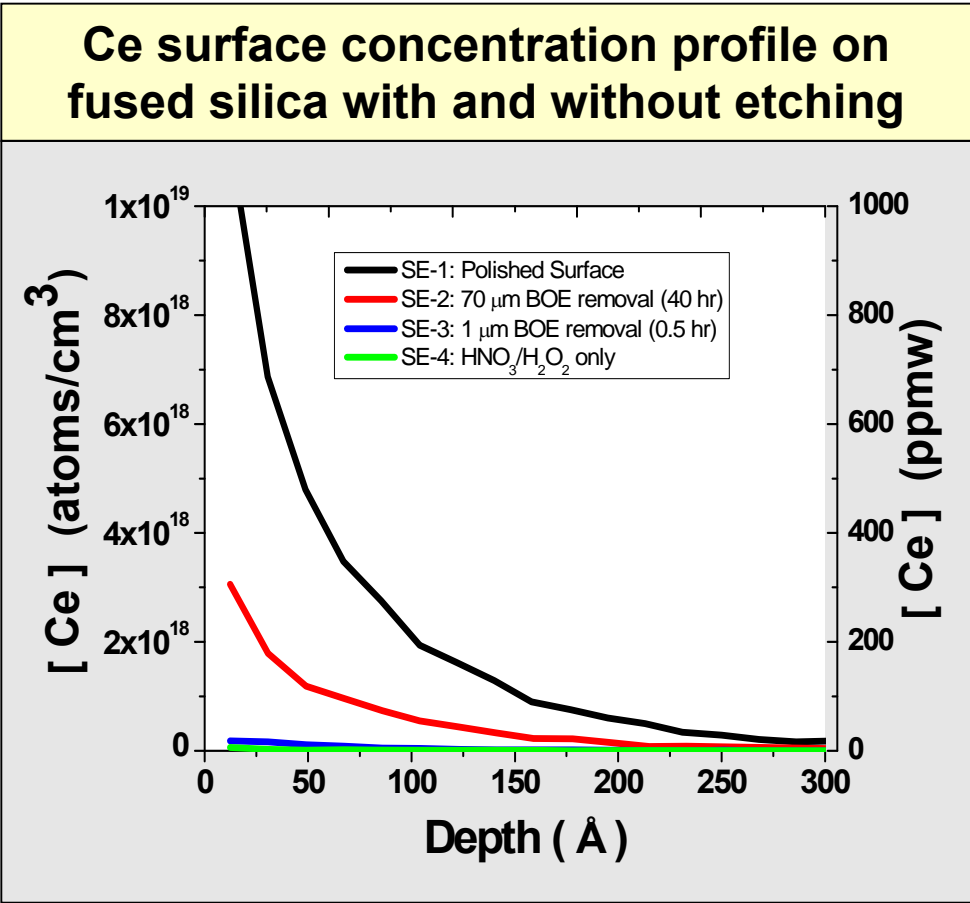


1. Near surface precursor is heated by absorption of laser light
2. T-activated bulk absorption,  $\alpha_{INT}(T)$ : precursor heats the bulk which begins to absorb (thermal runaway)
3. T-activated thermal conduction
4. Absorption front forms and propagates at velocity  $v_f$

# Fracture surfaces (not plastic deformation and densification) are low fluence absorbing precursors

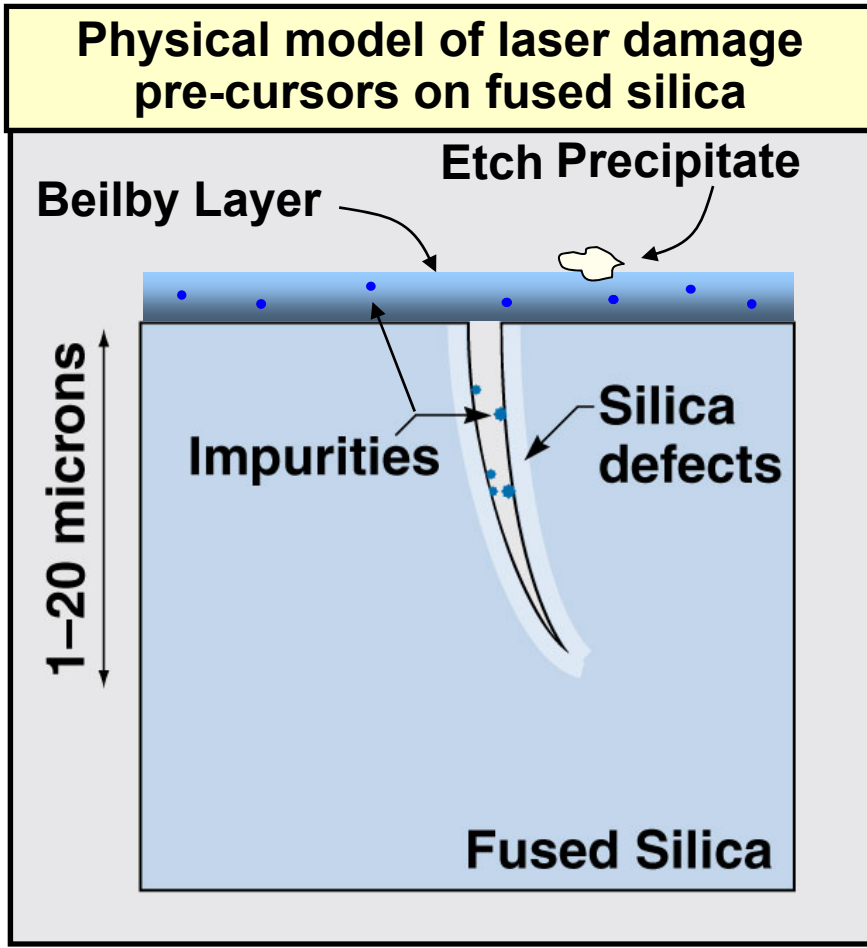


# Removal of subsurface impurities within the 'Beilby' polishing layer using $\text{HNO}_3:\text{H}_2\text{O}_2$ improves laser damage resistance



237 μm

# Three precursors on fused silica surface have been identified to lead to $3\omega$ laser damage



**1) CHEMICAL IMPURITIES** such as Ce in the Beilby layer and in fractures

**2) INTRINSIC SILICA DEFECTS ON FRACTURE SURFACES** (e.g. scratches)

**3) PRECIPITATION PRODUCTS** which can result from subsequent surface treatments (e.g. CO<sub>2</sub> laser, chemical etching)

# Our S&T has focused on understanding surface interactions on glass surfaces during fabrication, post processing and laser operation

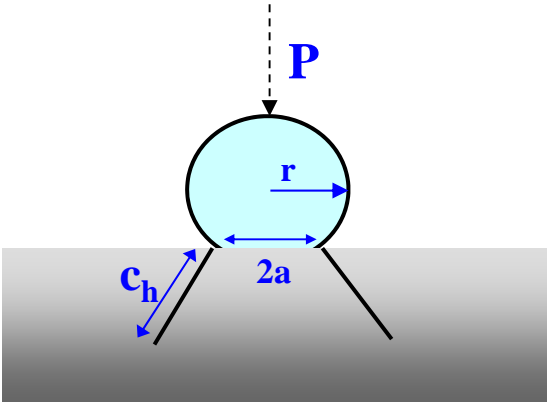
**Current Efforts**

**Future Challenges**

	1. Optical Fabrication	2. Post Processing & Coatings	3. Laser Operation
<b>Current Efforts</b>	<ul style="list-style-type: none"> <li>• Sub-surface damage management</li> <li>• Forensics of surface fractures</li> <li>• Fundamentals of material removal</li> <li>• Technology of full aperture &amp; small tool optical finishing</li> <li>• Low cost, precursor-free finishing techniques</li> </ul>	<ul style="list-style-type: none"> <li>• Development of chemical/thermal-based flaw/damage mitigation</li> <li>• Development of laser-based flaw/damage mitigation</li> <li>• Laser interference gratings development</li> </ul>	<ul style="list-style-type: none"> <li>• Mechanism of initiation &amp; growth (precursors &amp; modulation)</li> <li>• Precursor isolation &amp; identification</li> <li>• Quantitative understanding initiation &amp; growth behavior</li> <li>• Understanding solarization effects</li> <li>• Understanding modulation effects</li> </ul>
<b>Future Challenges</b>			<ul style="list-style-type: none"> <li>• Higher fluence precursor identification &amp; mitigation</li> <li>• Understand multi-pulse surface &amp; radiation effects</li> <li>• Understand/mitigating debris-induced damage</li> <li>• Understand damage mechanisms on other glass optics (including coatings)</li> <li>• Development of new glass optical materials (e.g., high fluence optical filters)</li> </ul>

# There are three basic types of cracks created by static brittle indentation

## Hertzian Cracks<sup>1</sup> (blunt)



**Initiation**

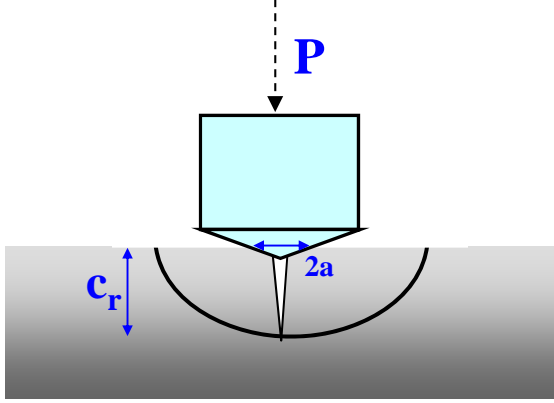
$$P_c = A r$$

**Growth**

$$c_h = \left( \frac{\chi_h P}{K_{Ic}} \right)^{2/3}$$

**Leads to subsurface damage**

## Radial Cracks<sup>1</sup> (sharp)

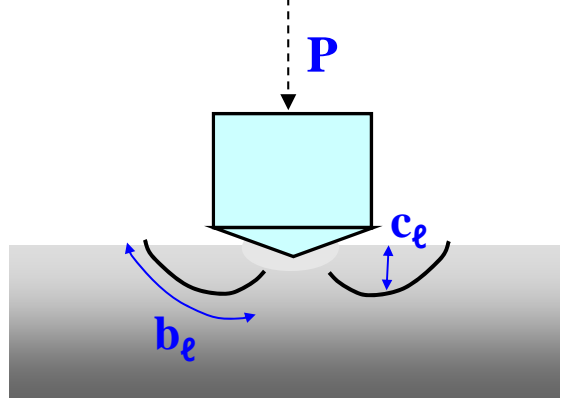


$$P_c = \alpha_r \frac{K_{Ic}^4}{H^3}$$

$$c_r = \left( \frac{\chi_r P}{K_{Ic}} \right)^{2/3}$$

**Leads to subsurface damage**

## Lateral Cracks<sup>2</sup> (sharp)



$$P_c = P_{cl}$$

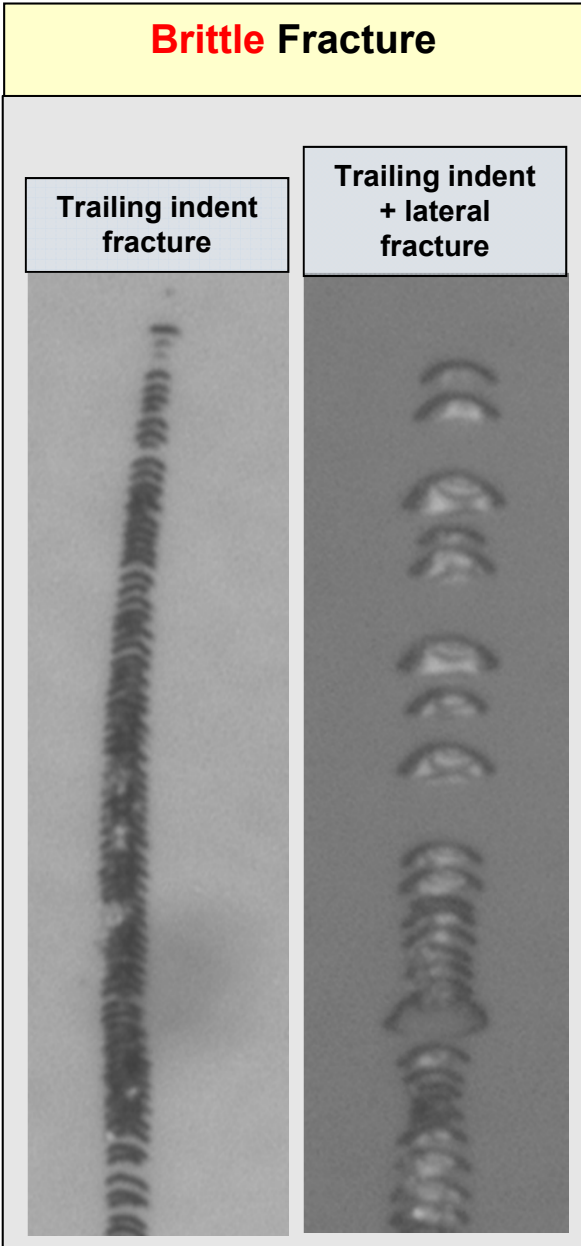
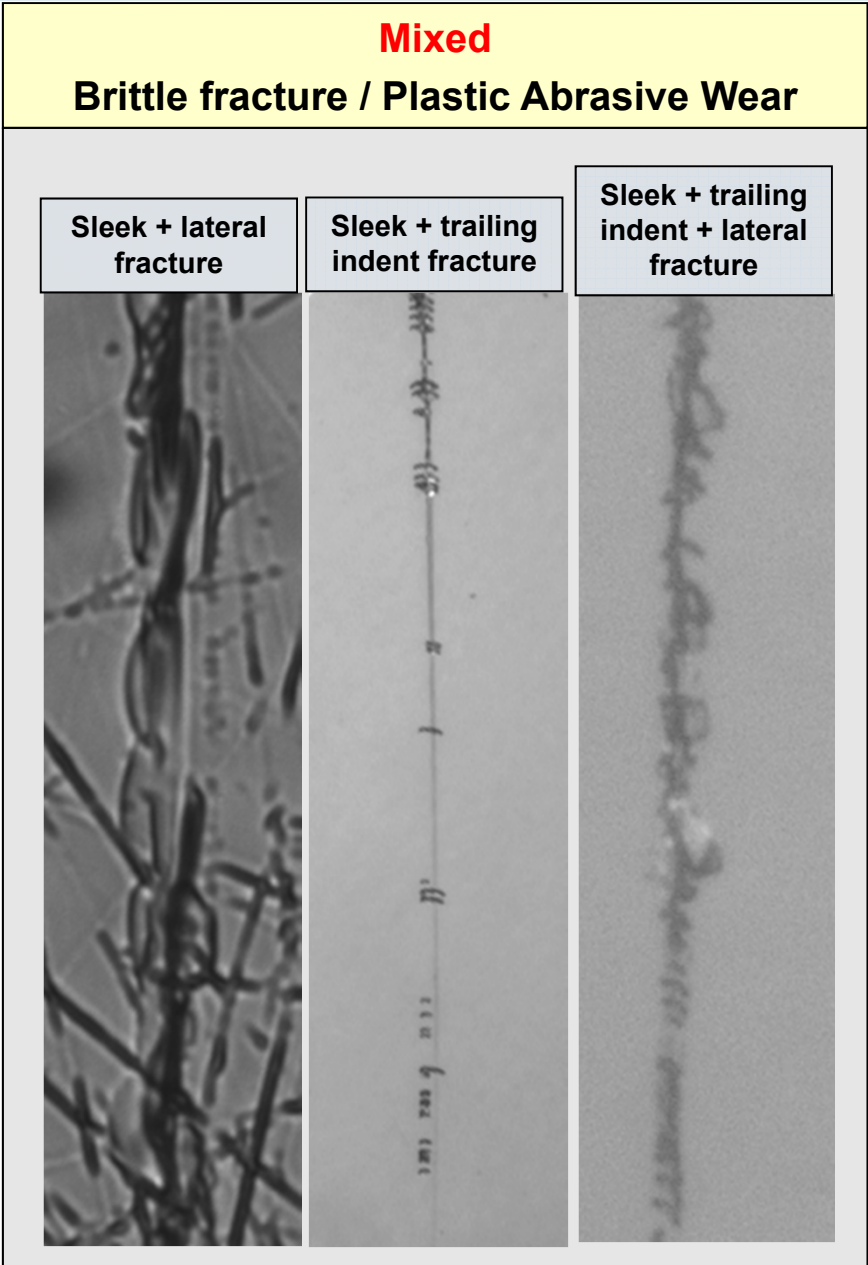
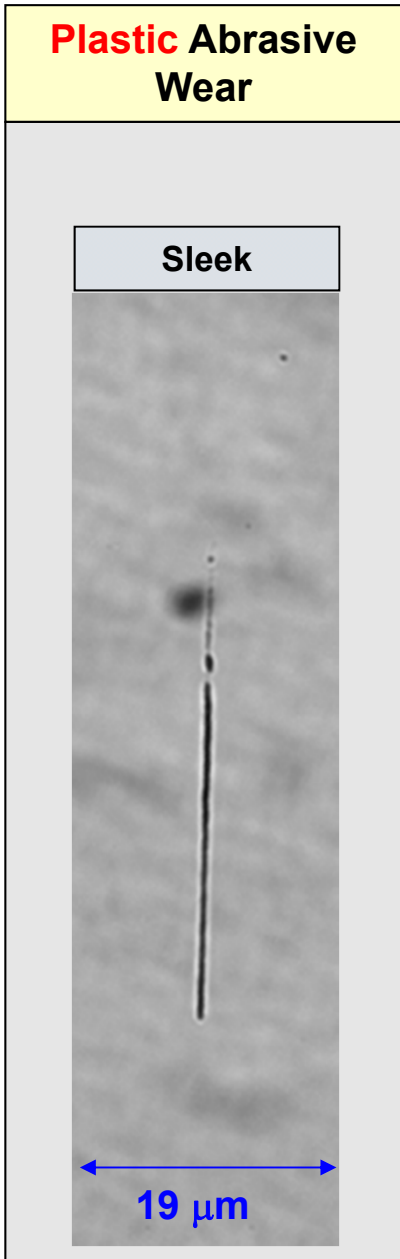
$$b_e = \chi_l \left( \frac{E}{H} \right)^{3/5} P^{5/8}$$

$$c_l = \frac{\chi_{l2} \left( \frac{E}{H} \right)^{2/5} P^{1/2}}{H^{1/2}}$$

**Leads to material removal**

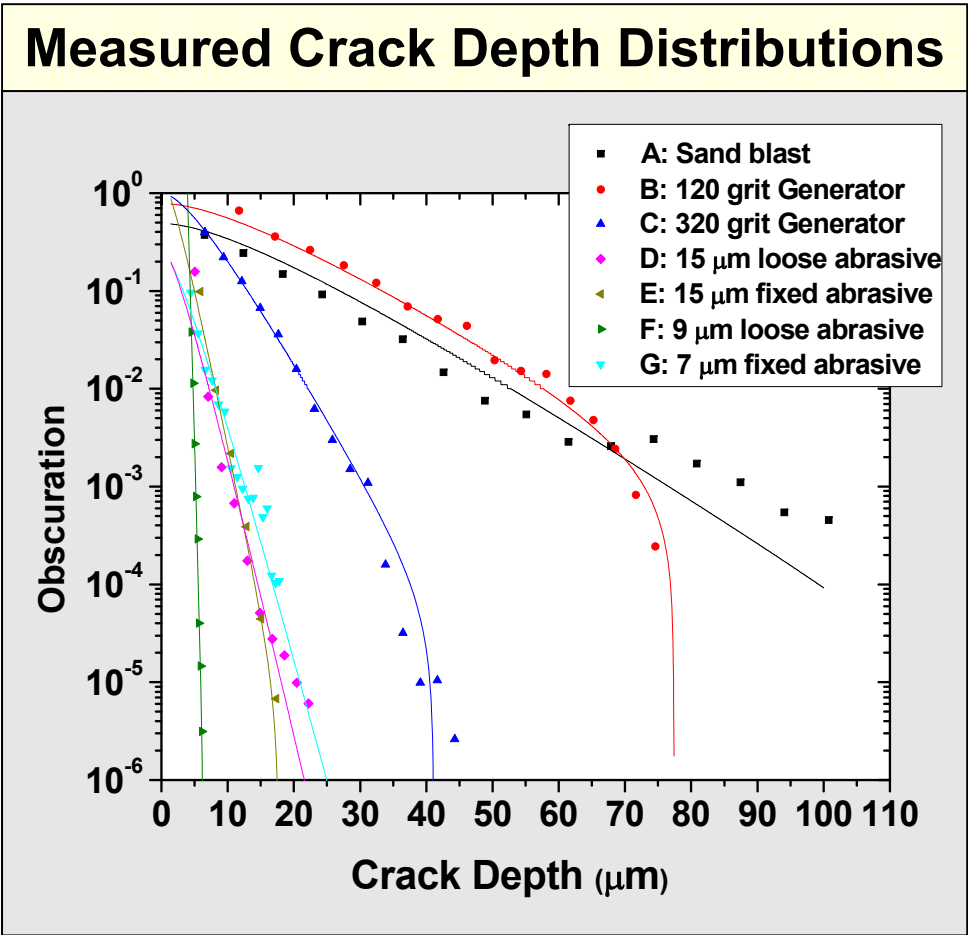
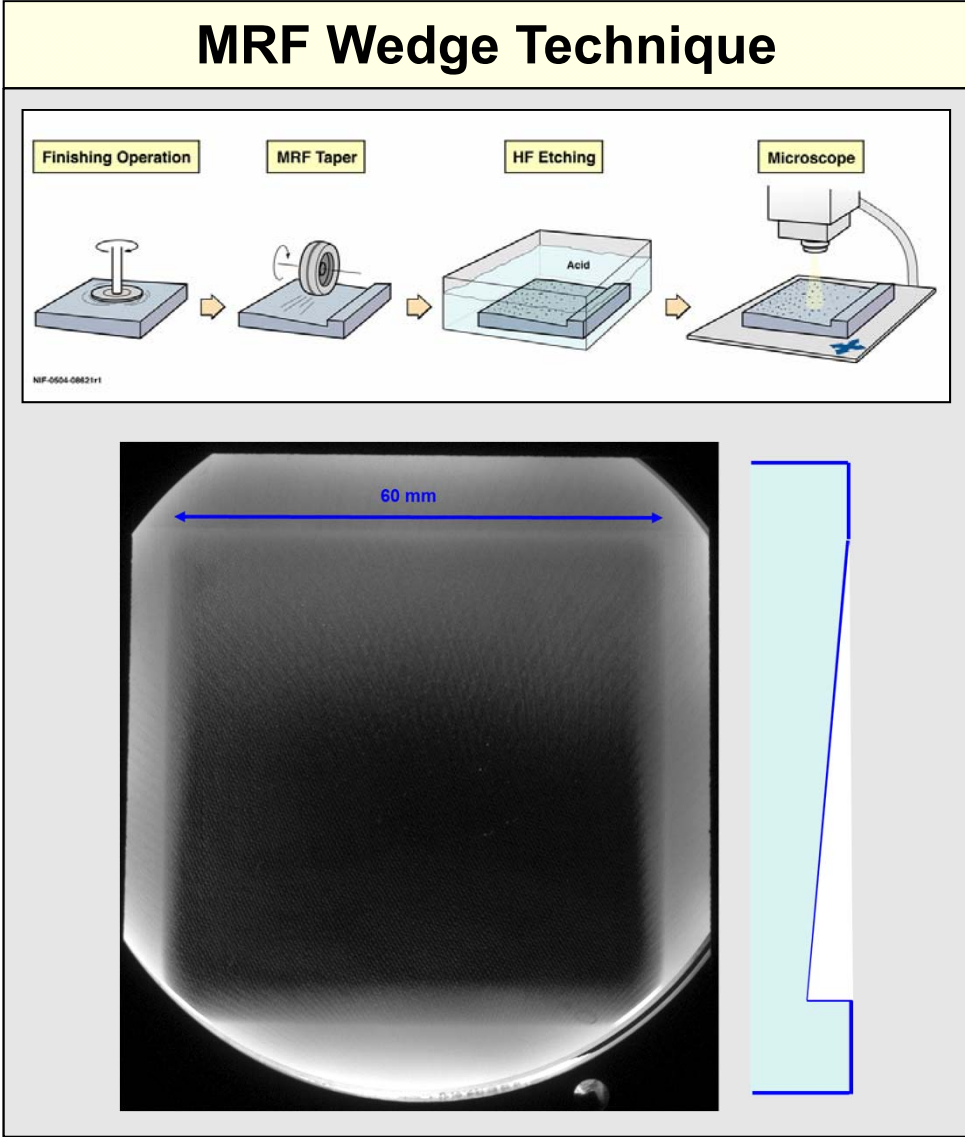
1. B. Lawn, "Fracture of Brittle Materials" (1993)  
 2. I. Hutchings "Tribology: Friction and Wear of Engineering Materials" (1992)

There are multiple types of scratches which can be divided into three basic categories

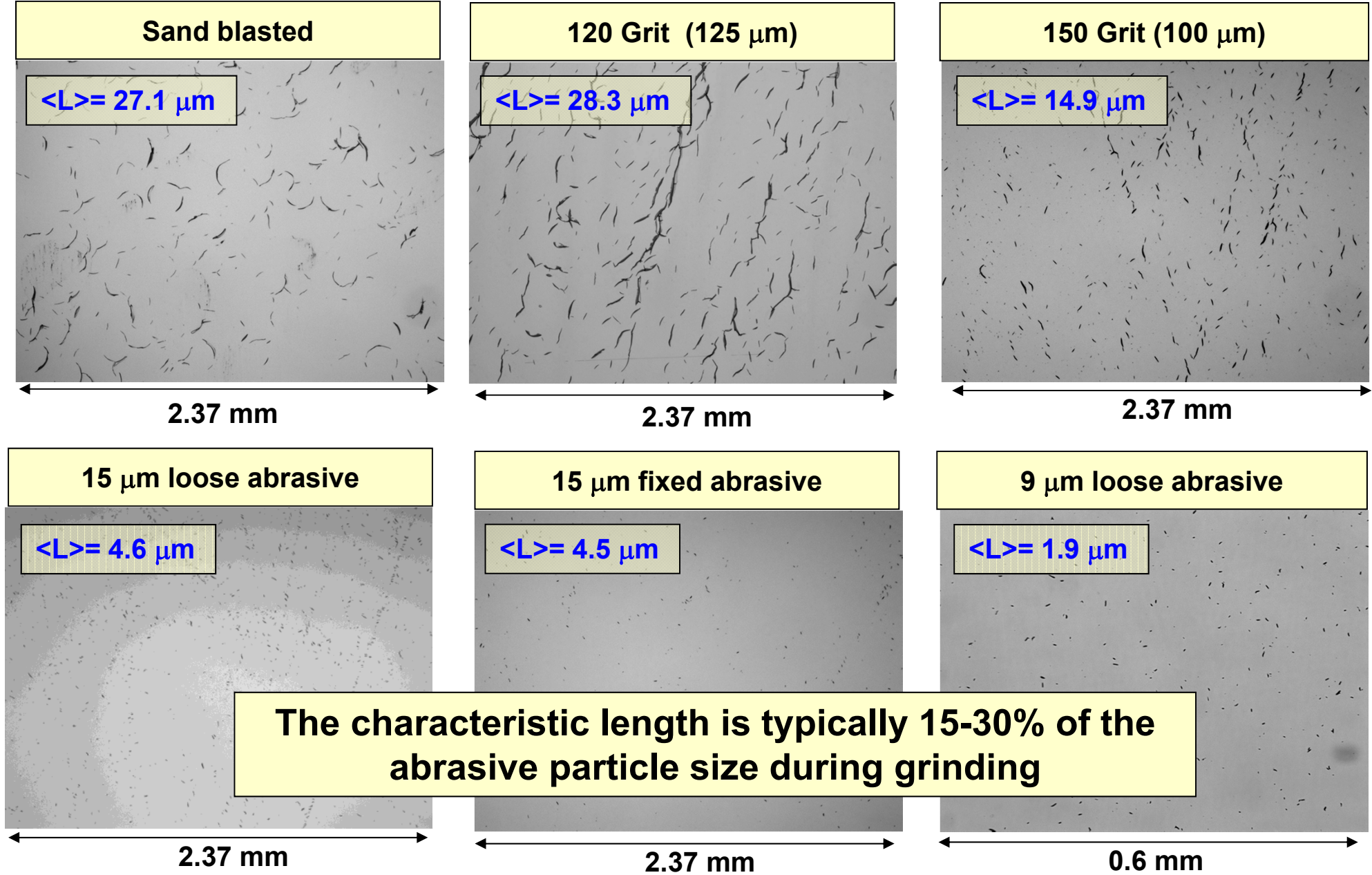




# The MRF wedge technique is a useful method to statistically measure the SSD length and depth distribution

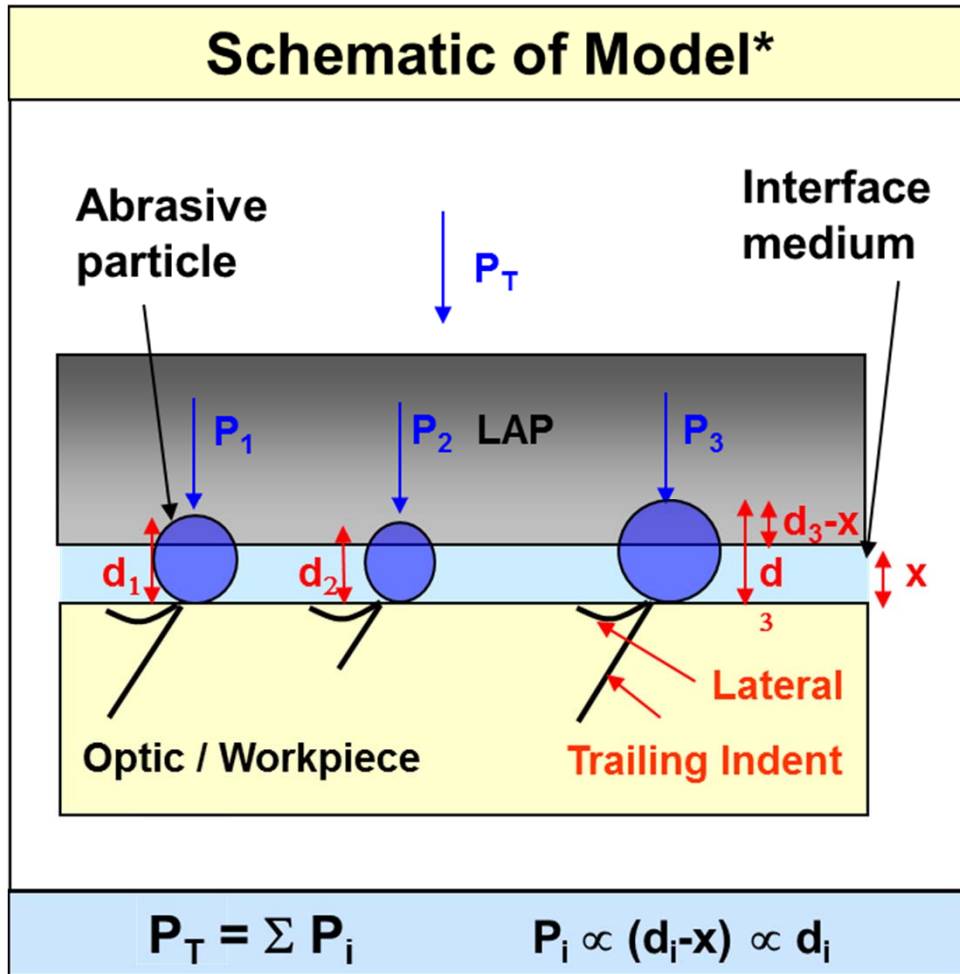


# Microscope images of the fractures show a unique size character for each grinding step



**The characteristic length is typically 15-30% of the abrasive particle size during grinding**

# A brittle fracture model has been successfully used to explain the observed distribution of crack depth and lengths



$$c(L) = \frac{L}{\frac{\pi}{2} \left( \frac{K_{Ic}}{\chi_h} \right)^{2/3} \left( \frac{2k N_L d_c}{3E P_T} \right)^{1/3}} = \frac{L}{\Omega}$$

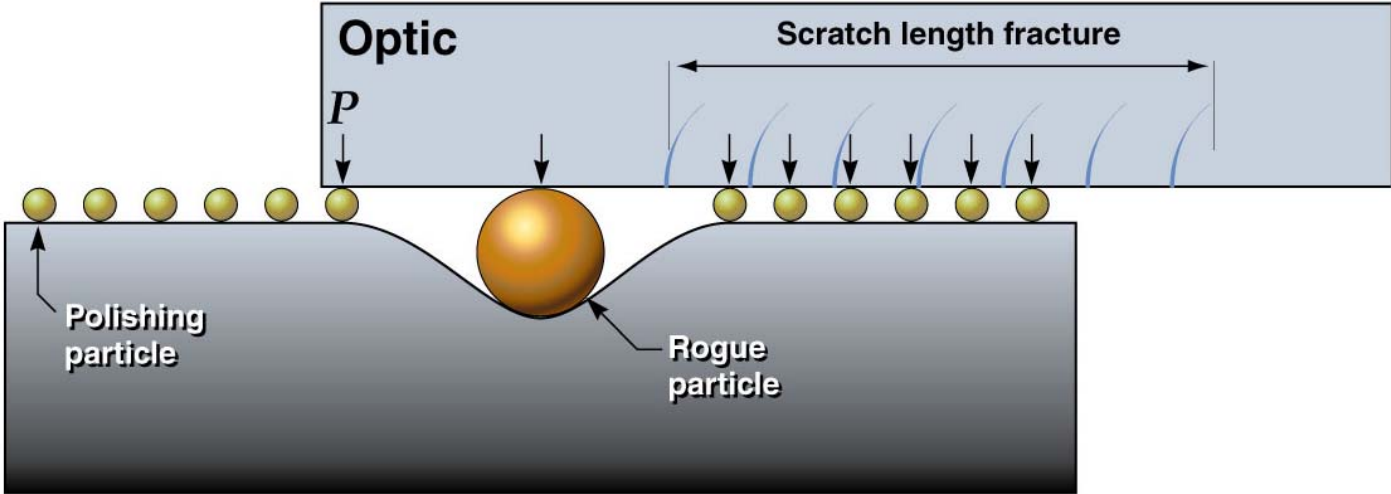
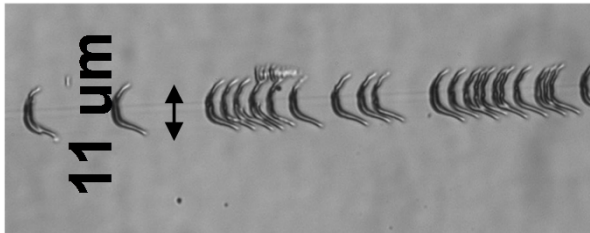
$$c_{90} = 0.9 \langle L \rangle$$

$$c_{max} = 2.8 \langle L \rangle$$

**Key assumption:** The load on particle is proportional to its vertical dimension

\*T. Suratwala, JNCS 352 (2006) 5601. P. Miller, SPIE 5991 (2005).

**During polishing large rogue particles or asperities bear high loads leading to sub-surface fractures (scratches)**



**Viscoelastic Lap (Pitch or Pad)**

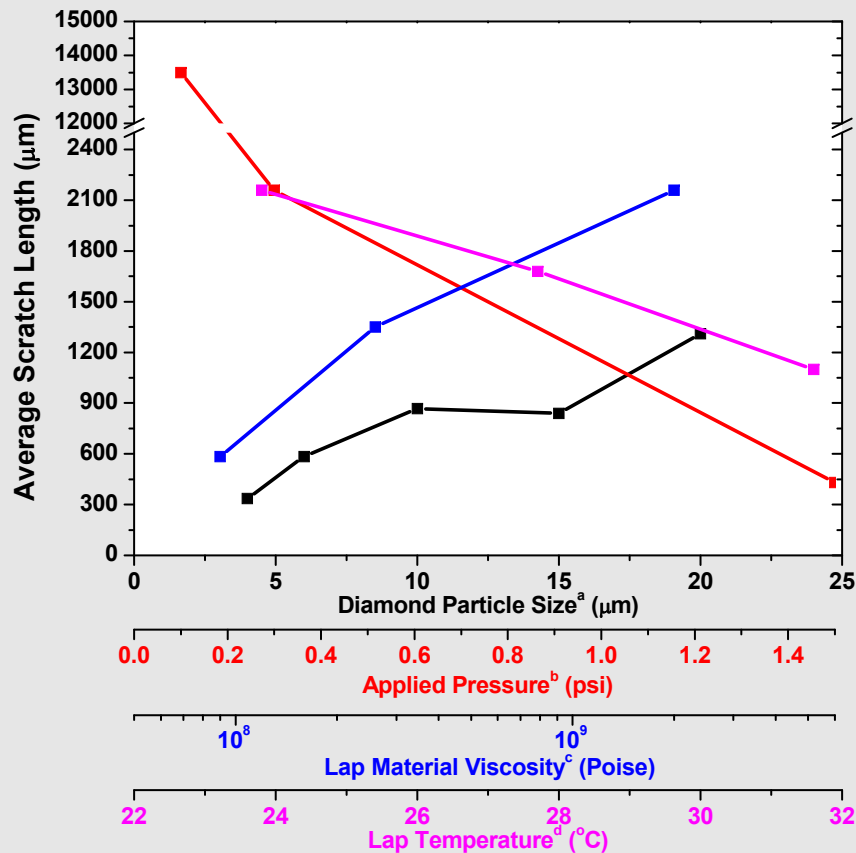
- Particle viscoelastically penetrates into pad
- Time frame of high load exposure determines scratch length

$$L_{scratch} = 8.9 \frac{v_{ave} \eta R^2}{P}$$

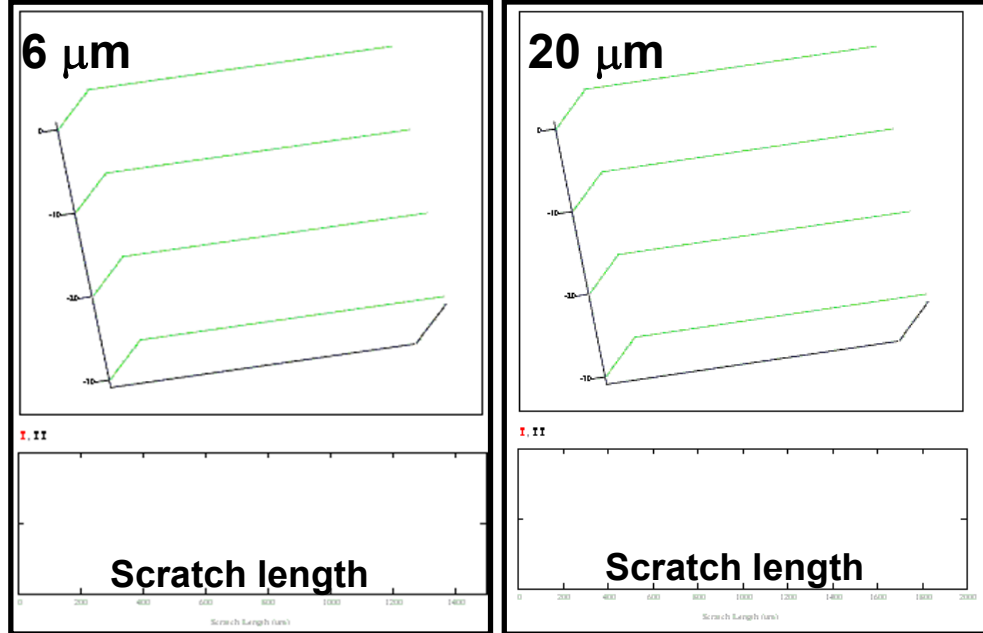
# The scratch length correlates with viscoelastic model wrt rogue particle size, pressure, lap viscosity, and lap temperature

tt = 0 msec

## Scratch length as a fn of various process parameters



## Simulation of rogue particle penetration



## Viscoelastic Penetration Model Solution: Ting model solution modified by Feit

$$h = \left\{ \begin{array}{l} \frac{a}{2} \log \left( \frac{R+a}{R-a} \right) - \left( R - \sqrt{R^2 - r^2} \right) \quad \text{for } r < a \\ \left[ \frac{a}{2} \log \left( \frac{R+a}{R-a} \right) - \left( R - \sqrt{R^2 - a^2} \right) \right] \left[ \left( 1 - \frac{1}{2} \left( \frac{r}{a} \right)^2 \right) \sin^{-1} \left( \frac{a}{r} \right) + \frac{1}{2} \left( \left( \frac{r}{a} \right)^2 - 1 \right)^{1/2} \right] \quad \text{for } r > a \end{array} \right.$$

# These studies have provided new rules that Opticians use to diagnose the cause of or to mitigate scratches

## Property of scratch

## What can it tell you?

## Rule / Example

1. Scratch width or trailing indent length (L)

- Size of rogue particle (d)
- Size distribution of Rogue Particles
- Process step
- Depth of fracture ( $c_{90}$  or  $c_{max}$ )

**For grinding**  

$$0.15 d \leq L \leq 0.3 d$$

**For polishing**  

$$0.3 d \leq L \leq 0.5 d$$

2. Number density  
 3. Scratch length ( $L_{scratch}$ )

- Rogue particle concentration
- Lap properties and rogue particle size

Sample	<L>
A: Sandblast	27.1 $\mu$ m
B: 120 grit	28.3 $\mu$ m
C: 320 grit	14.9 $\mu$ m
D: 15 $\mu$ m loose	4.6 $\mu$ m
E: 15 $\mu$ m fixed	4.5 $\mu$ m
F: 9 $\mu$ m loose	1.9 $\mu$ m
G: 7 $\mu$ m fixed	8.4 $\mu$ m

4. Scratch type (plastic, Brittle, mixed)

- Load during fracture
- Sharpness of particle
- Particle movement direction
- Particle rotation
- Stick slip behavior
- Pathway of indenting particle
- Shape of tool
- Handling vs polishing
- Material removal & figure

$$c_{90} = 0.9 \langle L \rangle \quad c_{max} = 2.8 \langle L \rangle$$

$P \approx 0.001 - 0.1 N$  Plastic only  
 $P \approx 0.1 - 5 N$  Plastic & Brittle  
 $P > 5 N$  Plastic & rubble

5. Orientation and Pattern of trailing indent

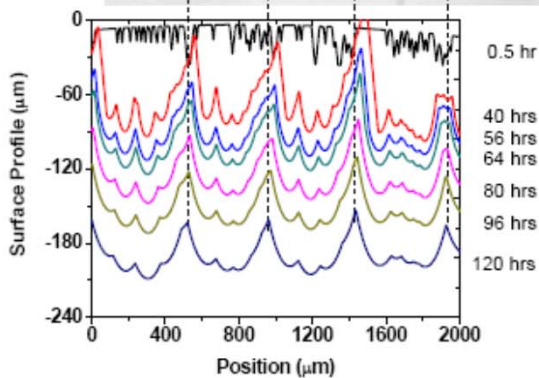
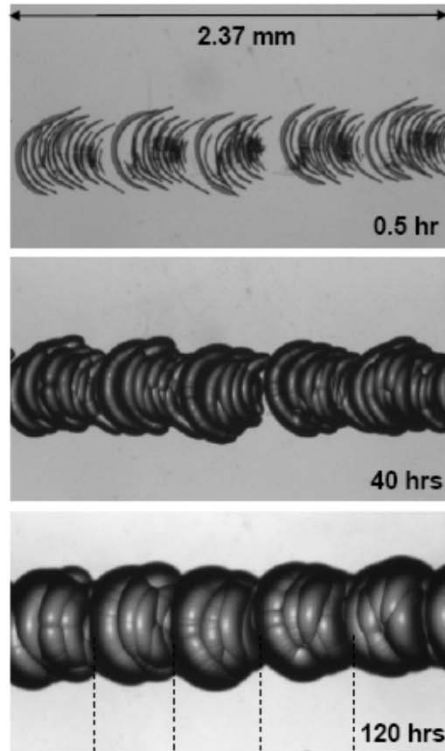
6. Curvature or scratch pattern

7. Location on optic

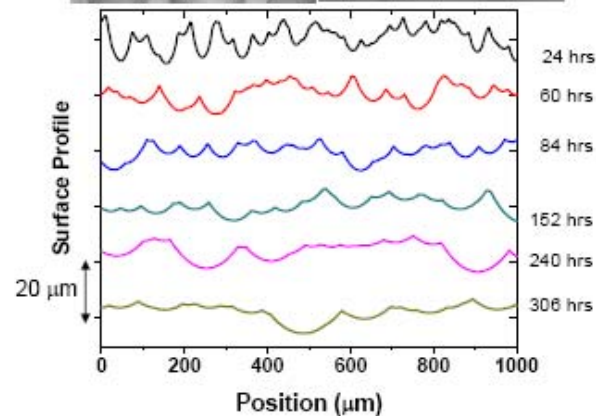
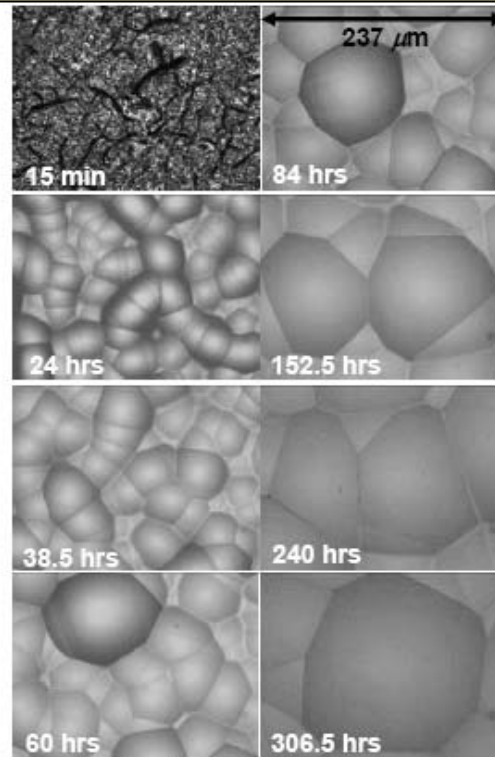
$$L_{scratch} = 8.9 \frac{v_{ave} \eta R^2}{P}$$

# HF etching can be used after grinding to remove subsurface fracture because it annihilates neighboring cracks

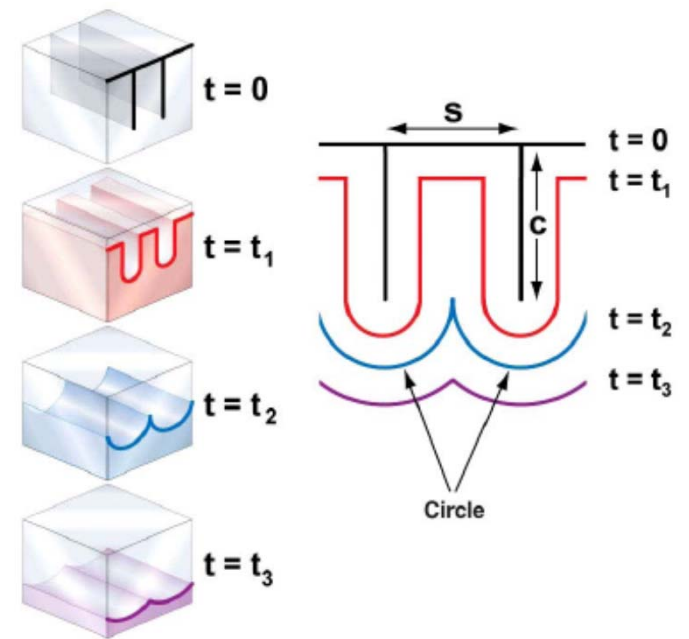
## Etching a scratch



## Etching ground surface

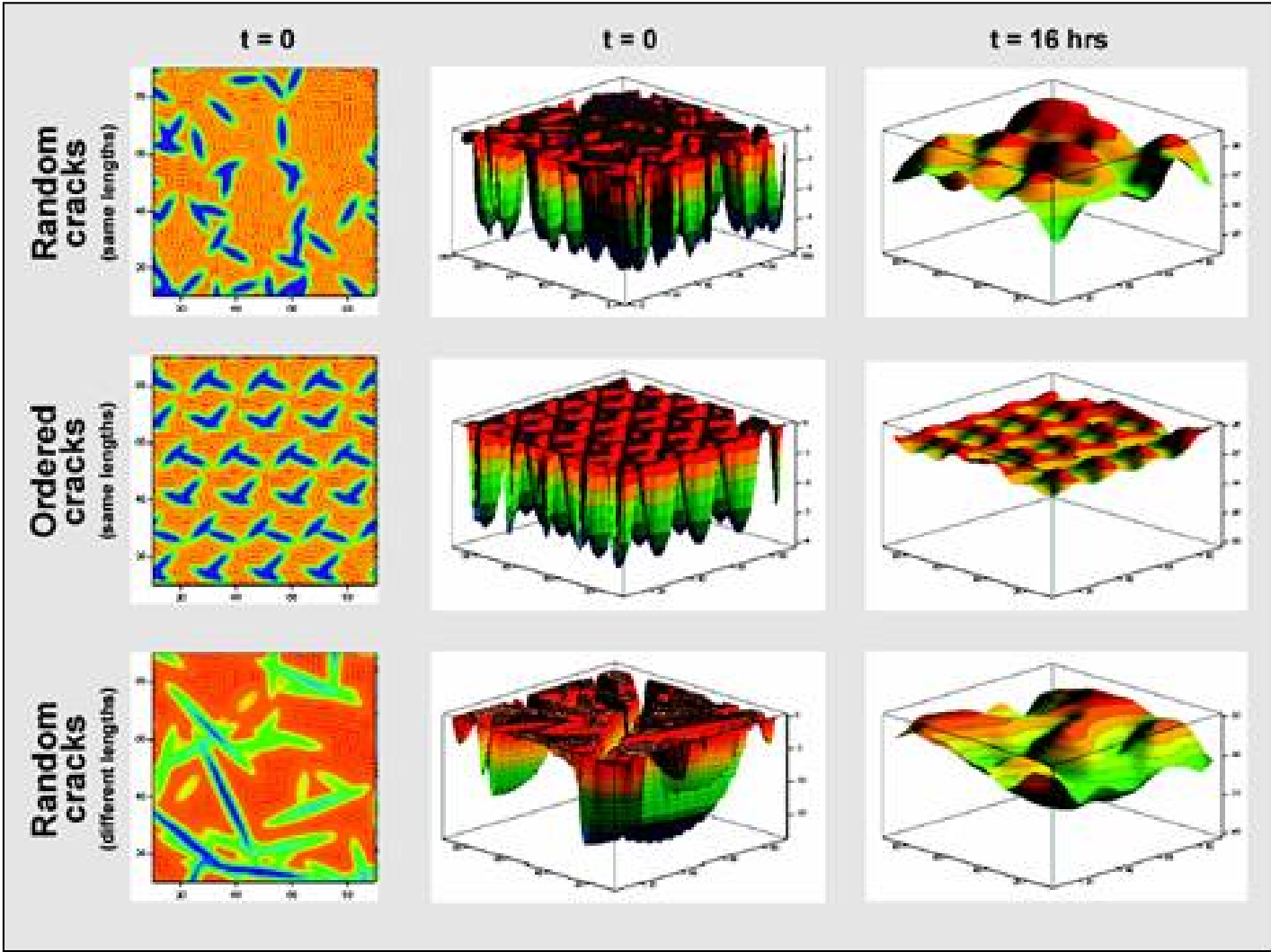


## Simple Geometric Model



A finite difference etching model has been developed to determine optimum etching times and key process variables

**Finite Difference Isotropic Etch Model**

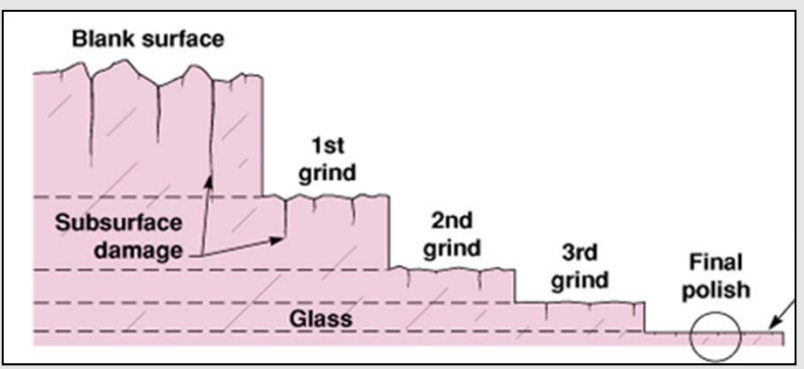


**Crack distribution strongly affects etching time needed for crack annihilation**



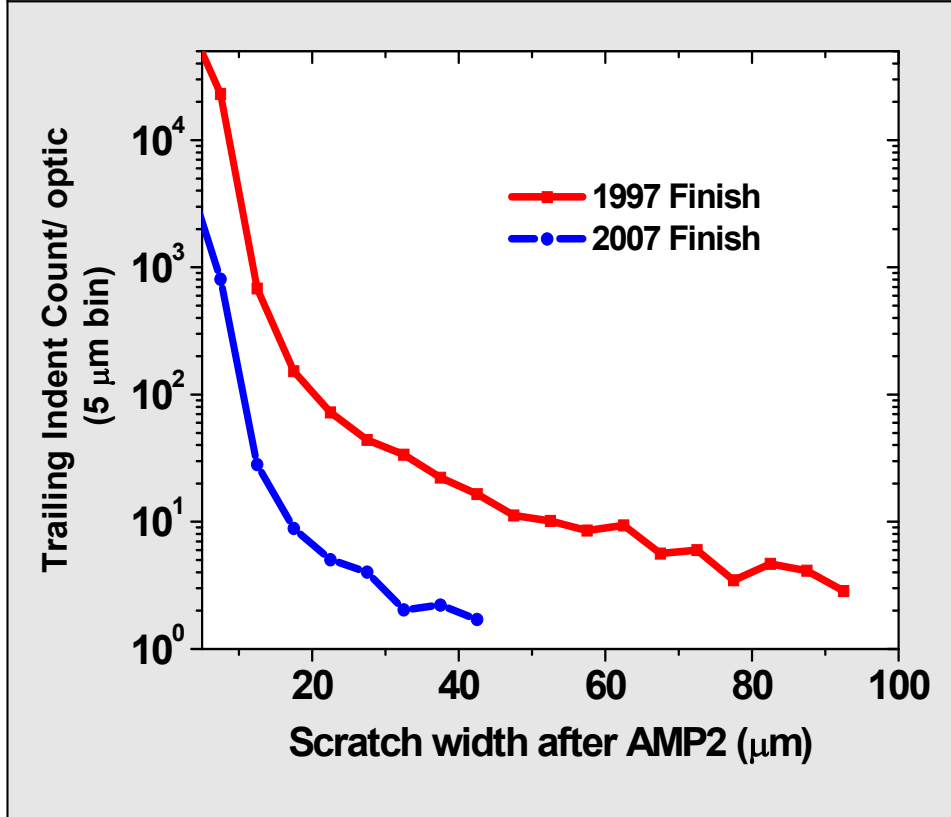
# Science & Technology based optical fabrication strategy was implemented to greatly reduced scratch densities

## Optical fabrication strategy



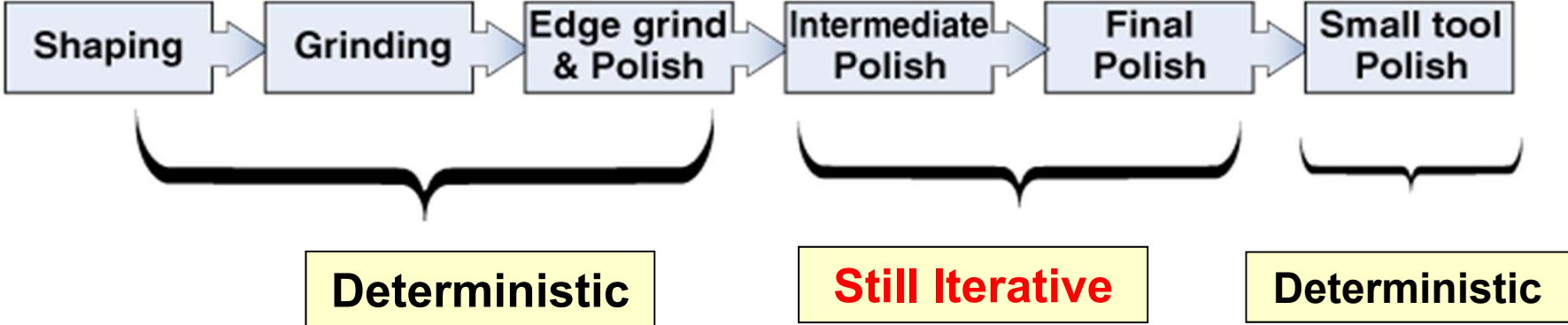
1. Measure the subsurface damage (SSD)
2. Define proper removal
3. Use etching to remove SSD after grinding
4. Ensure handling & cleaning prevents rogue particle contact
5. Remove rogue particles in polishers
6. Use etched scratch inspections
7. Use scratch forensics to identify & mitigate source of scratches

## The scratch density has dropped by ~20x in a 10 year period



Trailing indent = individual fractures in a scratch

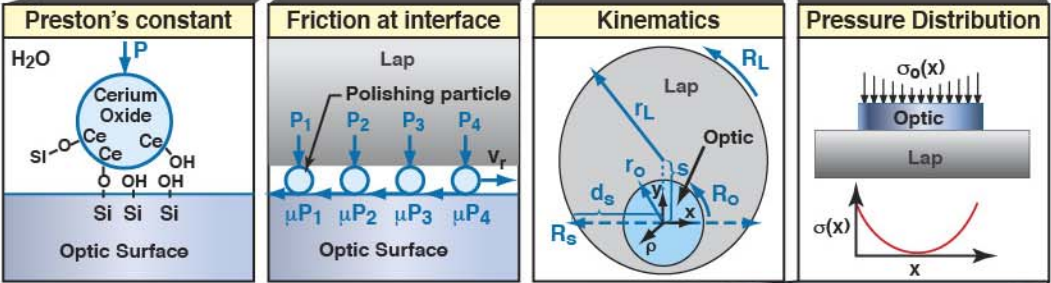
# Making intermediate and final polishing more deterministic will allow for making optics faster and cheaper



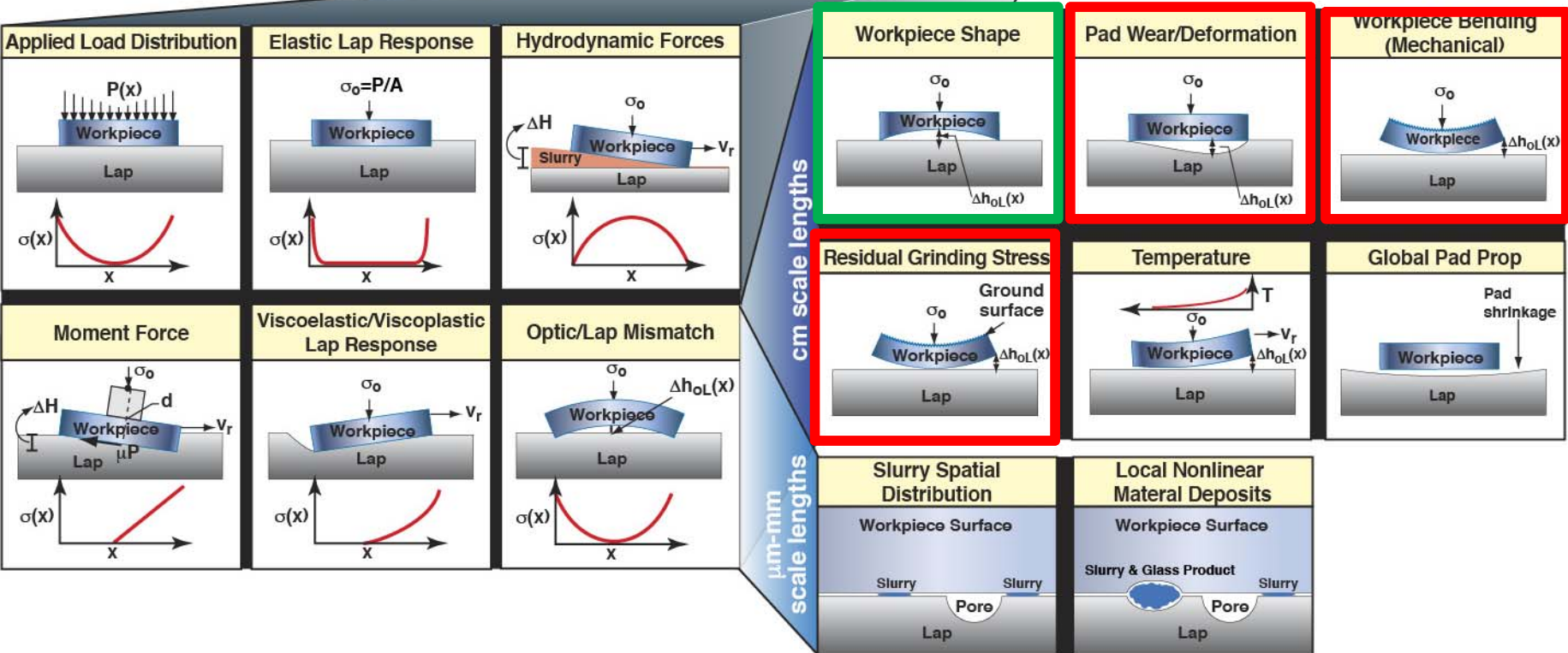
- Involves multiple polishing and metrology iterations
- Time consuming and labor intensive
- Figure not corrected here is performed by small tool

# Systematic effort to understand all the phenomena that affect material removal has been conducted

$$\frac{dh}{dt}(x, y, t) = k_p \underbrace{\mu(x, y, t)}_{\text{Friction at interface}} \underbrace{v_r(x, y, t)}_{\text{Kinematics}} \underbrace{s(x, y, z, t)}_{\text{Pressure Distribution}}$$

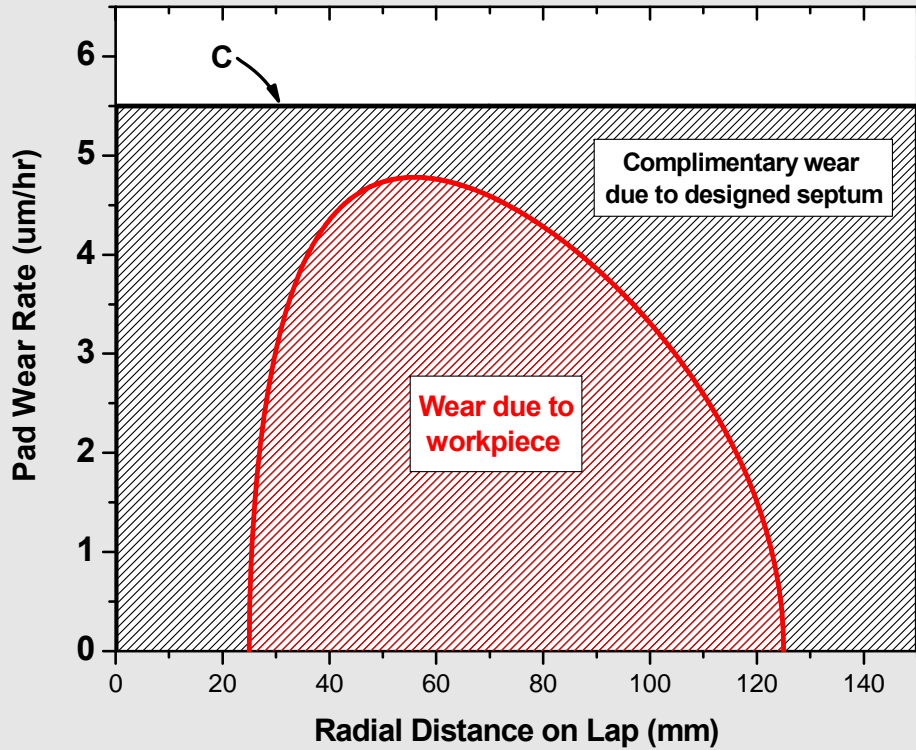


Our goal is to develop a polishing process which removes all spatial material removal non-uniformities except for Workpiece Shape



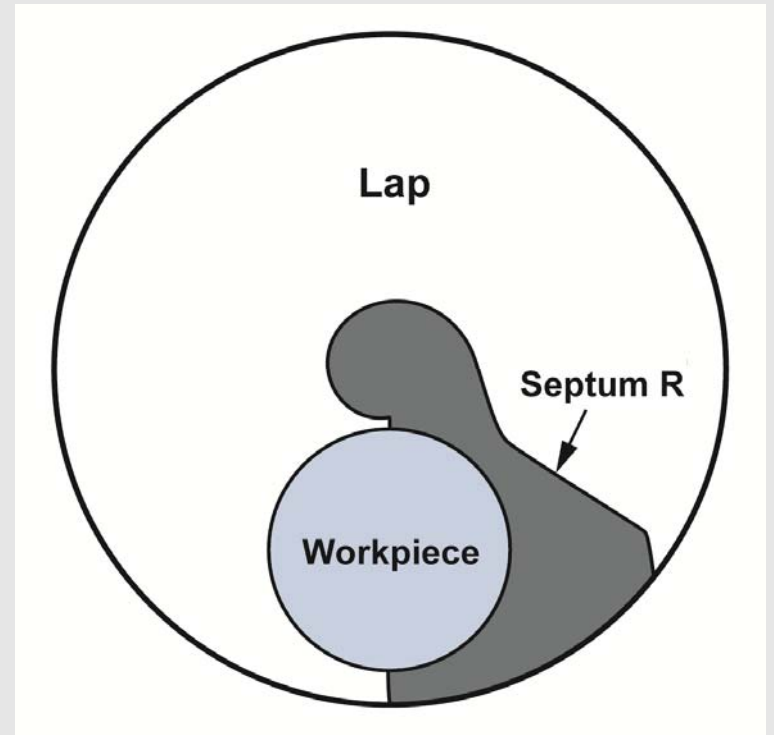
# A novel septum has been designed to counteract non-uniform wear on the pad

## Pad wear vs lap radius due to workpiece and engineered septum



$$\frac{dh_L(r)}{dt} = C = \underbrace{f_o(r)k_L\mu V_{r_o}\sigma}_{\text{lap wear due to workpiece}} + \underbrace{f_s(r)k_L\mu V_{r_s}\sigma}_{\text{lap wear due to septum}}$$

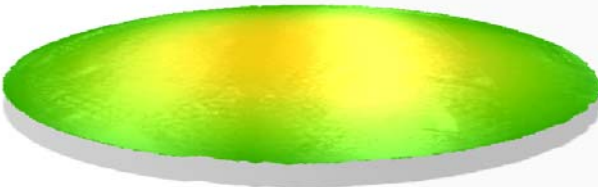
## Determined shape of Septum



$$w_s(r) = \frac{C - a \sin\left(\frac{x(r)}{r}\right) k_L \mu R_o s \sigma 2r}{k_L \mu R_L r \sigma}$$

# Chemical etching can effectively remove the residual stress and any complications to workpiece-lap mismatch

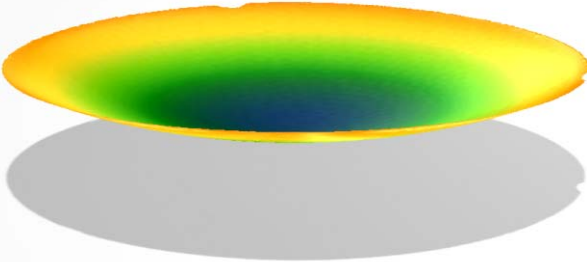
**Surface Figure of S2  
(Initial)**



**PV<sub>q</sub> = -1.29 μm**

- Polished Fused silica Workpiece (100 mm x 2.2 mm thick)

**Surface Figure of S2  
(After Grinding S1)**




**PV<sub>q</sub> = 3.65 μm**

- Grinding S1 puts compressive stress on S1; Hence S2 bends 4.8 μm
- Behavior shown to follow Twyman's Stress effect

$$PV = \frac{3 P_o(1-\nu)}{4 E} \left( \frac{D}{t} \right)^2$$

**Surface Figure of S2  
(After Grinding/Etching\*)**

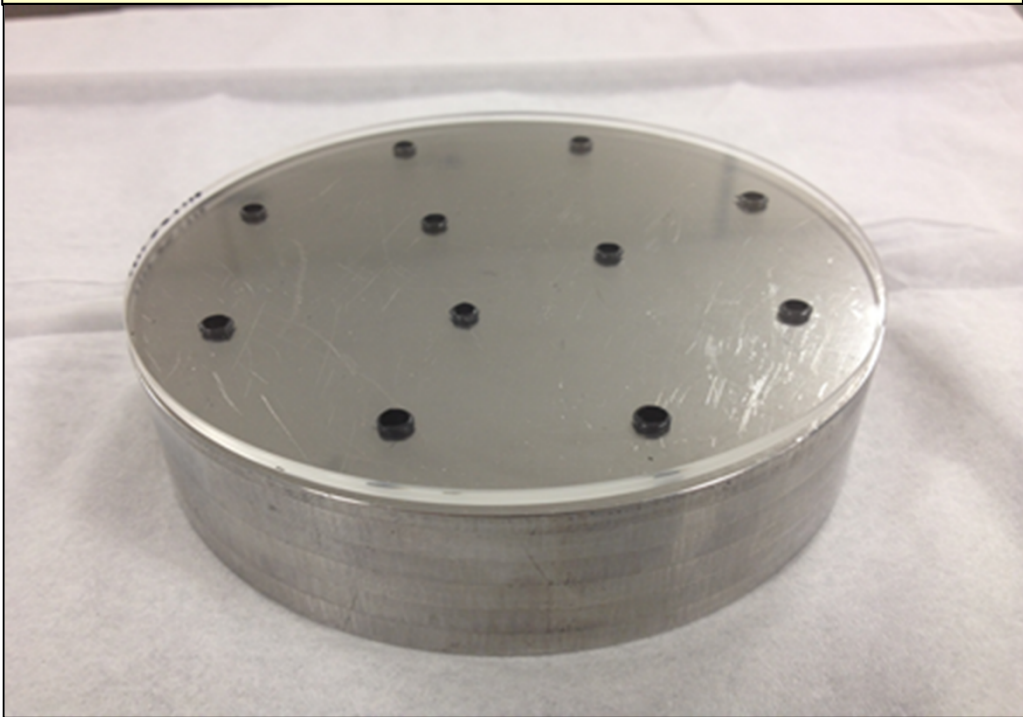


**PV<sub>q</sub> = -1.16 μm**

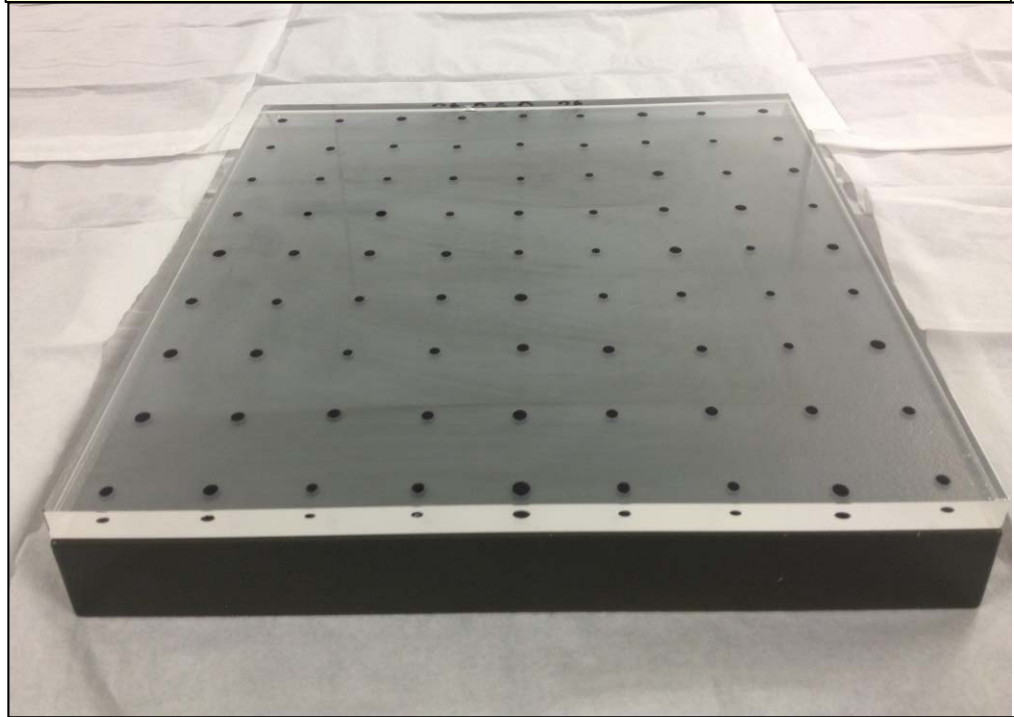
- Chemical etching removes residual stress & returns figure to initial state
- Etching after grinding will eliminate residual stress effects & contributions to non-uniform removal

# New Pitch Button Blocking (PBB) process provides low deflections for fused silica and phosphate glass

100 mm (diam) x 2.2 mm (thick)  
Fused Silica PBB



264 mm (side) x 8 mm (thick)  
Fused Silica PBB



<b>FS</b>	$\Delta PV = 0.003 \mu m$
<b>Phosphate</b>	$\Delta PV = 0.035 \mu m$

# A thermo-elastic model, with stress relaxation of pitch, can explain PBB behavior

**FlexPDE Model for PBB calculates deflection due to thermoelastic deflection**

**Setup**

**Workpiece Deflection**

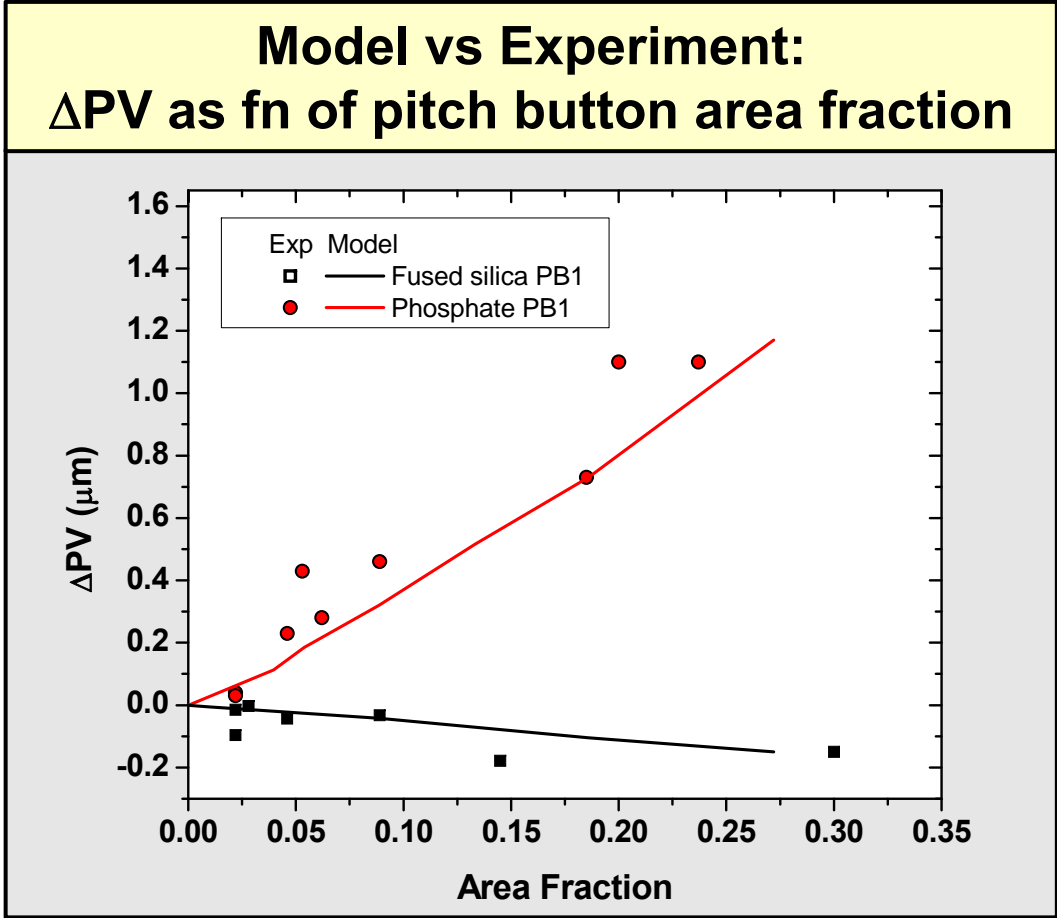
**Governing equations**

$$\frac{(1+2\nu)}{(1+\nu)(1-\nu)} - \alpha \frac{\partial T}{\partial x} = 0$$

$$\frac{(1+\nu)}{(1+\nu)} \frac{\partial T}{\partial y} = 0$$

$$\frac{(1+\nu)}{(1+\nu)} \frac{\partial T}{\partial z} = 0$$

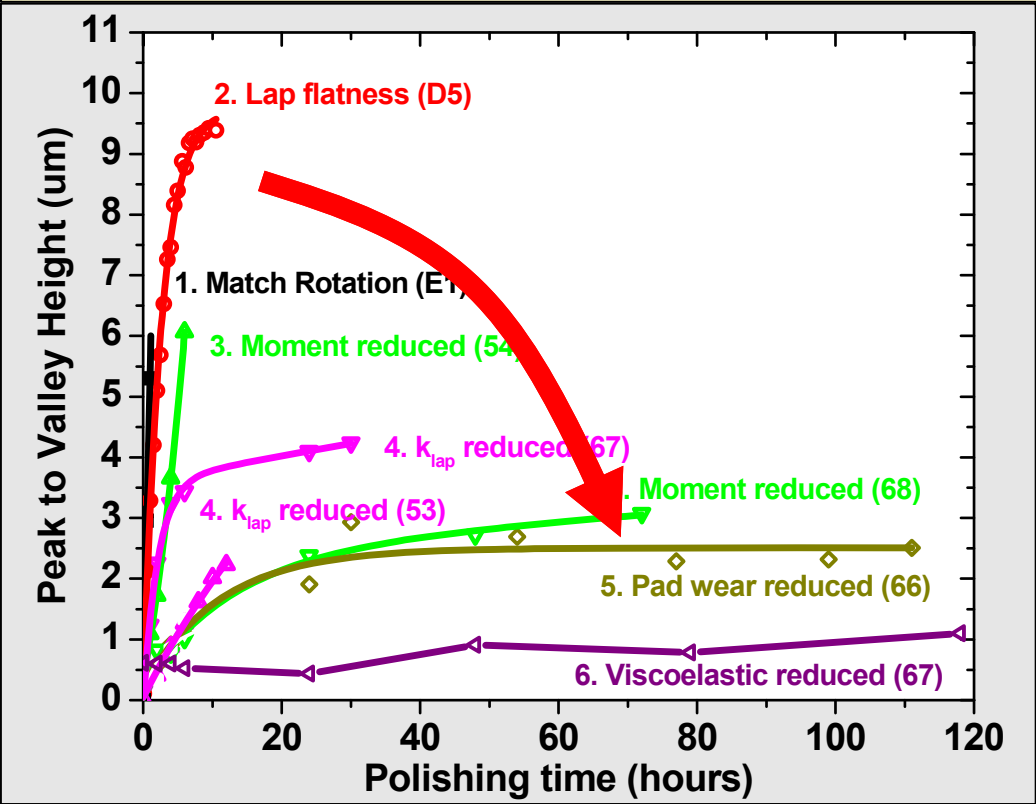
where  $du$  and  $(u, v, w)$  are displacement components



- Eff. thermal exp. coeff. of pitch to incorporate stress relaxation
  - Measured  $\alpha_{\text{pitch}} = 37.5 \times 10^6 \text{ K}^{-1}$
  - Used in Model  $\alpha_{\text{pitch}} = 2.4 \times 10^6 \text{ K}^{-1}$
- Have established an engineering rule for button design and repeatable process

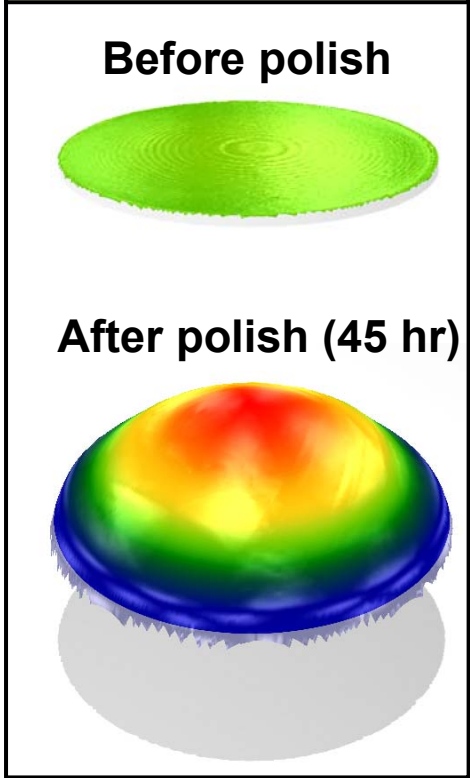
# The major sources of non-uniform spatial removal been identified and mitigated

**Workpiece Surface vs. Polishing Time for Different Configurations**



For all polishing runs:  $r_o=50$  mm;  $r_L=150$  mm;  $s = 75$  mm;  $r_s, d_s=0$ ;  $P_A=0.3$  psi

**Polishing Without Uniformity control**

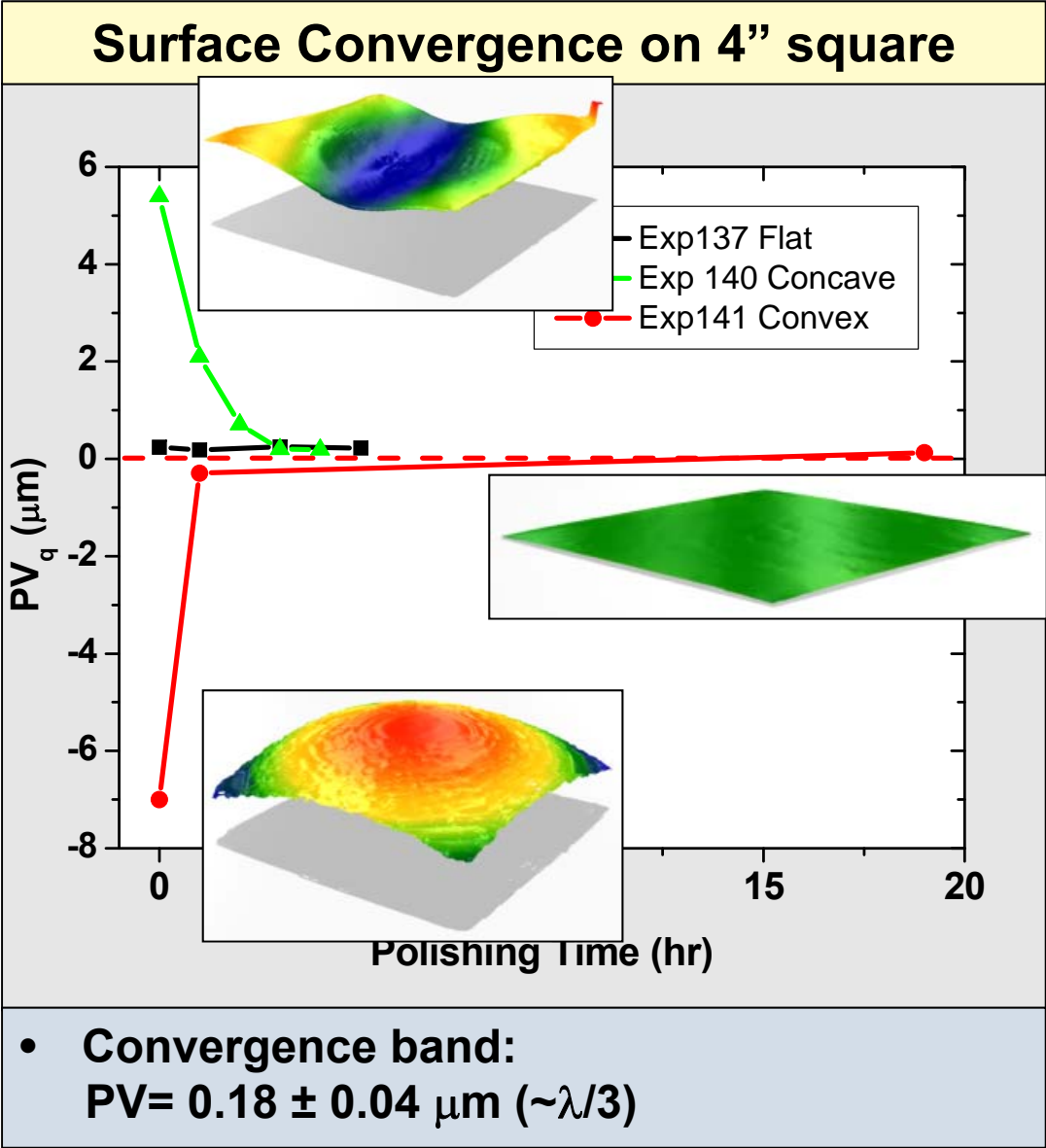


**Polishing With Uniformity Control**



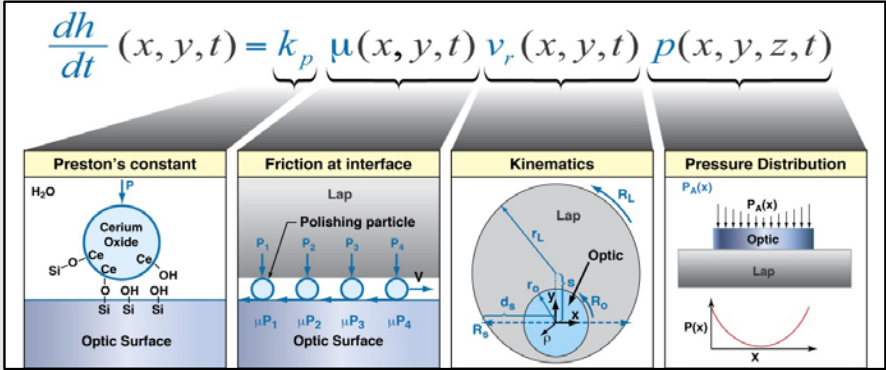


# New Convergent Polishing has been demonstrated on 4"-10" round & square plano glass optics



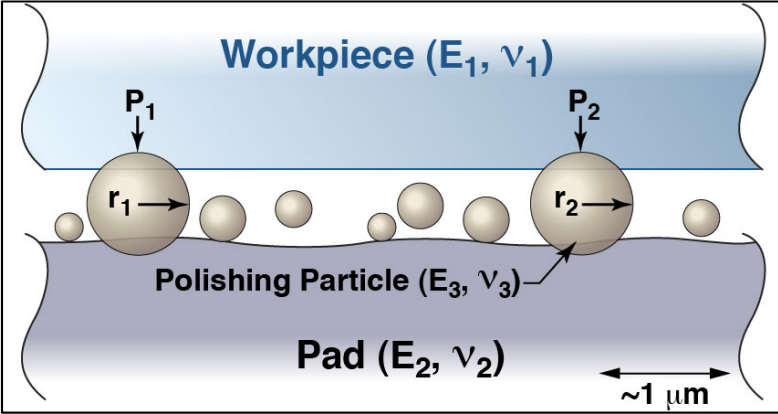
# The Preston model has been extended to the microscopic scale to describe smaller spatial scale length effects

## Macroscopic Material Removal



- Describes removal and surface for scales length **> 1 mm**
- $k_p$  and  $\mu$  is macroscopic ensemble values

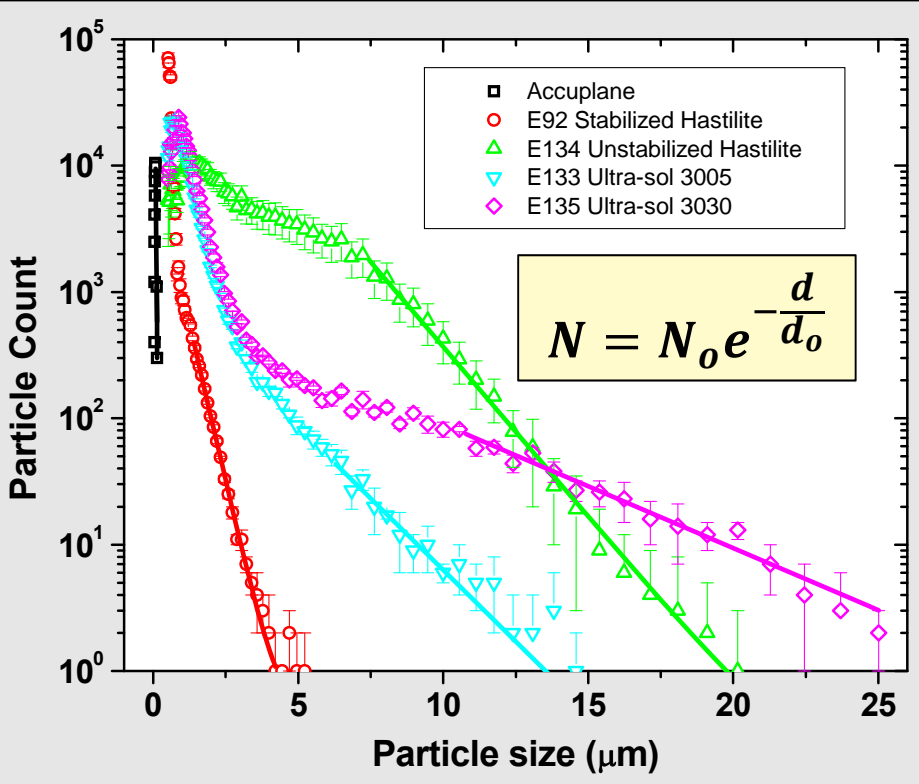
## Microscopic Material Removal



- Describes removal and surface for scales lengths **nm to mm**
- Hertzian contact zone determines removal area
- Lap topology and particle size dist determine number of contacts
- Ensemble determines macroscopic value of  $k_p$  and  $\mu$

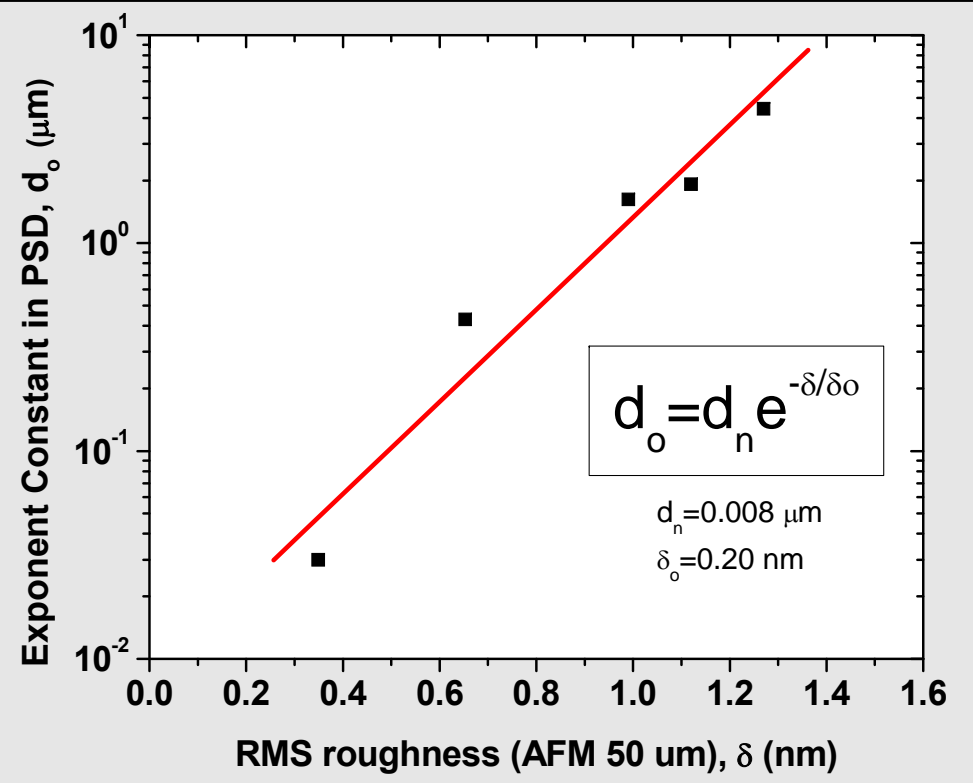
# The slurry's tail end of the distribution strongly correlates with workpiece roughness

**Measured particle size distributions of ceria slurries**



The tail end of each slurry can be fit to single exponential distribution

**Exponent constant in PSD of slurry vs RMS roughness of polished surface**

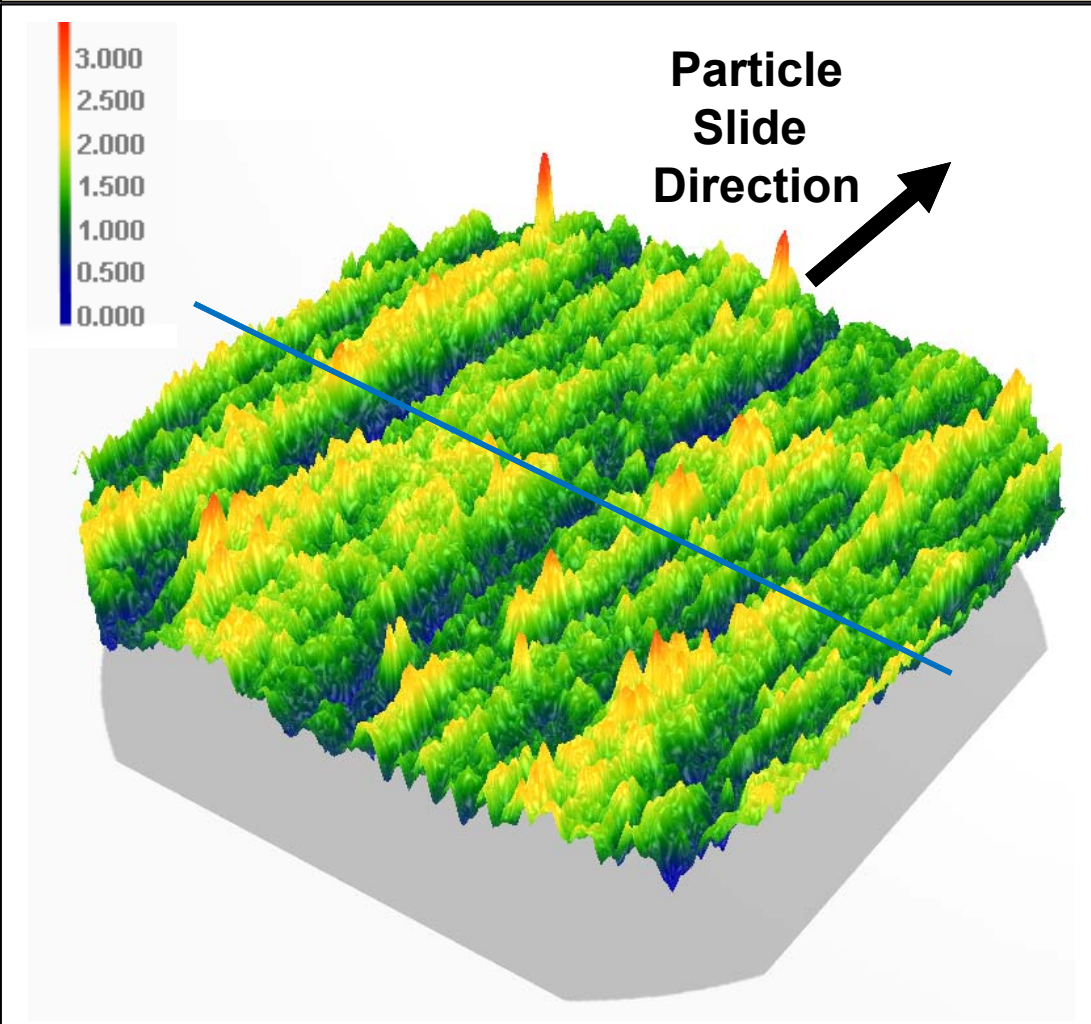


The slope of the slurry's particle size distribution quantitatively scales with the rms roughness

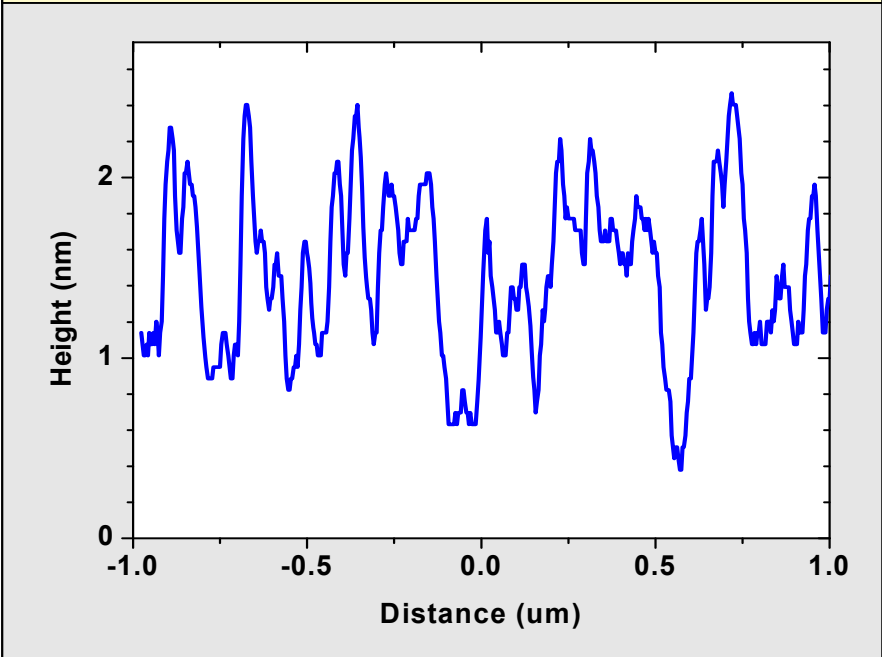
**Stresses the need to get slurry PSD with small  $d_0$  to get low roughness surface; Mean particle size is not as important!**

# Single pass of ceria particle removes ~1 nm of material (~7 Si-O units)

AFM Image (2  $\mu\text{m}$  x 2 $\mu\text{m}$ ) of Sample 4

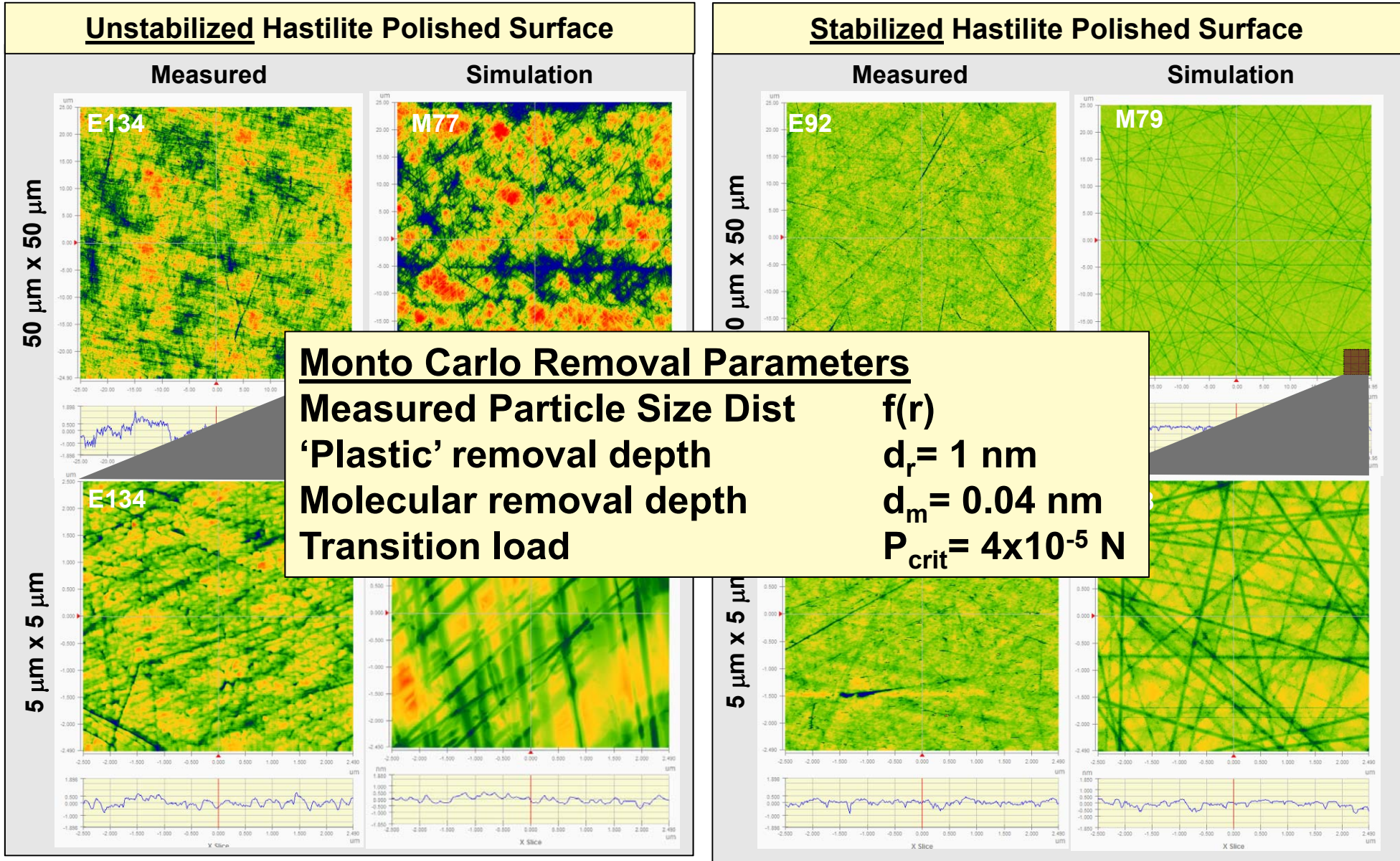


Lineout of AFM Perp. to slide particle slide direction



# Using a single set of parameters, polished surfaces have been simulated over multiple spatial scale lengths using different slurry particle size distributions

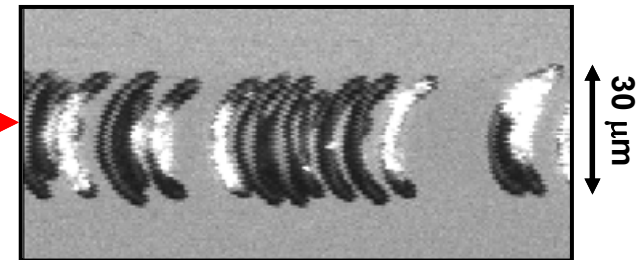
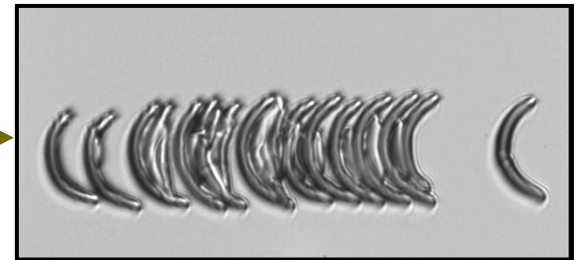
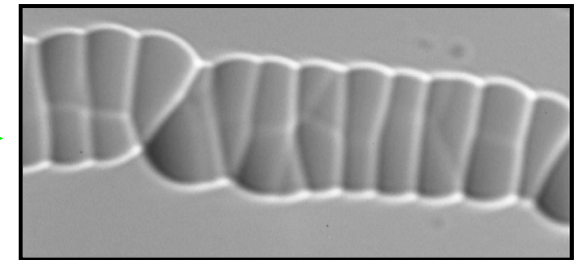
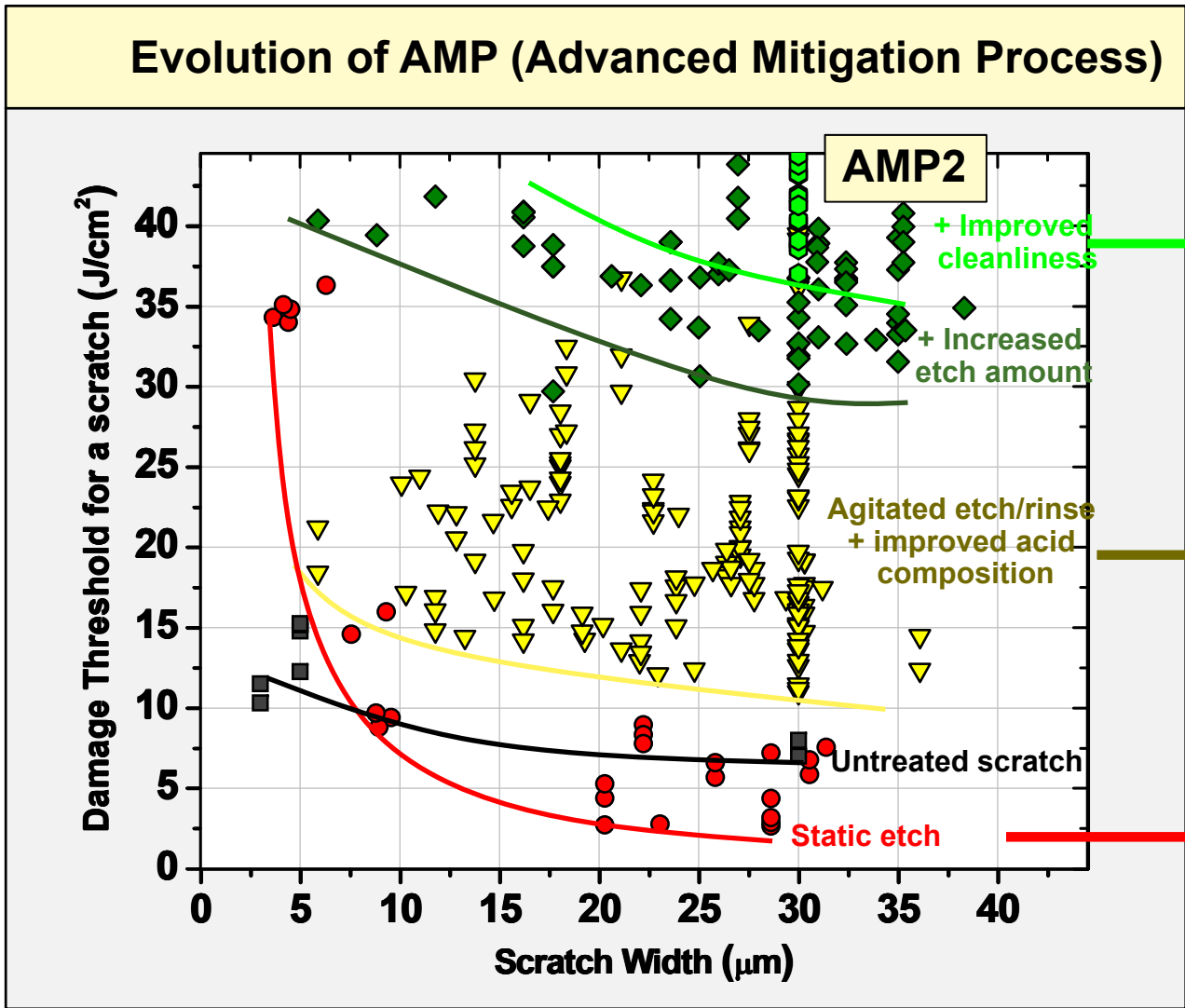
11/23/12



# Our S&T has focused on understanding surface interactions on glass surfaces during fabrication, post processing and laser operation

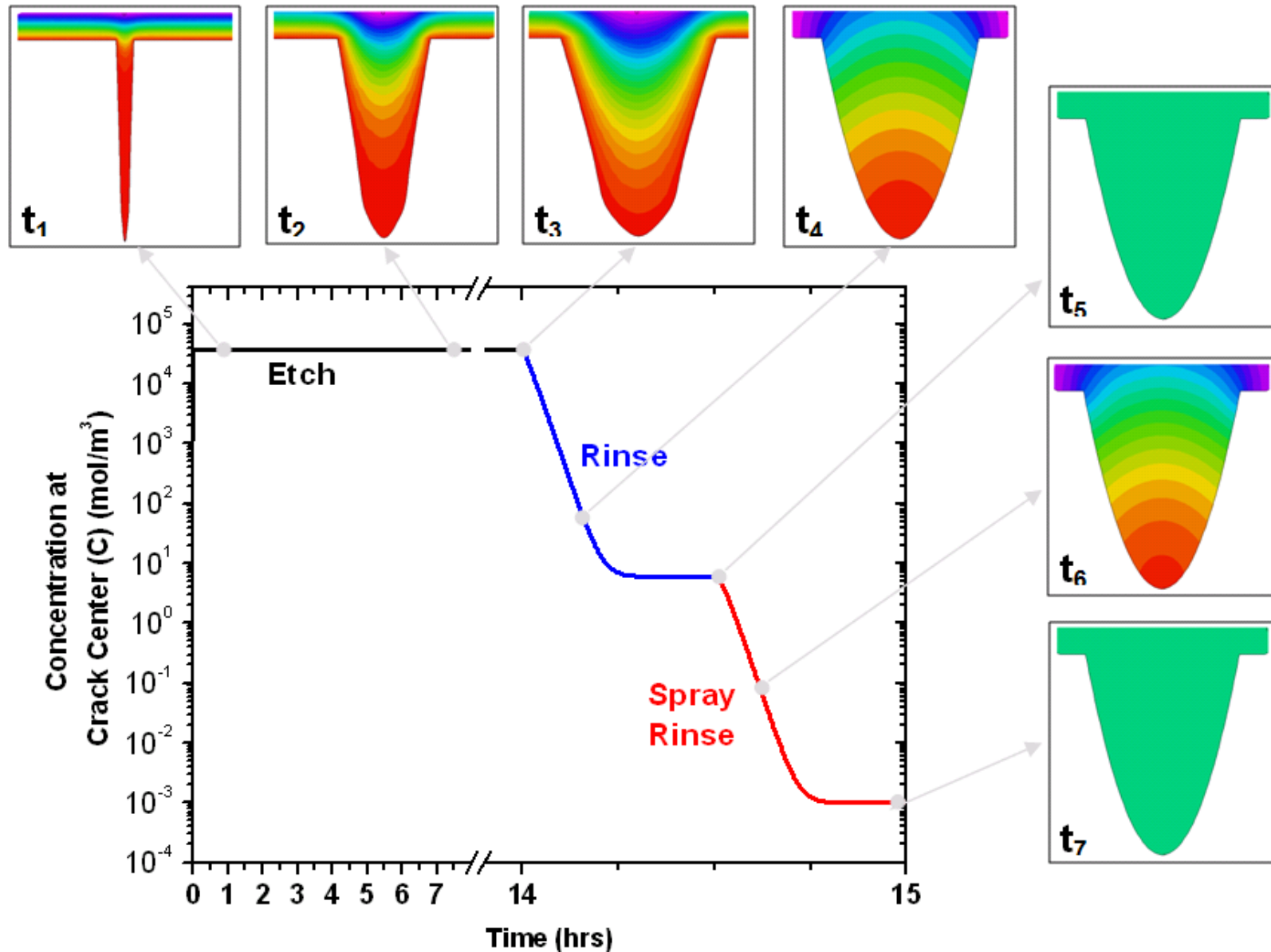
	1. Optical Fabrication	2. Post Processing & Coatings	3. Laser Operation
Current Efforts	<ul style="list-style-type: none"> <li>• Sub-surface damage management</li> <li>• Forensics of surface fractures</li> <li>• Fundamentals of material removal</li> <li>• Technology of full aperture &amp; small tool optical finishing</li> <li>• Low cost, precursor-free finishing techniques</li> </ul>	<ul style="list-style-type: none"> <li>• Development of chemical/thermal-based flaw/damage mitigation</li> <li>• Development of laser-based flaw/damage mitigation</li> <li>• Laser interference gratings development</li> </ul>	<ul style="list-style-type: none"> <li>• Mechanism of initiation &amp; growth (precursors &amp; modulation)</li> <li>• Precursor isolation &amp; identification</li> <li>• Quantitative understanding initiation &amp; growth behavior</li> <li>• Understanding solarization effects</li> <li>• Understanding modulation effects</li> </ul>
Future Challenges	<ul style="list-style-type: none"> <li>• Toward deterministic finishing (away from artisan, iterative finishing)</li> <li>• Science of finishing continued (microscopic, molecular, &amp; chemical interactions)</li> <li>• Development of new finishing techniques</li> </ul>		<ul style="list-style-type: none"> <li>• Higher fluence precursor identification &amp; mitigation</li> <li>• Understand multi-pulse surface &amp; radiation effects</li> <li>• Understand/mitigating debris-induced damage</li> <li>• Understand damage mechanisms on other glass optics (including coatings)</li> <li>• Development of new glass optical materials (e.g., high fluence optical filters)</li> </ul>

# Optimization of etching processes have led to large increases in the damage resistance of scratches



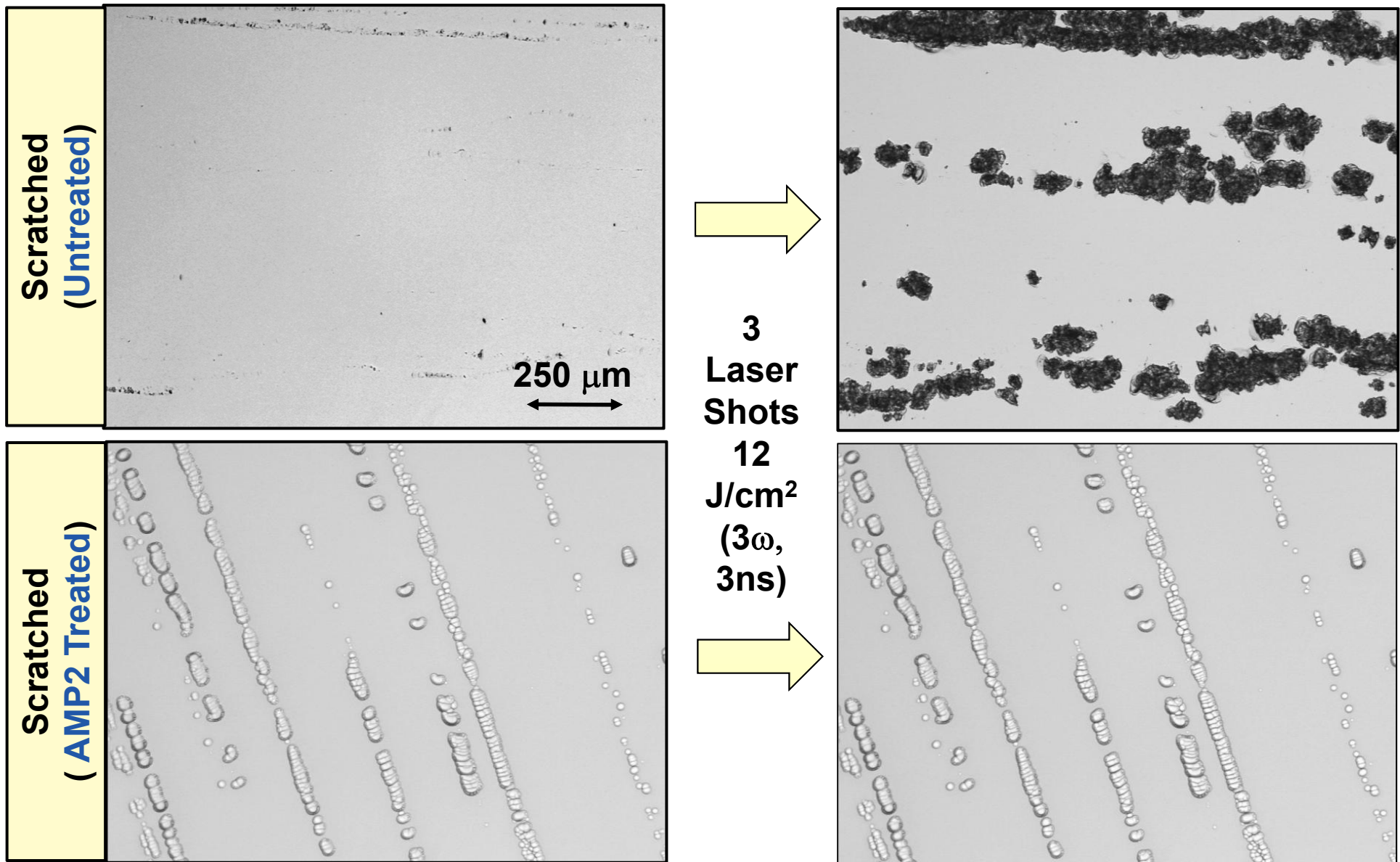
Using a mass transport model, process has been optimized to minimize reaction product concentration left in the crack

### Calculated $\text{SiF}_6^{2-}$ concentration during AMP Process





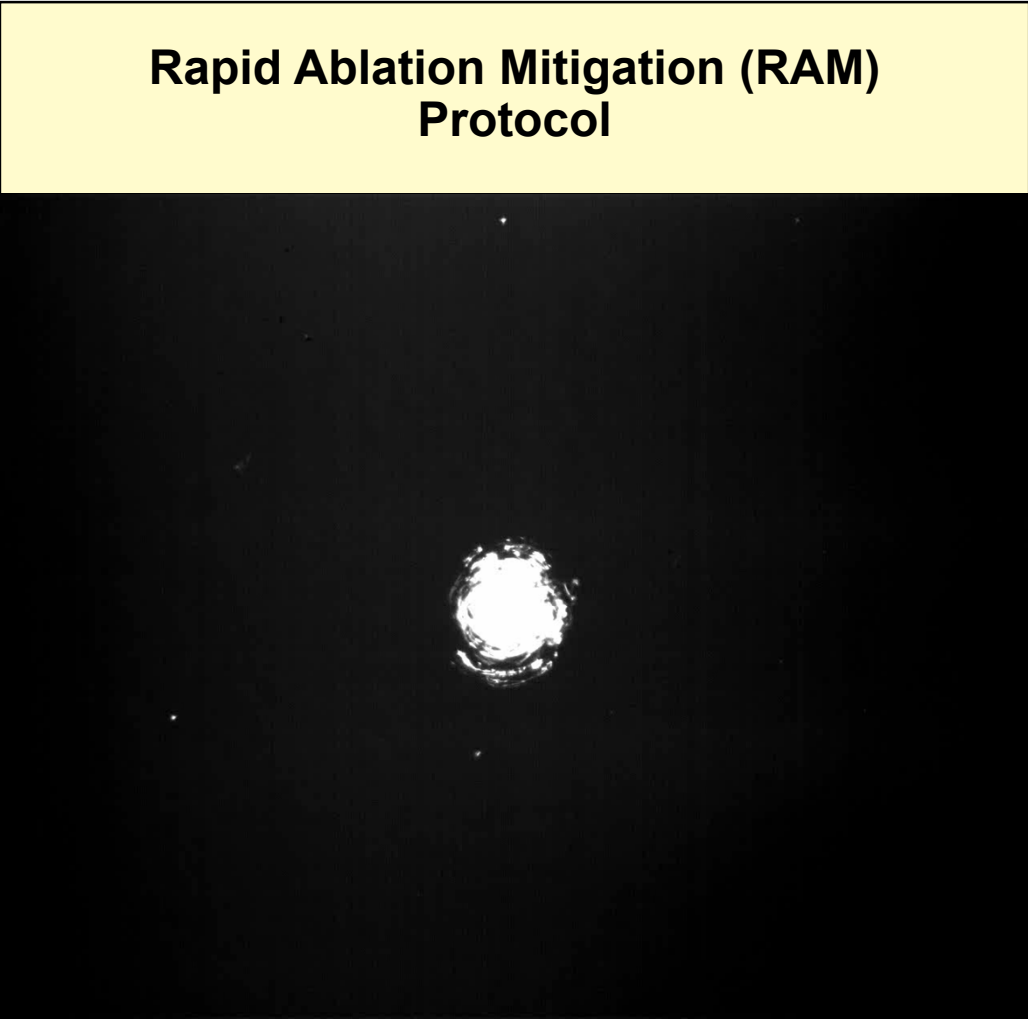
# Using AMP2, scratches as a damage precursor in NIF have been eliminated



# AMP2 in production



# Flaws on fused silica are mitigated with a small-beam CO<sub>2</sub> laser operating at 10.6- $\mu$ m

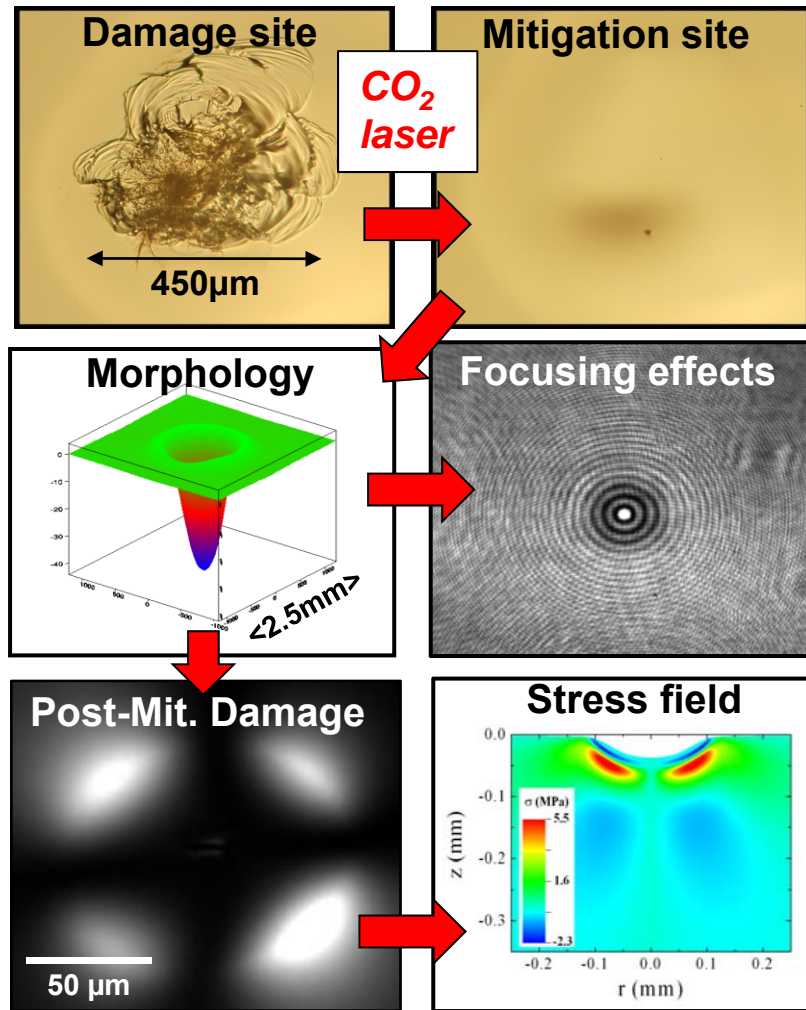


- Utilizes rapid scanning of tightly-focused high-power CO<sub>2</sub> laser pulses to remove flaws up to ~0.5 mm diameter
  - Precise shape control
  - Fairly wide process margin
  - Scalable
  - Damage robust
  
- The cone is the only shape identified that does not lead to downstream intensification



**RAM “cone” protocol on  
fused silica**

# Successful optics damage mitigation can only be achieved through careful balance of coupled, sometimes competing effects



- **UV damage threshold**
  - Remove or re-flow damaged material
  - Free of damage-prone re-deposit
- **Light propagation**
  - Resulting morphology that does not intensify/focus UV light
- **Residual stress & densification**
  - Stress below critical fracture limit
  - Minimally-extended densification

# Our S&T has focused on understanding surface interactions on glass surfaces during fabrication, post processing and laser operation

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# The optics S&T effort is a multi-disciplinary, multi-team effort

PLS		NIF	ENG
<ul style="list-style-type: none"> <li>• D. Aberg</li> <li>• S. Baxamusa</li> <li>• J. Bude</li> <li>• S. Demos</li> <li>• R. Dylla Spears</li> <li>• P. Ehrmann</li> <li>• P. Erhart</li> <li>• S. Elhadj</li> <li>• J. Fair</li> <li>• G. Gilmer</li> <li>• T. Laurence</li> <li>• M. Johnson</li> <li>• M. Matthews</li> <li>• J. Menapace</li> </ul>	<ul style="list-style-type: none"> <li>• P. Miller</li> <li>• M. Monticelli</li> <li>• R. Negres</li> <li>• R. Qiu</li> <li>• R. Raman</li> <li>• B. Sadigh</li> <li>• K. Schaffers</li> <li>• E. Schwegler</li> <li>• R. Steele</li> <li>• C. Stolz</li> <li>• T. Suratwala</li> <li>• L. Wong</li> <li>• J. Wolfe</li> </ul>	<ul style="list-style-type: none"> <li>• J. Adams</li> <li>• I. Bass</li> <li>• W. Carr</li> <li>• D. Cross</li> <li>• R. Desjardin</li> <li>• M. Feit</li> <li>• G. Guss</li> <li>• Z. Liao</li> <li>• K. Manes</li> <li>• M. Norton</li> <li>• M. Nostrand</li> <li>• M. Spaeth</li> <li>• T. Weiland &amp; the OSL Team</li> <li>• P. Wegner</li> <li>• C. Widmayer</li> <li>• S. Yang</li> </ul>	<ul style="list-style-type: none"> <li>• R. Vignes</li> <li>• J. Stolken</li> </ul>

- + Production Facilities (Optic Mitigation Factory, Optics Processing Lab)
- + Engineering Group (Design & Fabrication)
- + Metrology and Coordination Group

**NIF**

