SAND2018-1330PE

Use of Hertzian Cracking to Measure Surface Flaw Densities and Probe Interfacial Adhesion Behavior

Rajan Tandon

Component Science, Engineering and Production Center

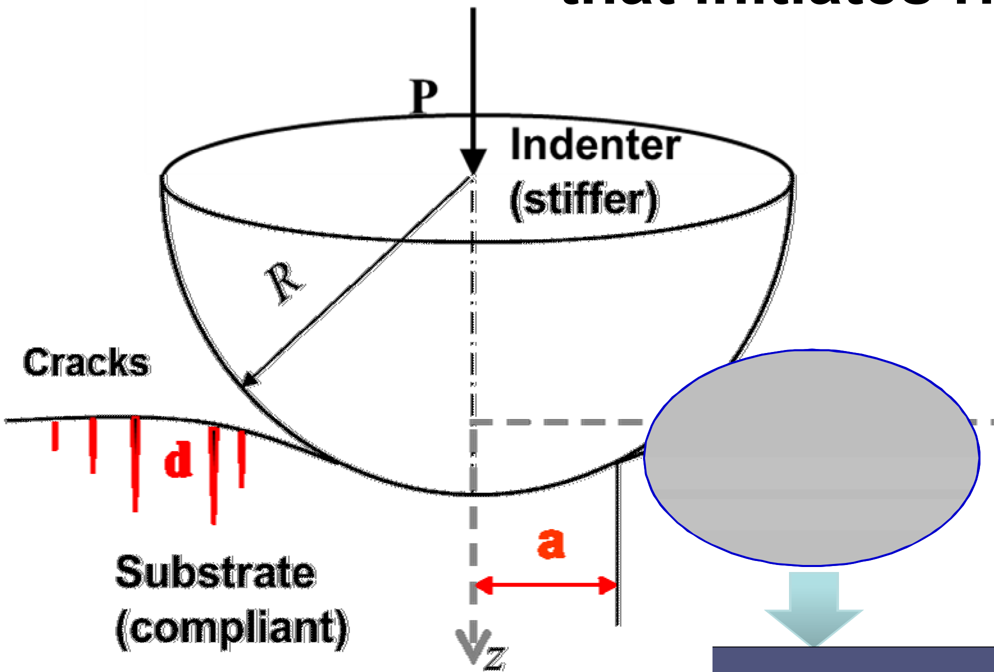
Sandia National Laboratories, Albuquerque

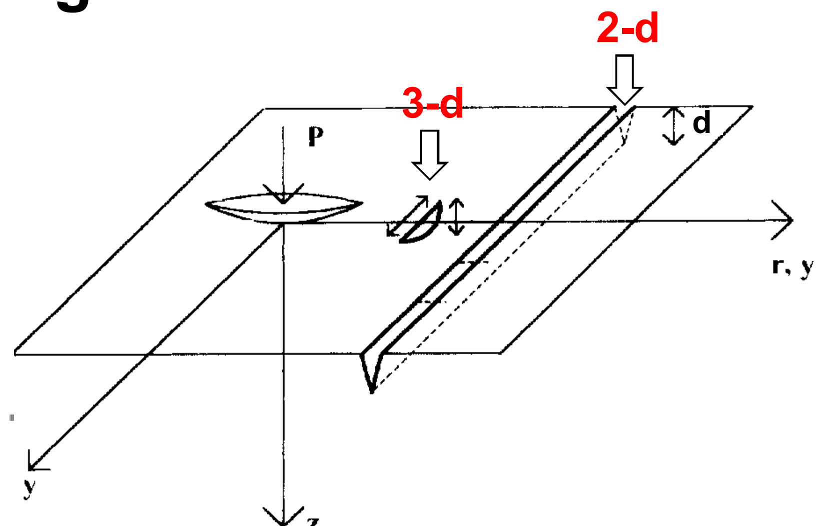
Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc. for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

Sandia National Laboratories

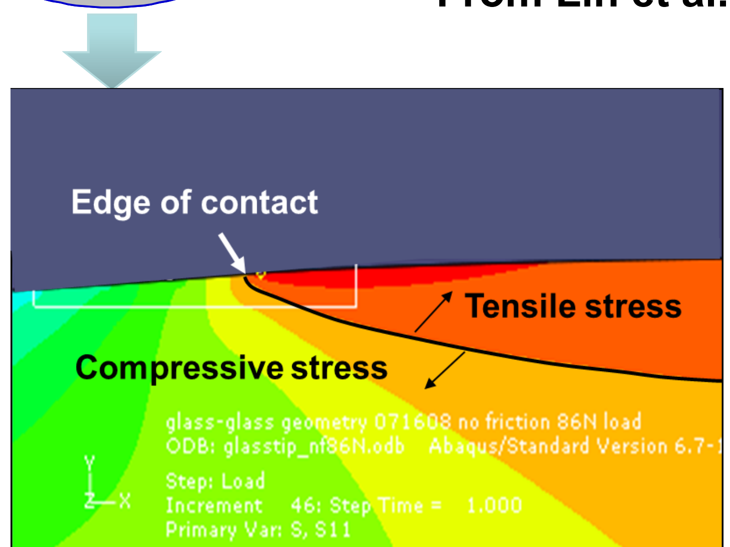
Ring Crack Initiation

Sphere loaded onto a flat surface generates radial tension that initiates ring cracks





From Lin et al., JECS,1998



Motivation

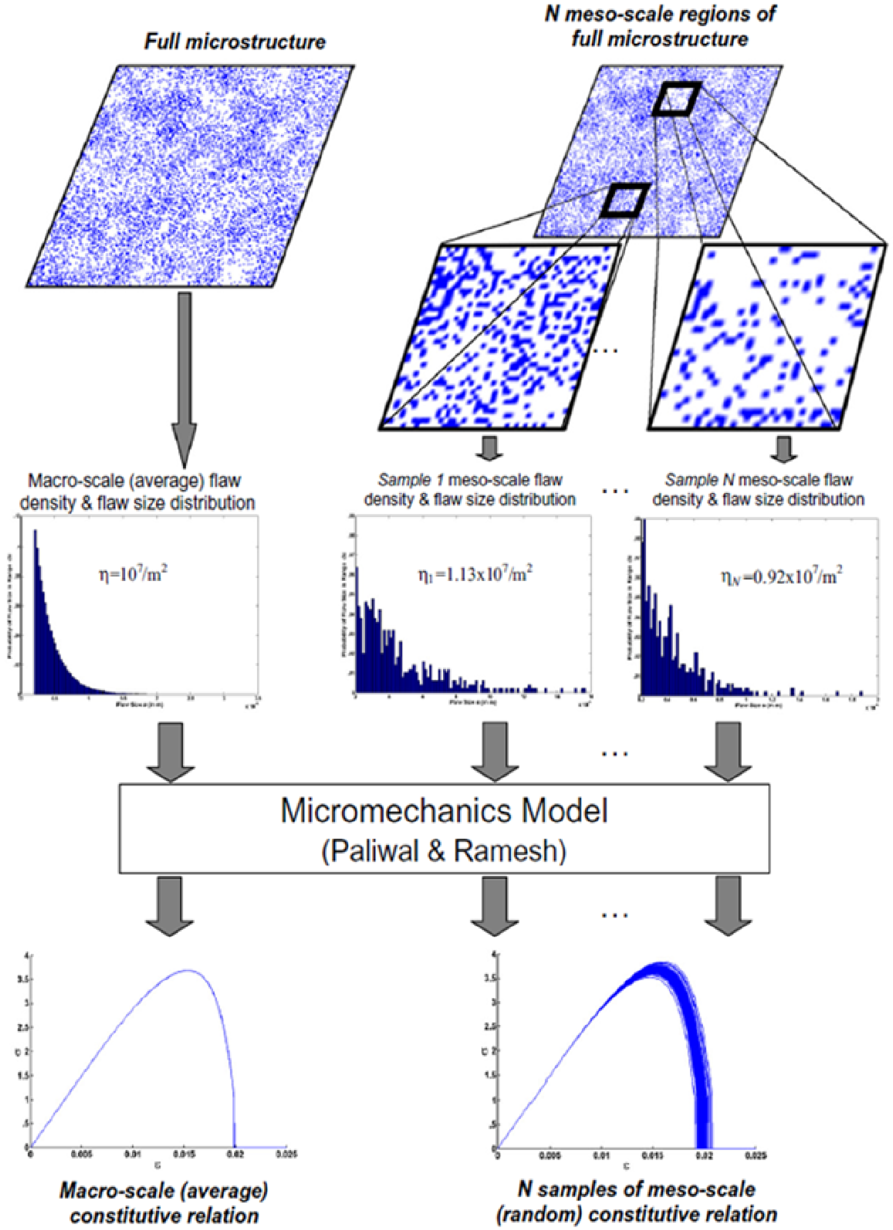
- Conventional strength tests usually measure the largest flaw in a sample.

- Here it is possible to sample the smaller flaws, and maybe even the entire distribution.

Smaller flaws: Crack initiation under intense local loads, assessing surface quality, laser-damage thresholds, models for damage initiation under impact

- Role of friction:	? on how significant-Wang et. al- JMS-2003 -how to use- Jadaan et. al- IJACT-2011
- Role of Humidity:	Largely ignored (Argon, JACS, 1960, Langitan, Lawn, JAP 1971)
- Surface flaw density-	# of flaws in a size range/area- (Wilshaw-JAP-1971)

Damage Propagation Models need Crack Density



Graham-Brady, Int. J. Sol. Struc., 2010

Outline

- Describe stress, stress intensity factors for dissimilar contact (WC-Glass)

- Using experimental and analytical results:

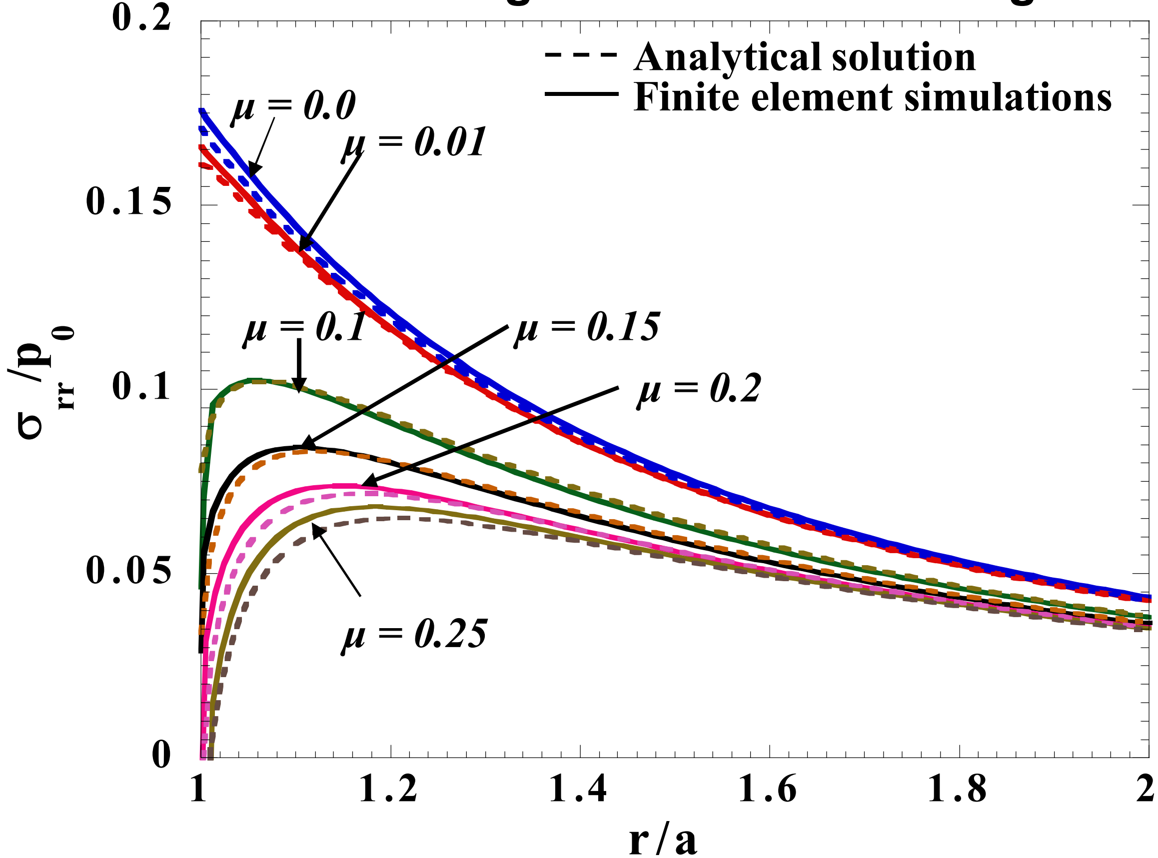
1. Demonstrate the importance of controlling humidity

2. Demonstrate the effect of friction on stress distributions under dissimilar elastic contact

3. Flaw size densities on brittle material surface under similar/dissimilar contacts

Dissimilar Contact with Stiffer Indenter: Surface Stresses

Elastic mismatch creates differential slip
Friction resists this leading to radial traction on glass surface



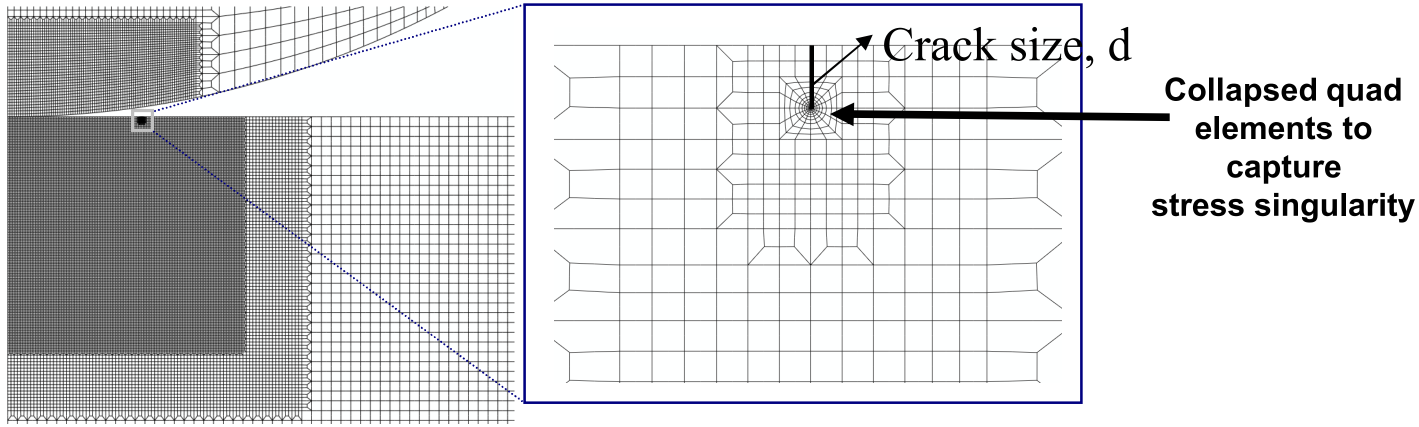
Friction shifts the maximum in the stress farther from contact edge
Friction also leads to reduction in stress

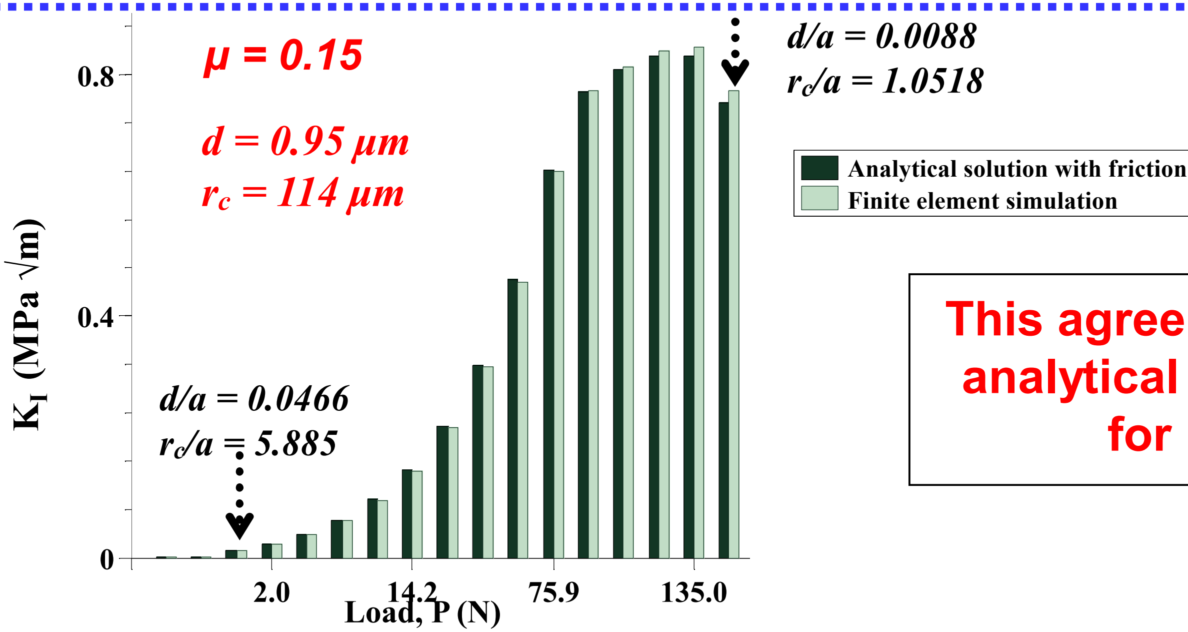
Paliwal B , R Tandon et al., Assessing the Hertzian Indentation Approach for Measuring Fracture Toughness, J. Amer. Ceram. Soc., 2011

Comparison of Analytical and FE Approach to K

Knowing the <u>radial stress distribution in the presence of friction</u>, mode I stress intensity factor can be written as

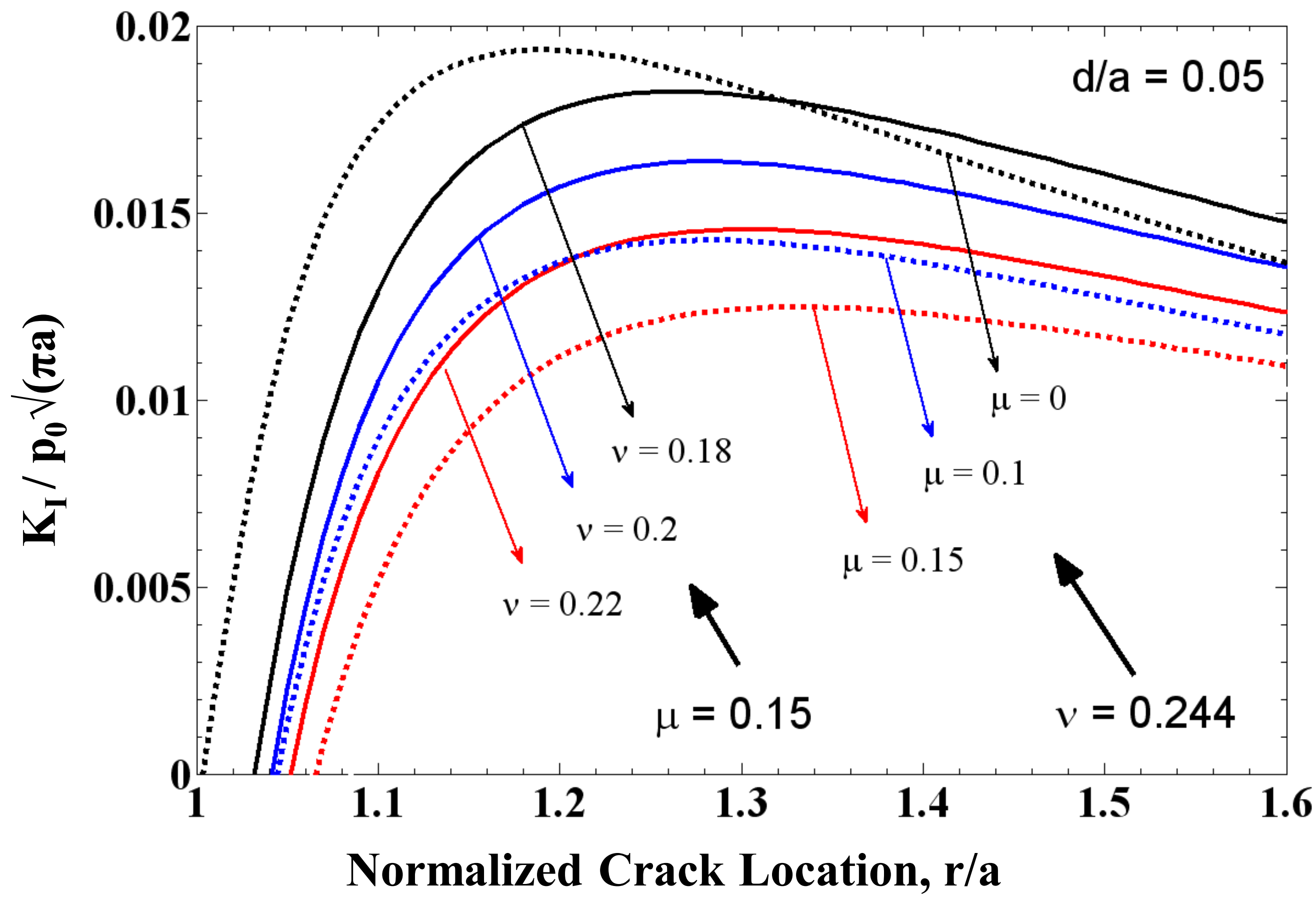
$$\frac{K_I}{p_0\sqrt{\pi a}} = \frac{2}{\pi}\sqrt{\frac{d}{a}}\int_0^{d/a}\frac{\sigma_{rr}(r_c,z)/p_0}{\sqrt{(d/a)^2-(z/a)^2}}\,d(z/a)$$





This agreement allows us to use our analytical formulation, <u>with friction</u>, for parametric studies

K_1 for a particular crack size, d=0.05a



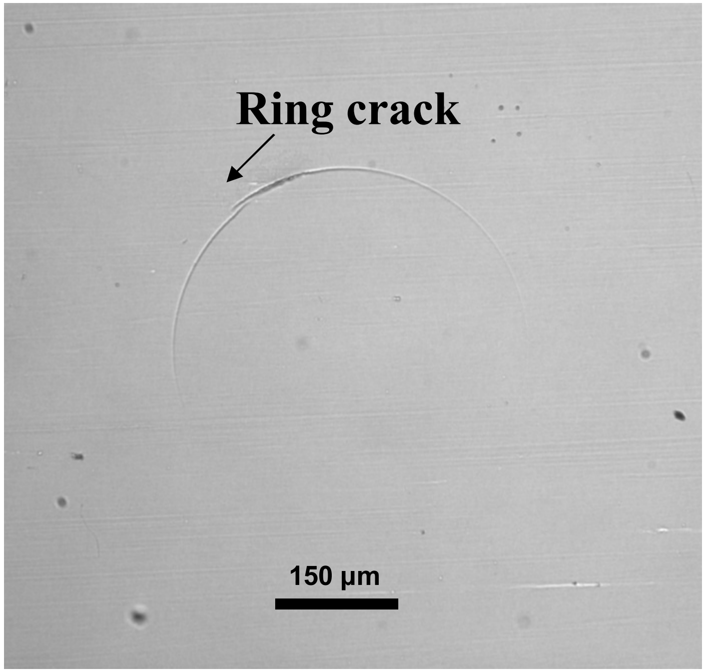
Maximum in K is shifted well away from edge of contact
Values of K are very sensitive to ν and μ

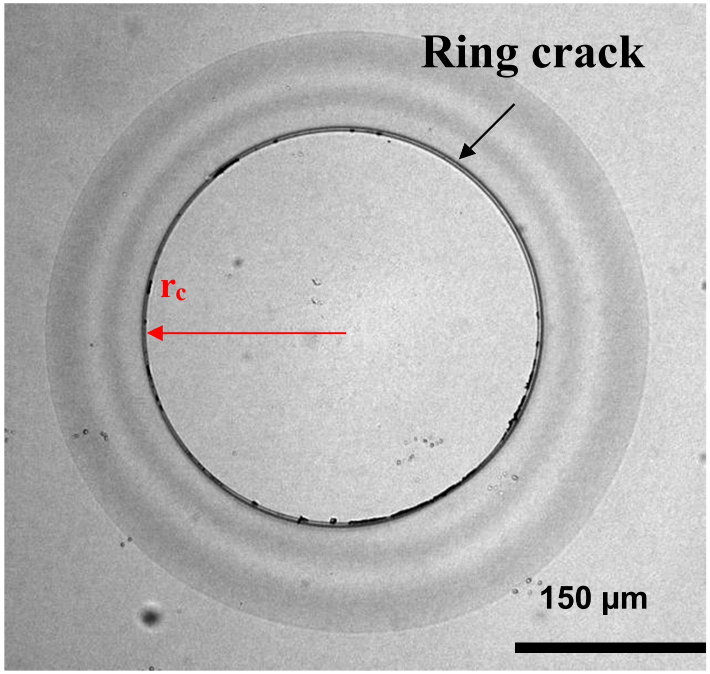
Paliwal B , R Tandon et al., Assessing the Hertzian Indentation Approach for Measuring Fracture Toughness, J. Amer. Ceram. Soc., Volume 94, Issue 5, May 2011

Indentation on SLS Glass

- >500 tests with WC sphere (dissimilar contact), R=0.8, … mm
- >500 tests with Glass sphere (similar contact), R=0.8, … mm
- Controlled RH's, loading rate=0.2 mm/min, 10-15 sec per test

- Measured: Load at cracking by AE
  Friction coefficient of glass/ WC (μ=0.15) [scratch test]
  Cracking locations (r_c) were measured





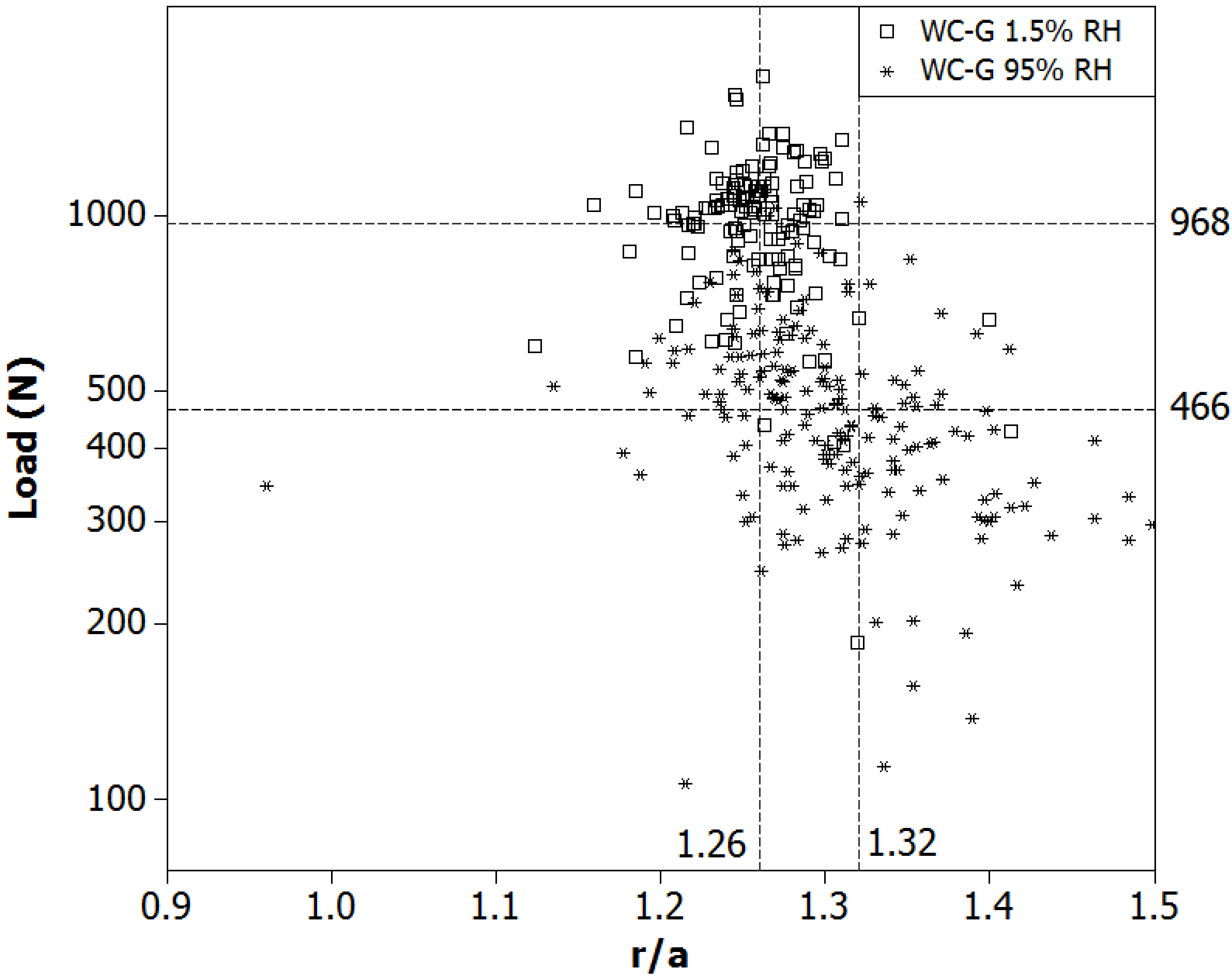
As-received Surface

Sandia National Laboratories

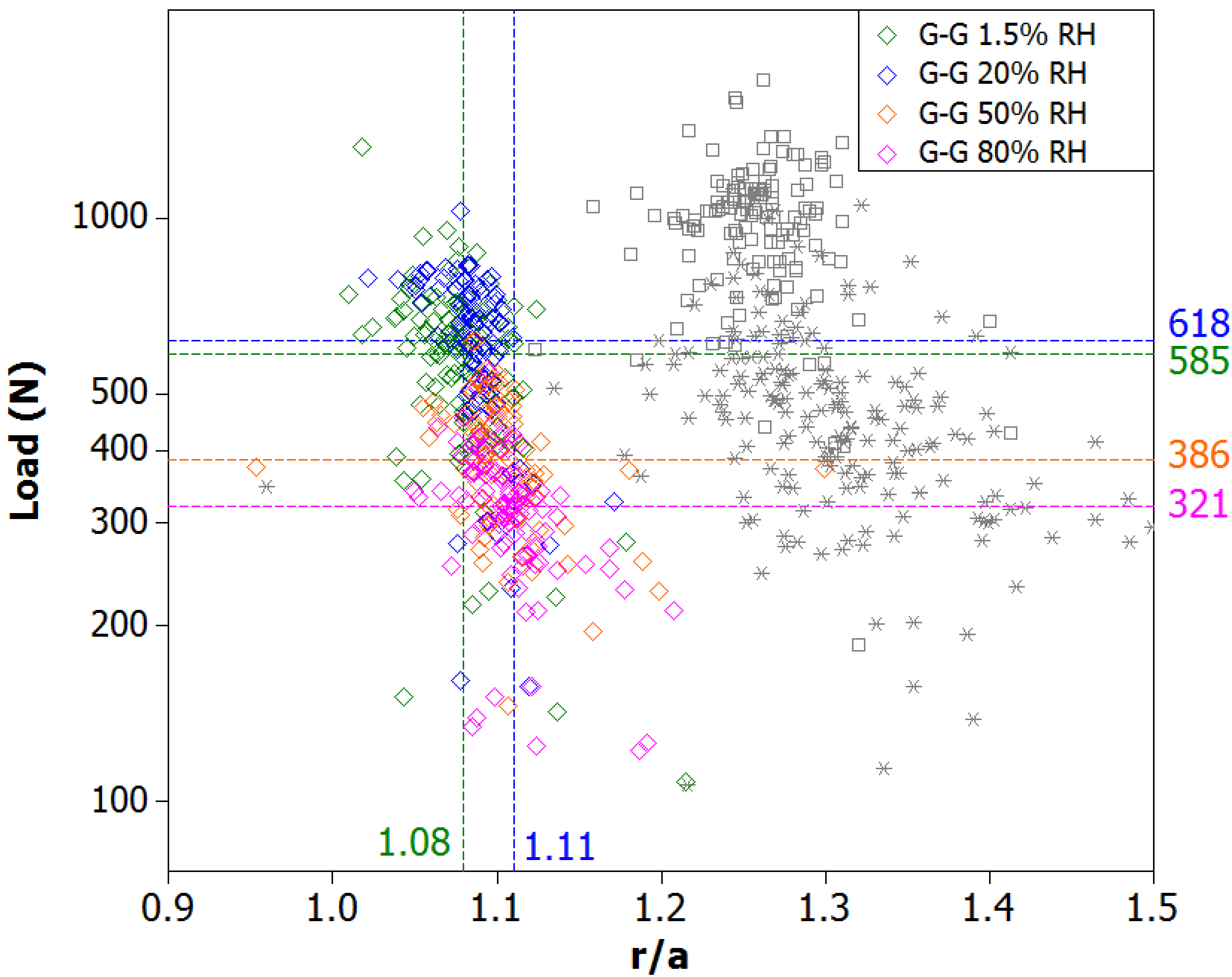
- C

- E

ed



WC-G 1.5% RH
WC-G 95% RH
Load (N)
1000
500
400
300
200
100
968
466
1.26
1.32
0.9
1.0
1.1
1.2
1.3
1.4
1.5
r/a

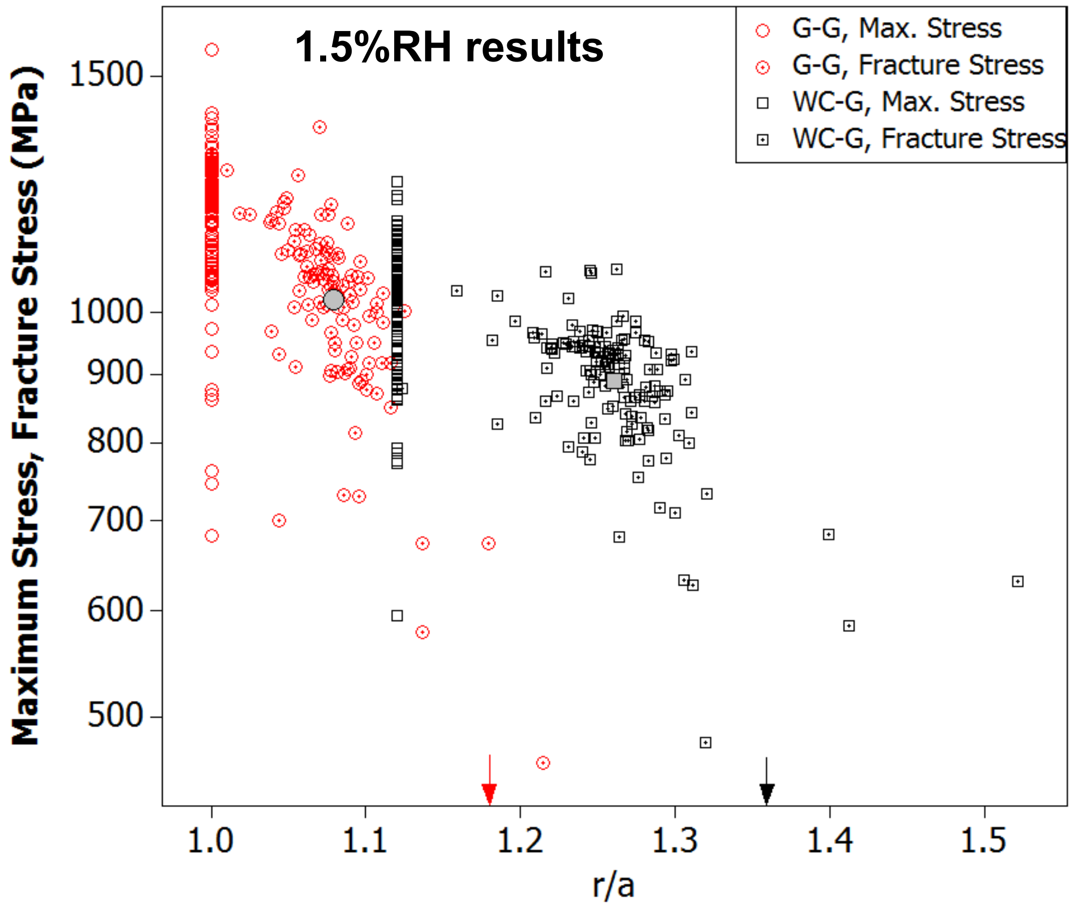


G-G 1.5% RH
G-G 20% RH
G-G 50% RH
G-G 80% RH
Load (N)
1000
500
400
300
200
100
618
585
386
321
1.08
1.11
0.9
1.0
1.1
1.2
1.3
1.4
1.5
r/a

er to

k

Fracture Stresses



Boundary lubrication friction value (μ=0.15) obtained from simple friction tests are used

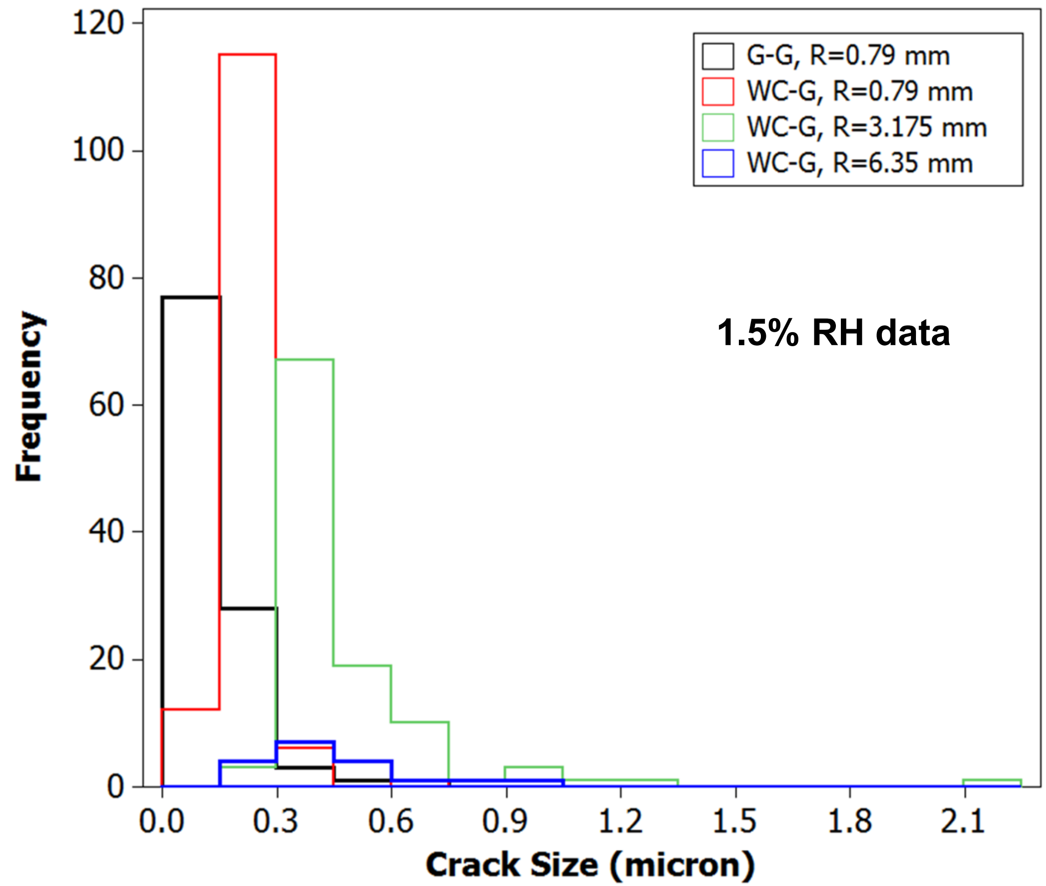
Fair agreement when the stresses at fracture for WC-G, G-G compared.

Calculation of Crack Sizes

$$\sigma_{rr} = \sigma_{surf}\left(1 - \alpha\left(\frac{z}{a}\right)\frac{3\left(r/a\right)^2}{(1-2\nu)}\right)$$

$$\frac{K_1}{\sqrt{\pi a}} = \sigma_{surf}\left[1.12 - \frac{2\alpha}{\pi}\left(\frac{d}{a}\right)\frac{3\left(r/a\right)^2}{(1-2\nu)}\right]\left(\frac{d}{a}\right)^{1/2}$$

(Warren, JECS, 1995)



r = radial coord.
a=contact rad.
d=crack size
z=depth coord.

Cracks are
Small

(d/a<0.03)

These crack sizes are << than those in routine strength tests

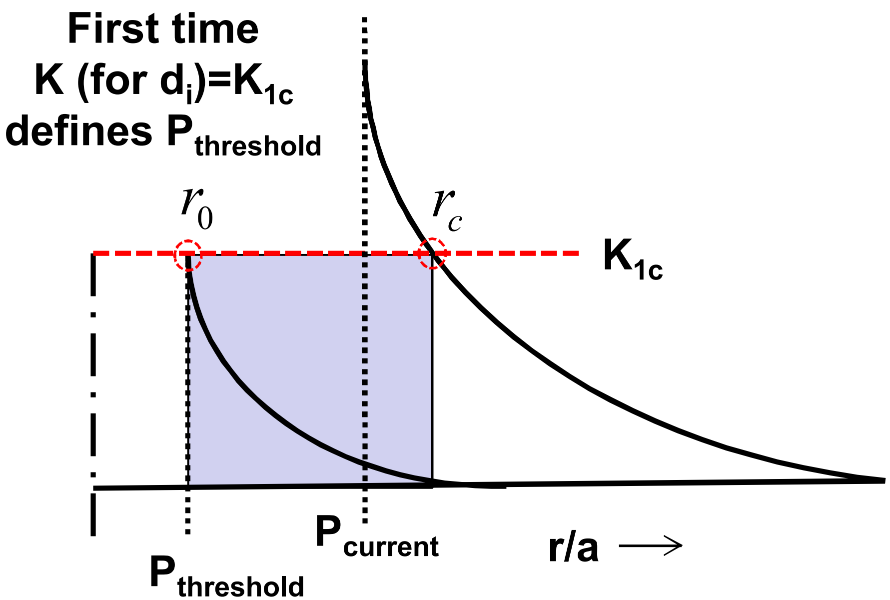
Sandia National Laboratories

"Searched Area" for small cracks (d/a<0.03)

For any particular test, the "area searched" for a crack of depth d_i by loading up to a force *P* is that area of the surface within which, had there existed a crack of depth d_i, it would have caused fracture under some force ≤P.

$$\text{For small cracks, } K_1 = 1.12\sigma_{surf}\left(\pi d_i\right)^{1/2}$$

Frictionless Case



$$\text{Searched area} = \pi\left(r_c^{\,2} - r_0^{\,2}\right)$$

$$P_f = \frac{2\pi^{1/2} K_{Ic}}{1.12\left(1-2\nu\right)} \frac{r^2}{d_i^{1/2}} = \beta \frac{r^2}{d_i^{1/2}}$$

$$r_0^{\,2} = \frac{\beta^2 \alpha_{G-G}^6}{d_i}$$

$$P_{threshold} = \frac{\beta^3 \alpha_{G-G}^6}{d_i^{3/2}}$$

$$r_c^{\,2} = \frac{P_{current}\, d_i^{1/2}}{\beta}$$

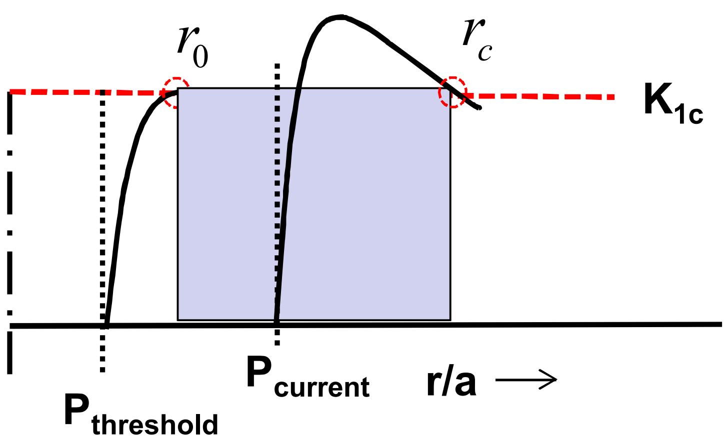
r = radial coord.
a=contact rad.
d=crack size
z=depth coord.

Sandia National Laboratories

"Searched Area" for small cracks (d/a<0.03)

Case with Friction

First time
K (for d_i)=K_{1c}
defines $P_{threshold}$



$$\frac{\sigma_{rr,fric.}}{p_0} = F\left(\frac{r}{a},\mu\right) = \frac{(1-2\upsilon)}{3\left(\frac{r}{a}\right)^2}\frac{1}{\phi\left(\frac{r}{a},\mu\right)}$$

$$P_{f,fric.} = \beta\frac{r^2}{d_i^{1/2}}\phi\left(\frac{r}{a},\mu\right)$$

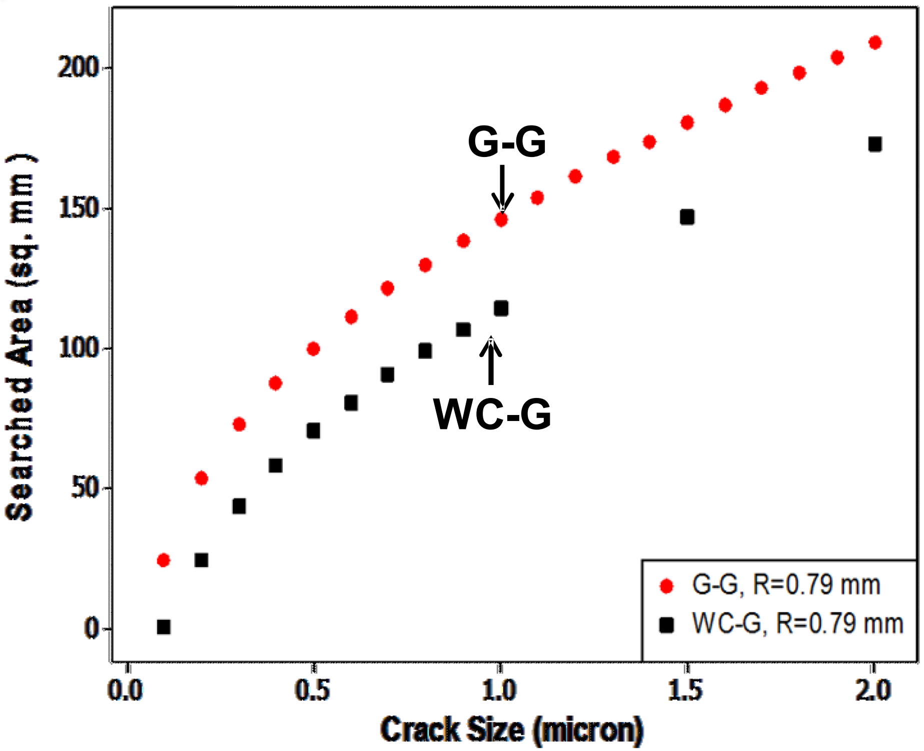
$$r_0^{\,2} = \frac{1.12^6\phi(1.12,0.15)^2\beta^2\alpha_{WC-G}^6}{d_i}$$

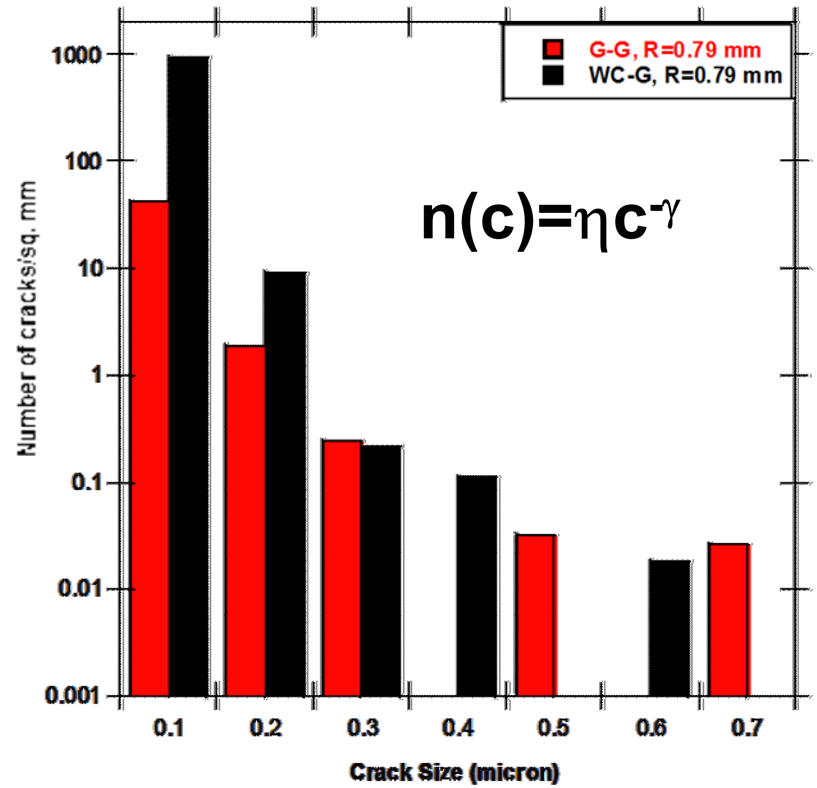
$$r_c^{\,2}\phi\left(\frac{r_c}{a},0.15\right) = \frac{P_{current}d_i^{1/2}}{\beta}$$

$$P_{threshold,fric.} = \frac{1.12^6\phi(1.12,0.15)^3\beta^3\alpha_{WC-G}^6}{d_i^{3/2}}$$

Sum up the area searched from each test for different flaw sizes Two hundred simulations of loads from 10-2000 N, 10 N increments were run. Looking for flaws 0.1-2 micron in size

"Searched Area" and Crack Size Distributions





CSD on this glass surface: $n(c)=\eta c^{-\gamma}$
γ~3.8 for G-G contact, ~6.2 for WC-G contact

If exponential CSD is followed, $m=2\gamma-2$
m~5.6 which is a reasonable value

R Tandon et al., Practical Aspects of Using Ring Crack Initiation to measure Surface Flaw Densities;, Phil. Mag., 2013

Sandia National Laboratories

Conclusions

- Humidity control in Hertzian tests on brittle materials is critical

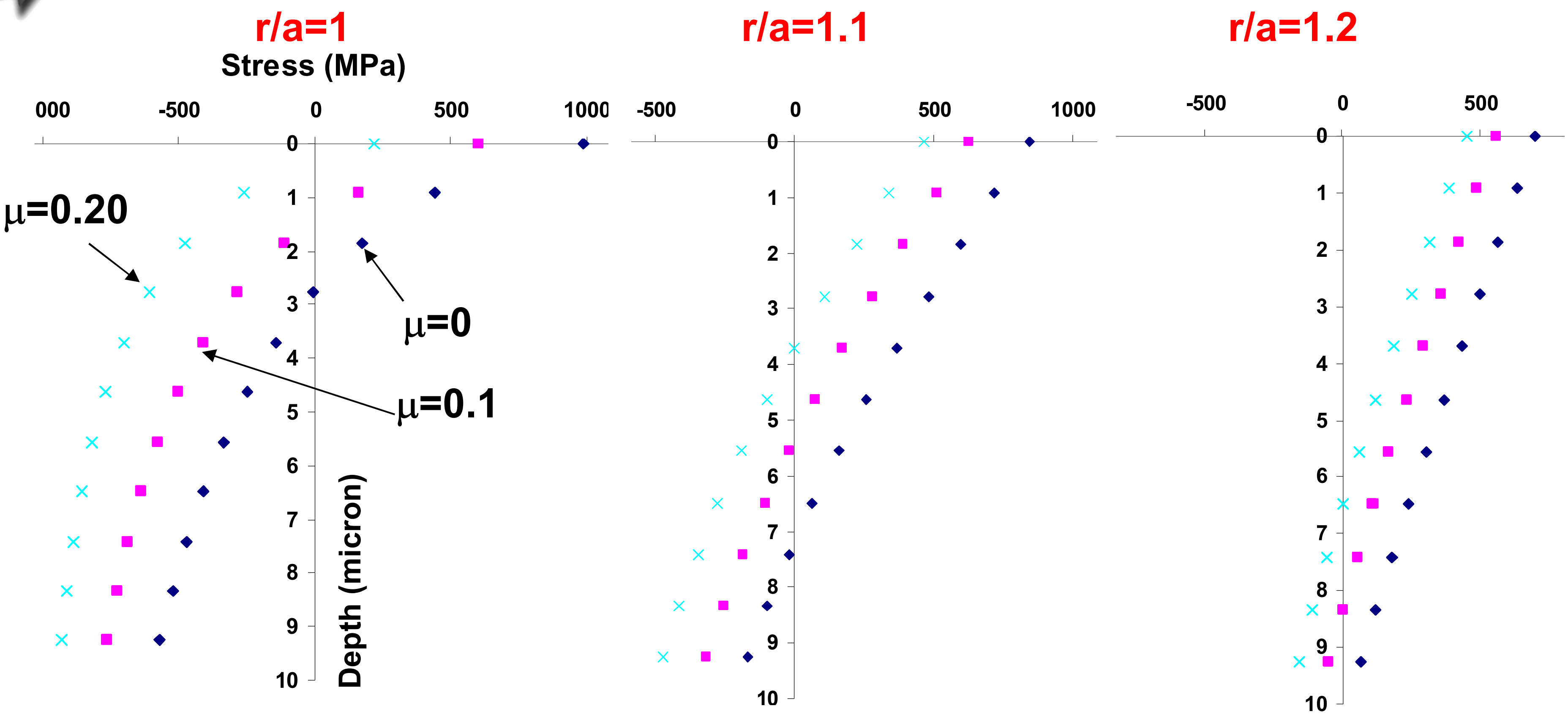
- r/a locations, and higher loads for WC-on-G support the calculated shape, and magnitude of the stress reduction due to friction
    - This effect must be accounted for in meaningful data analyses

- Results from two contacts for crack size distribution are close
    - The approach detects very small flaws/high strengths

- The searched area concept provides reasonable fits to exponential crack size distribution (CSD) on glass surfaces

- CSD can be related to Weibull moduli measured in bulk strength tests

Calculated sub-surface stress distributions



Subsurface distribution are parallel to each other for various r/a values.
Therefore, the effect of friction can be captured by the lowered surface stress and the Hertzian sub-surface drop-off in stress (no slope change)
The drop-offs are ~linear for r/a≥1.1

Sandia National Laboratories

Analytical Approach for Radial Stress in Dissimilar Elastic Contact

Radial stress in non-friction Hertzian solution is:

$$\sigma_{rr}^{p}(r_a, z_a) = \left[\frac{(1-2\nu)}{3r_a^2} \left\{ 1 - \left(\frac{z_a}{\sqrt{u}} \right)^3 \right\} + \frac{z_a}{\sqrt{u}} \left\{ (1+\nu)\sqrt{u} \tan^{-1} \frac{1}{\sqrt{u}} + (1-\nu) \frac{u}{1+u} + \frac{z_a^2}{u^2 + z_a^2} - 2 \right\} \right] p_0$$

It is modified due to friction [7, 8]

(a) Calculating stick-slip boundary in terms of elastic parameters & friction
(b) Calculating the surface shear tractions via the function

$$q(r_a) = \mu p_0 \, \mathrm{sgn}(\beta) \left[\sqrt{1-r_a^2} - r \int_{r_a}^{c_a} \frac{\chi(x, c_a)}{x^2 \sqrt{1-x^2}} dx \right]$$

(c) The modifying stress in terms of the potential function, ψ, is

$$\sigma_{rr}^{q} = 2\psi_{rr} + 2\nu \frac{\psi_r}{\bar{r}} + z_a \psi_{rrz}$$

and can be calculated using the boundary values for the problem.

7. Spence DA. The Hertz contact problem with finite friction. J. Elasticity 1975; 5:297.
8. Hills DA, Sackfield A. The stress field induced by normal contact between dissimilar spheres. J. Appl. Mech. 1987;54:8.

Sandia National Laboratories

Measurement of Adhesion in Alumina-Epoxy System Using Spherical Indentation

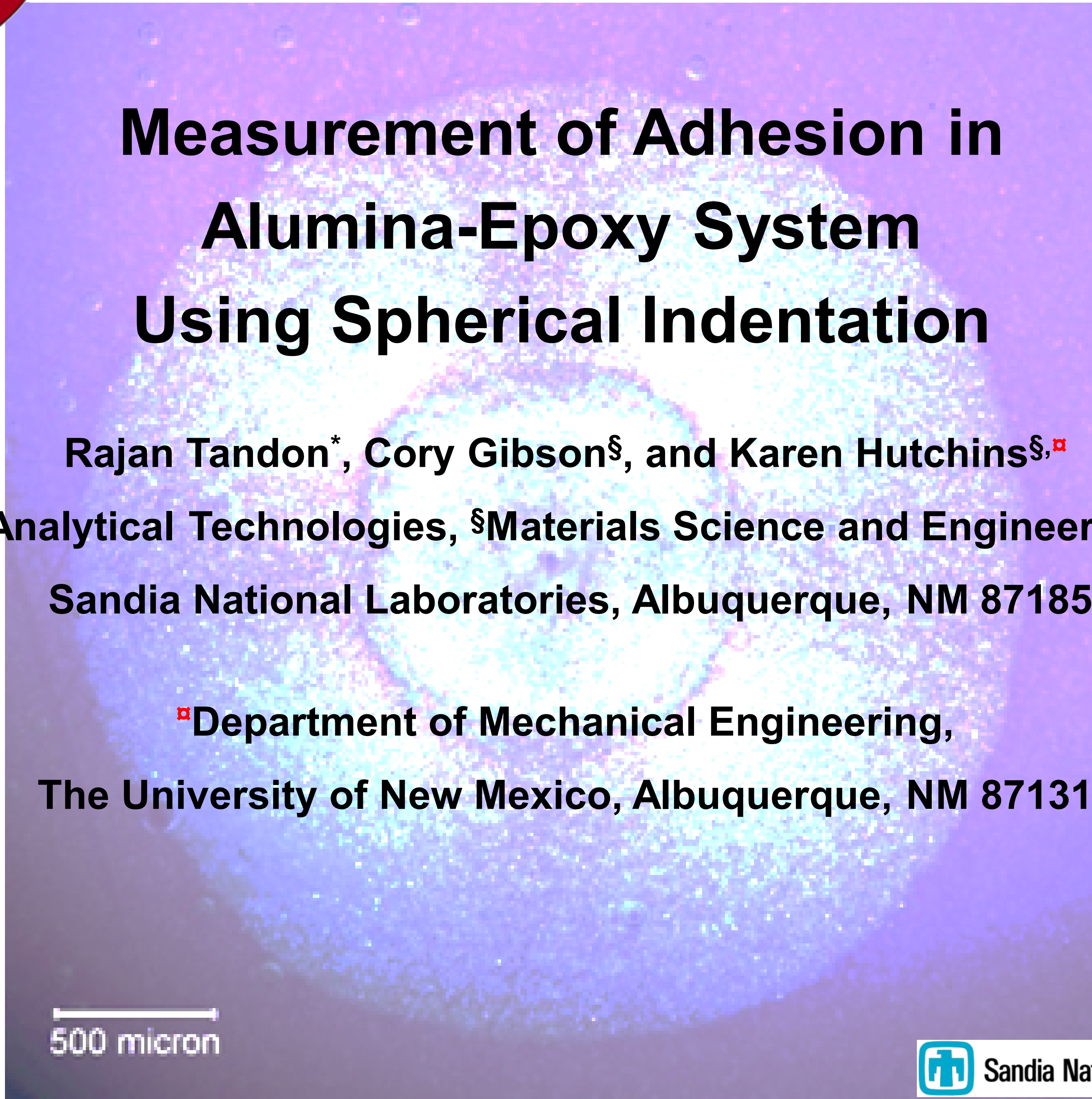
Rajan Tandon[*], Cory Gibson[§], and Karen Hutchins[§,¤]

[*]Analytical Technologies, [§]Materials Science and Engineering

Sandia National Laboratories, Albuquerque, NM 87185

[¤]Department of Mechanical Engineering,

The University of New Mexico, Albuquerque, NM 87131



Sandia National Laboratories

Outline

- Test Method and Mode-mixity

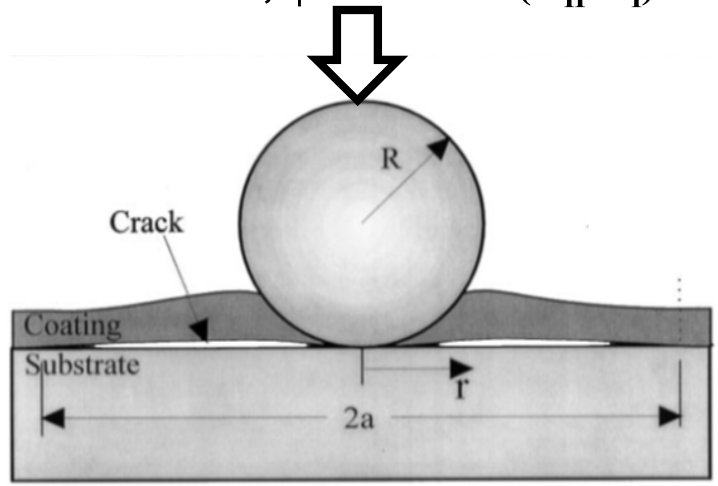
- Initial Room Temperature Results: Alumina-epoxy

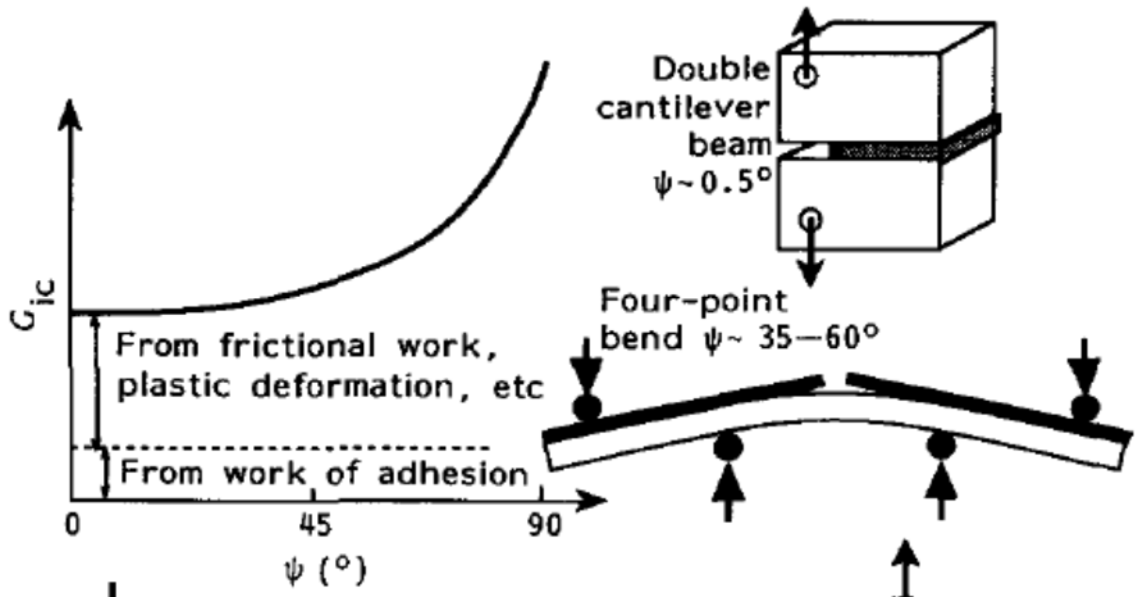
- Stress Distributions and Cold Temperature Results
    - Alumina (temperature, load, and cleanliness)

- ADCB Tests, and preliminary results of simulation of indentation process

Characterization of interfacial adhesion

Mode-mix, ψ ~45°=atan(K_{II}/K_I)

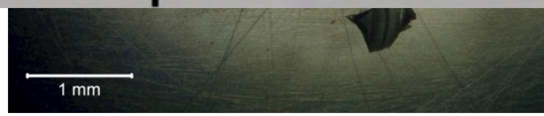


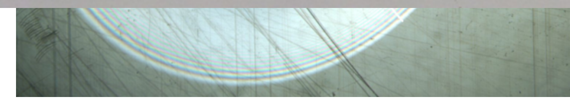


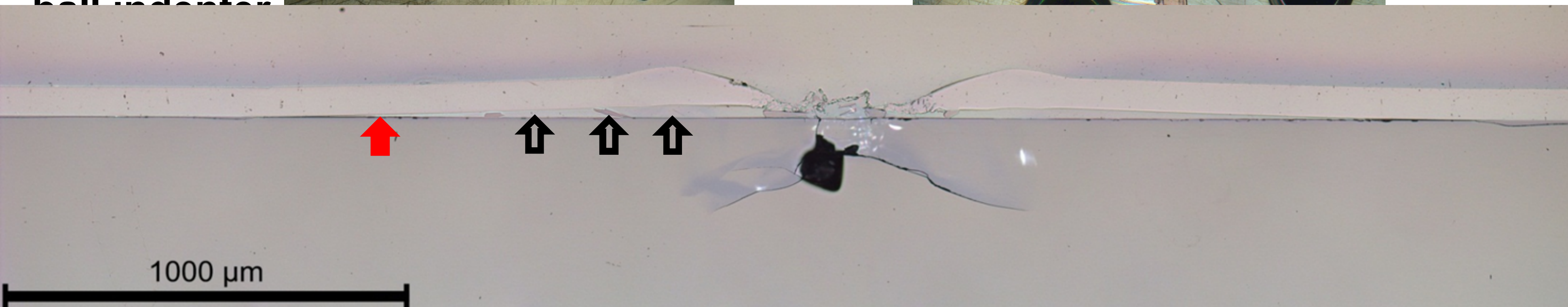
Focus on indentation work as both interfacial adhesion, and shear strength can be obtained

Room Temperature Results

1/8” dia. WC

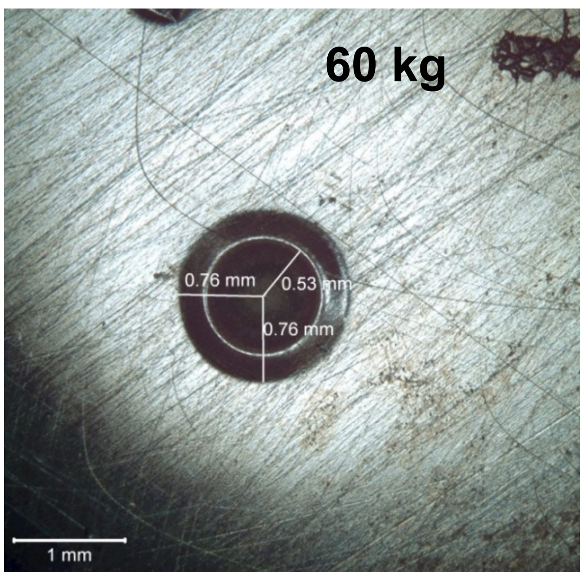


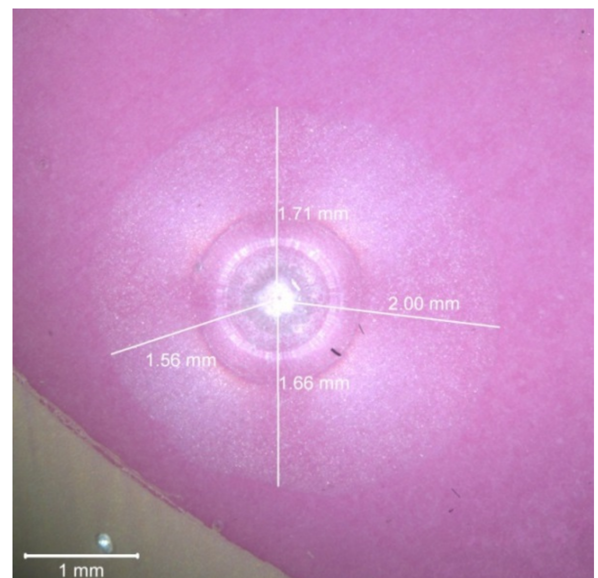




observed.

A 1/8” diameter spherical WC indenter used

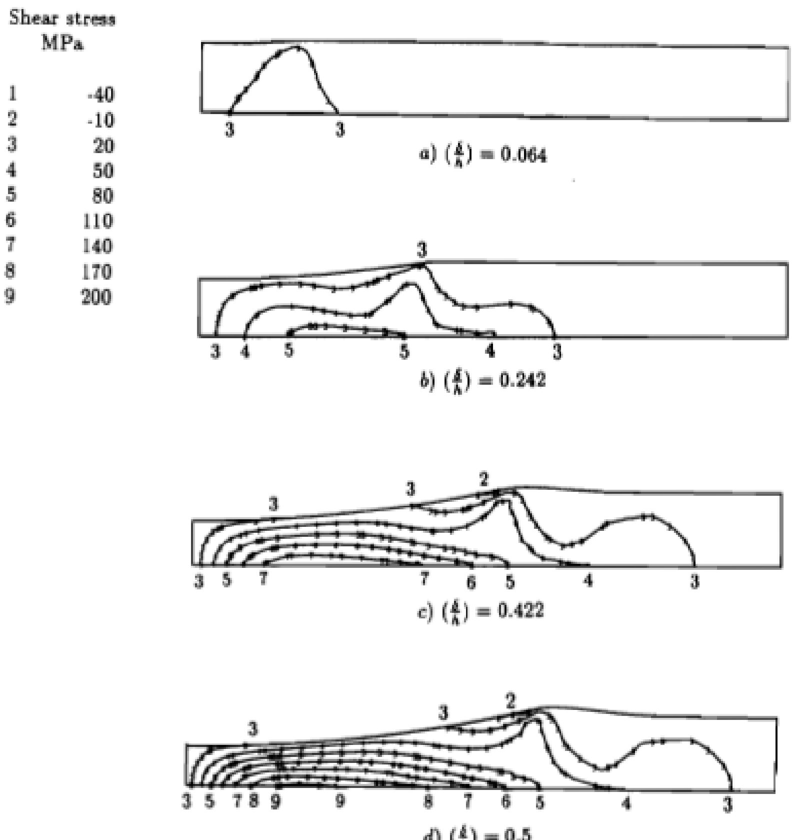


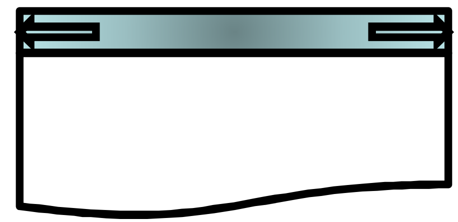


Coating thickness ~100 μm (?)

Key experimental insight-use of sub-ambient

Shear stress contours-PMMA on rigid substrate (elastic-plastic behavior for PMMA)- Argon et. al., J. Ad. Sci.& Tech.





$\alpha_{Epoxy} >> \alpha_{Alumina}$

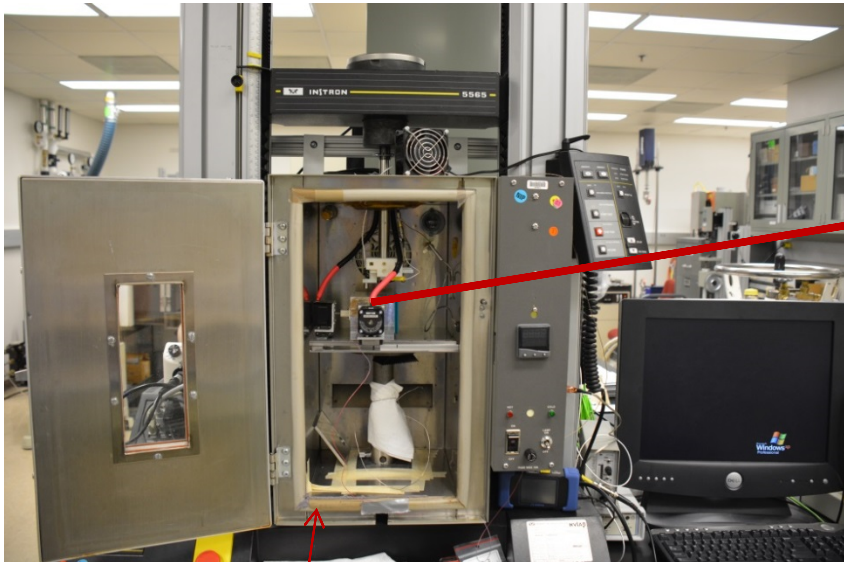
Epoxy cured at ~ 70^{o}C

Epoxy wants to contract much more than alumina, and hence at sub-ambient a large tensile stress exists in the polymer layer.

This adds to the shear stress induced by indentation itself.

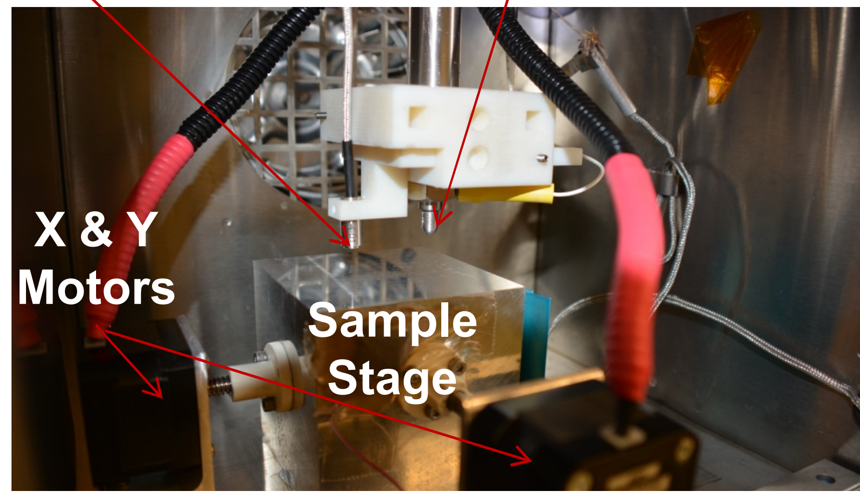
- Hybrid method

Experimental Setup



1/16" WC Spherical Indenter

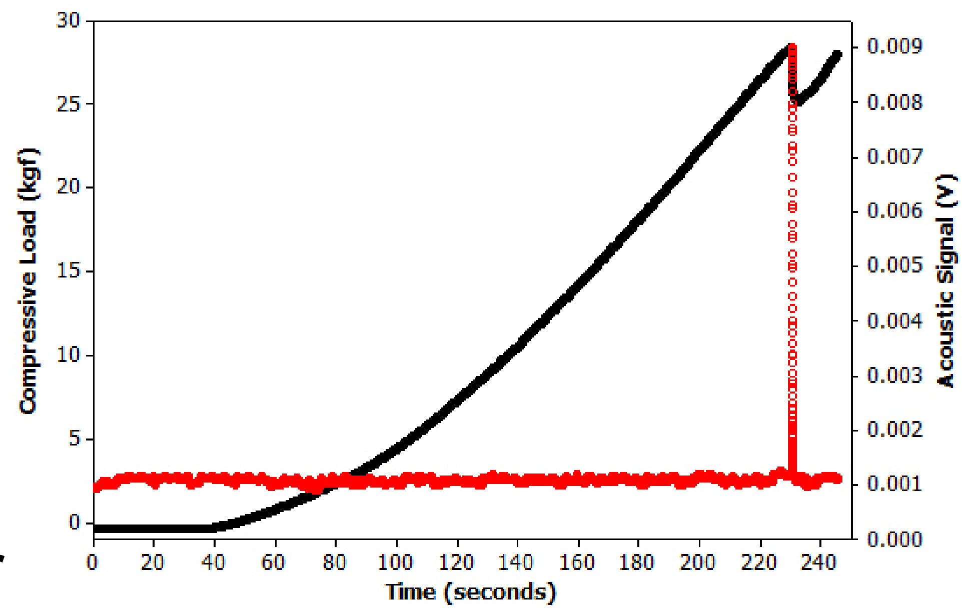
Capacitive Gauge



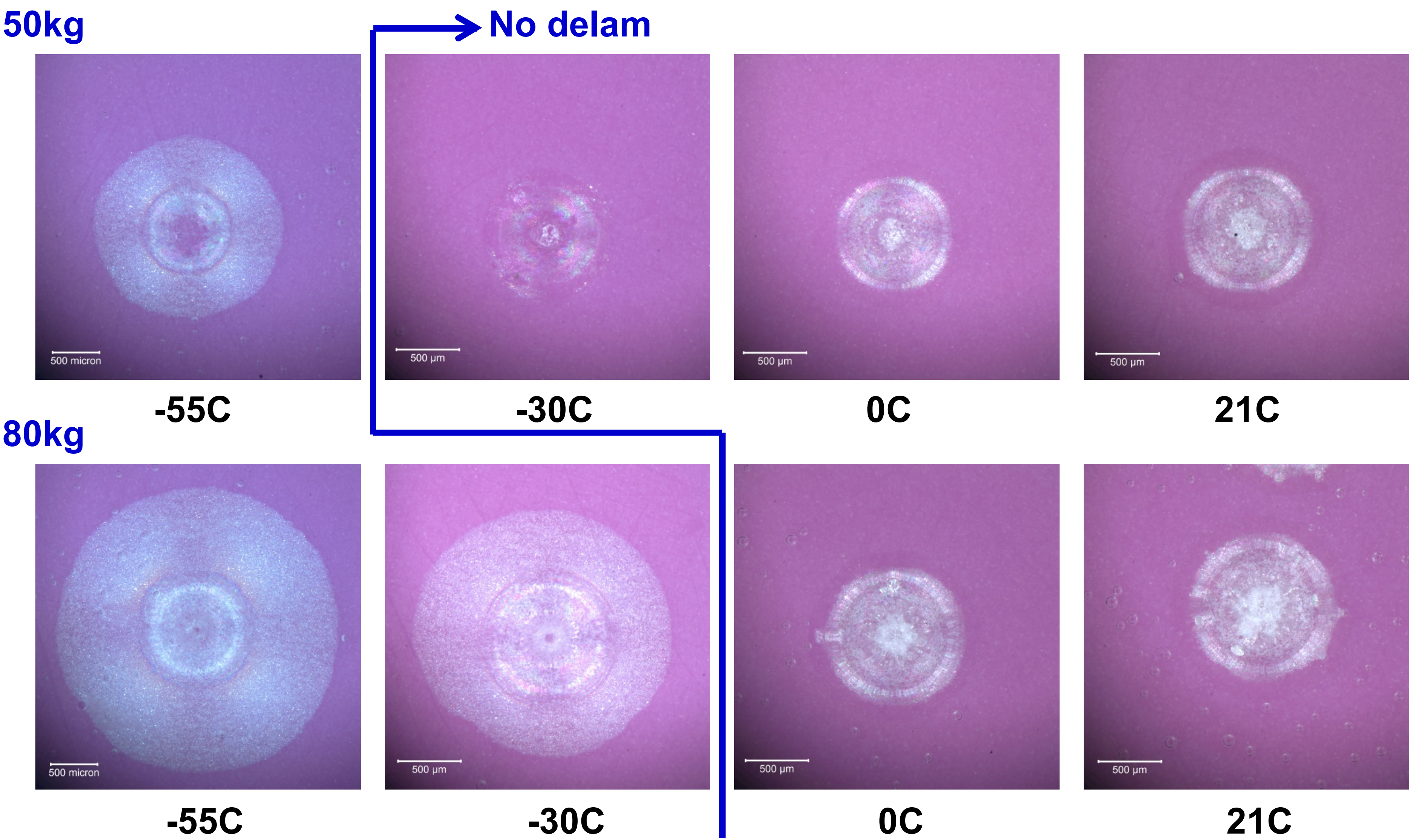
Environmental Chamber (LN_2 chilled)

- Indenter attached to load cell bolts to bottom of crosshead.
- Crosshead rate 0.05 mm/min.

- Computer records load and displacement every 2 ms
- Acoustic signals are monitored by sensors attached to indenter



Alumina Sample – Temp./load effects



Increased delam size: Lower temperature and increase load

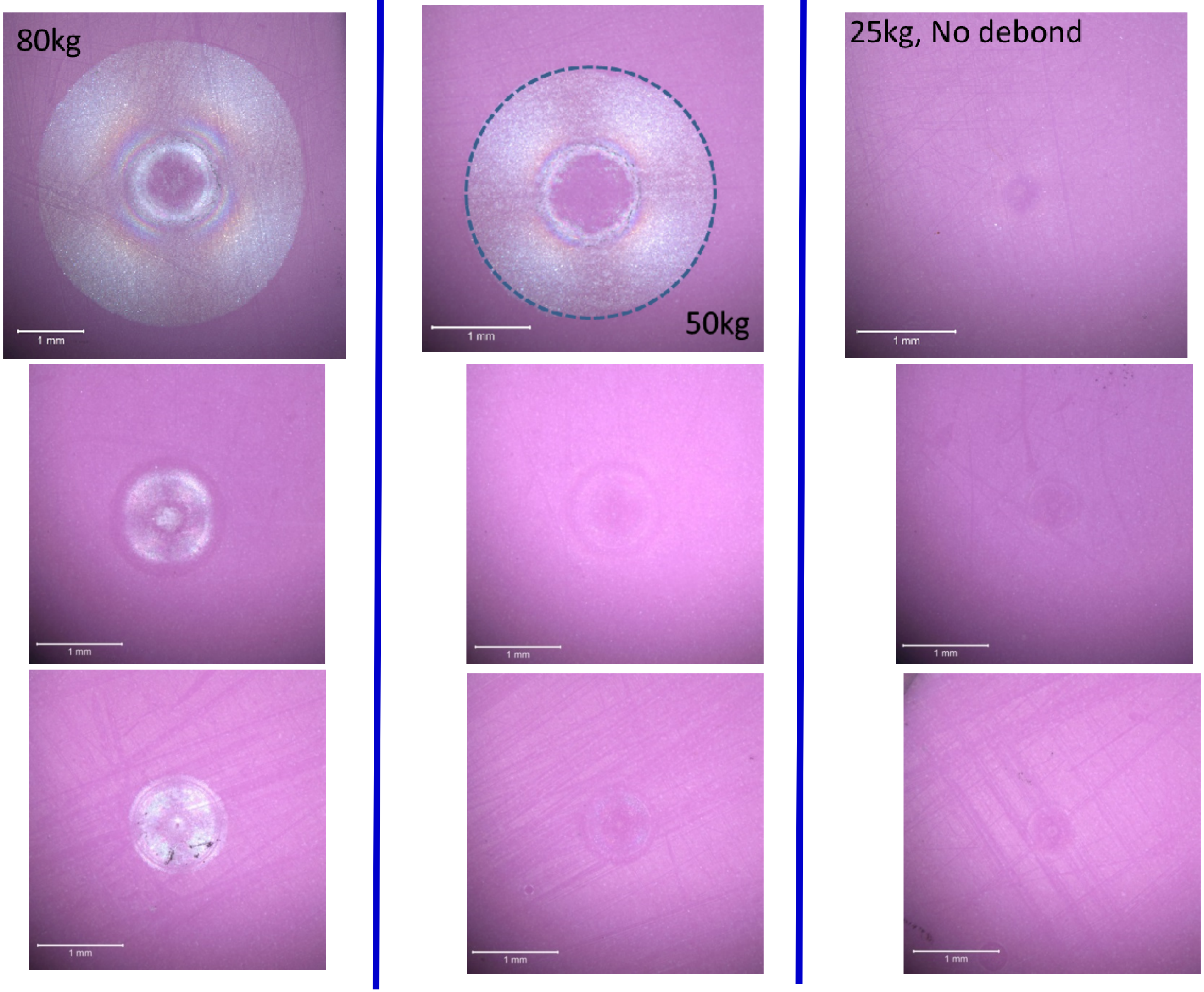
Alumina surface modification

Control

Test at -55°C

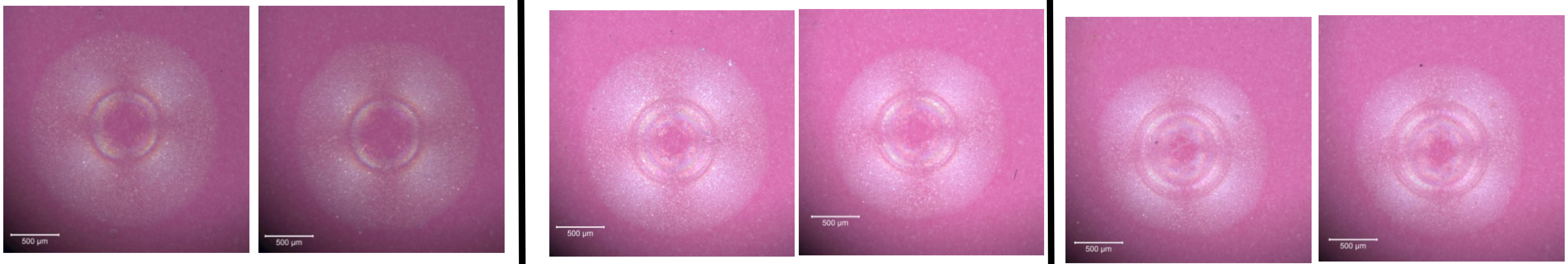
Surface Mod. 1

Surface Mod. 2



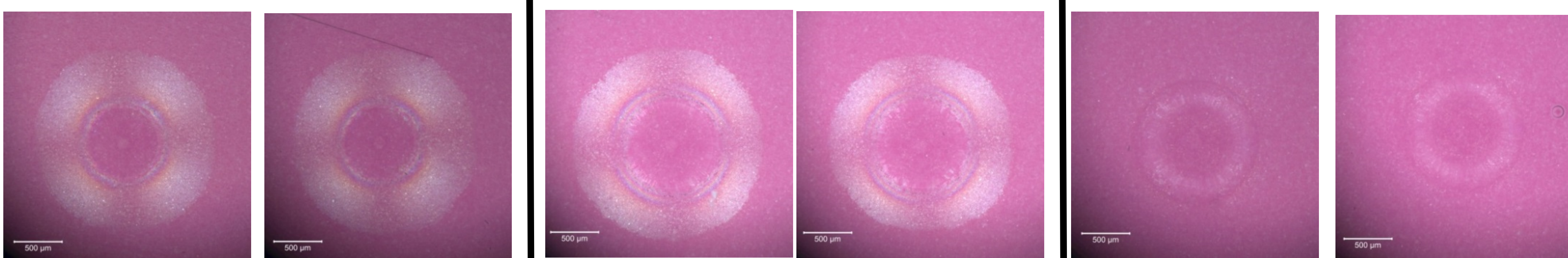
Contaminated vs. Clean surfaces

Contaminated **25 kg, -30°C** | **0°C** | **Room temperature**

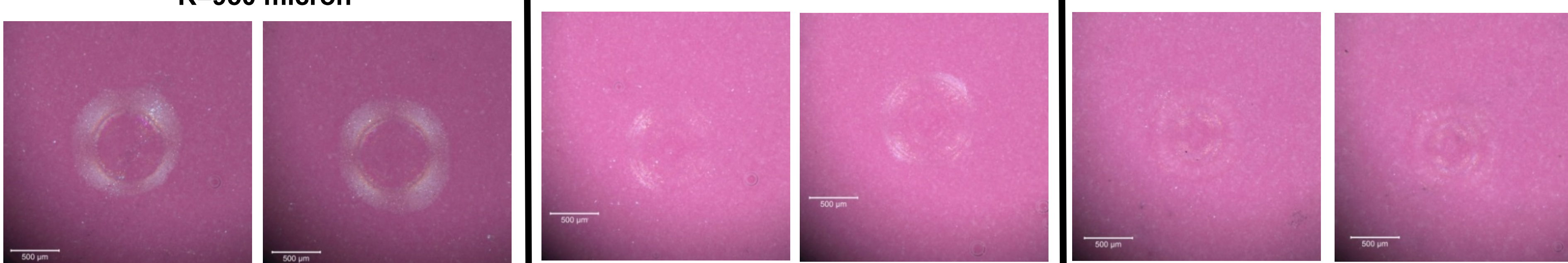


R=1000 micron, 880 micron

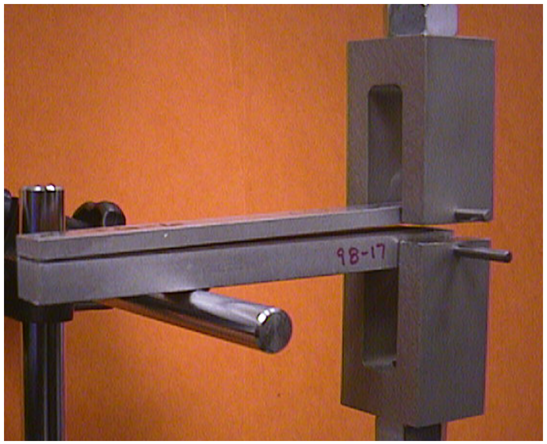
Identical Cleans (?)

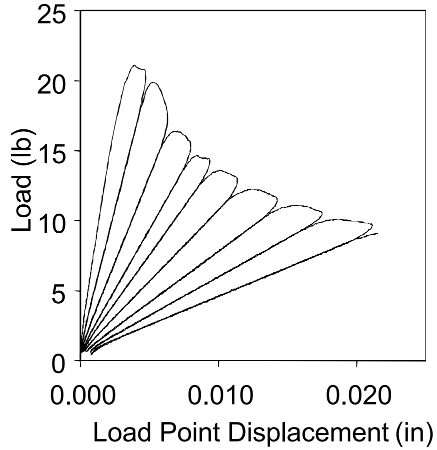


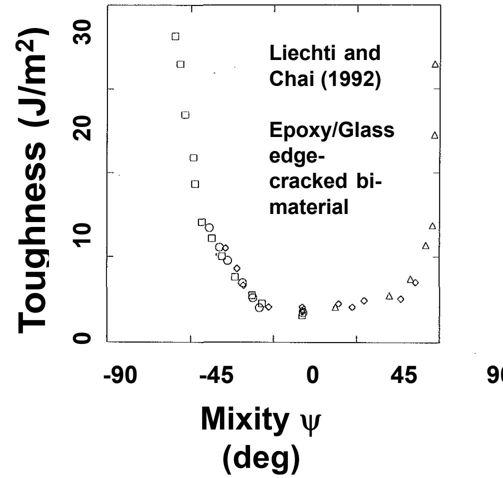
R=950 micron



Asymmetric Double Cantilevered Beam Sandwich (ADCBS) Specimen







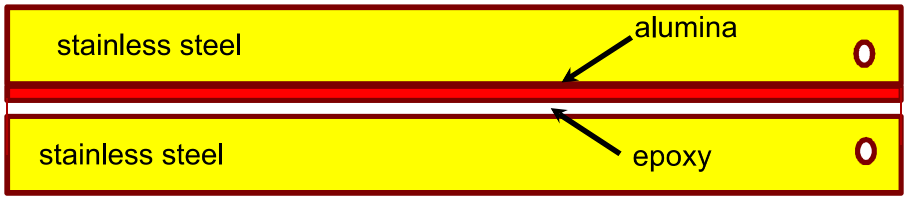
•Can make multiple Γ measurements per specimen (crack propagates stably).

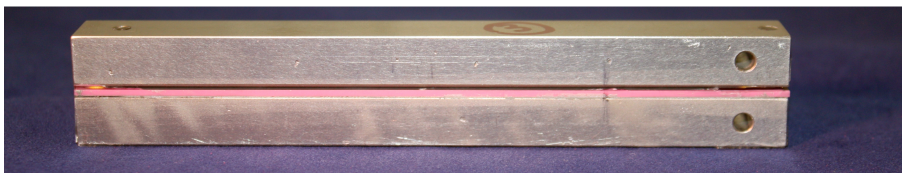
•Use unloading compliance to determine crack length.

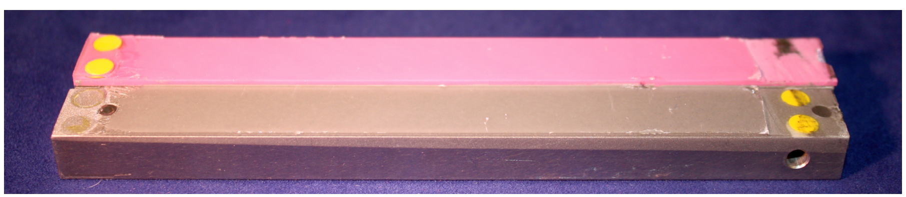
•Measured interfacial toughness Γ at a low ratio of crack tip shear to opening.

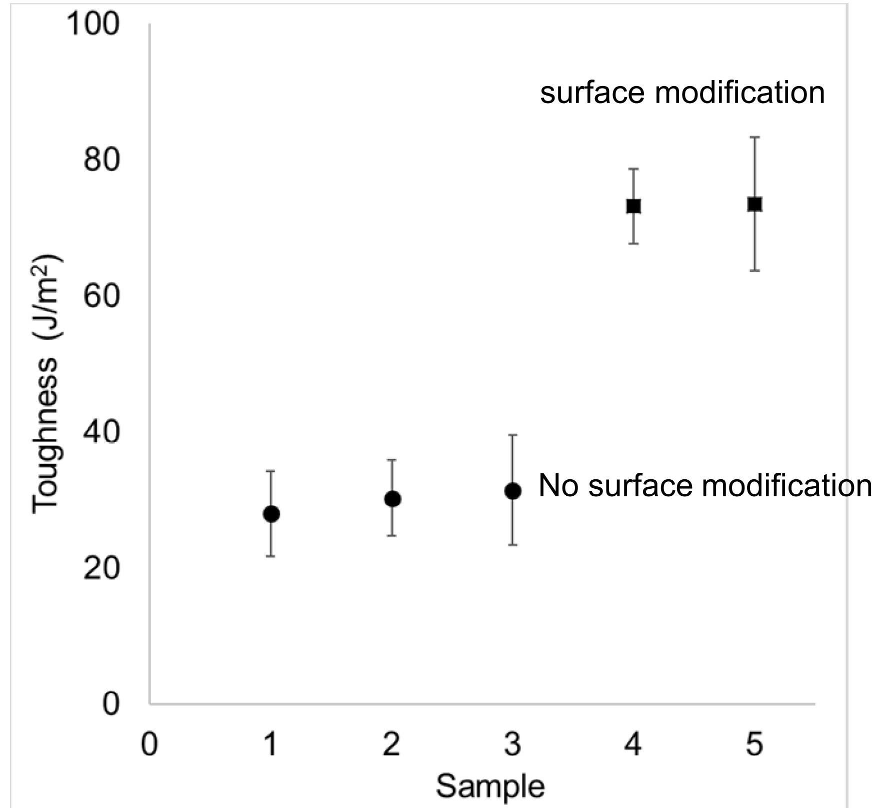
• Γ depends on surface chemistry, roughness, adjacent bulk materials, temperature, loading rate, environmental aging, etc.

Developed techniques to measure the toughness of an alumina/epoxy interface



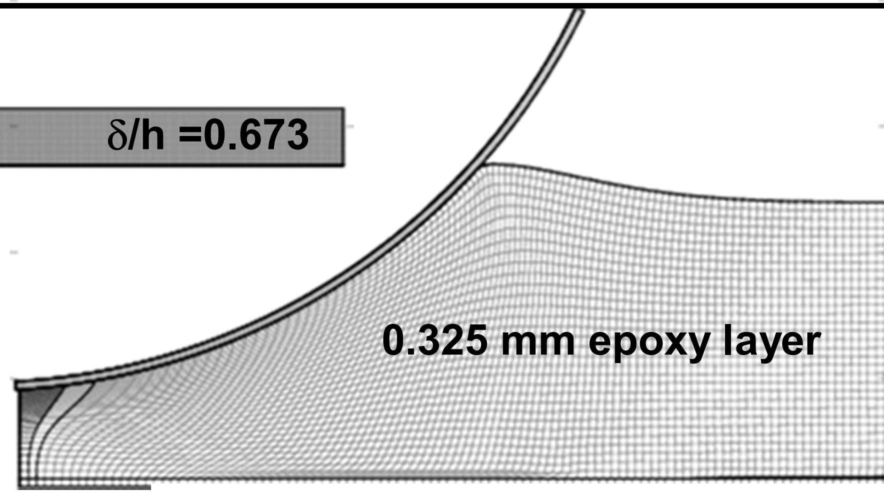


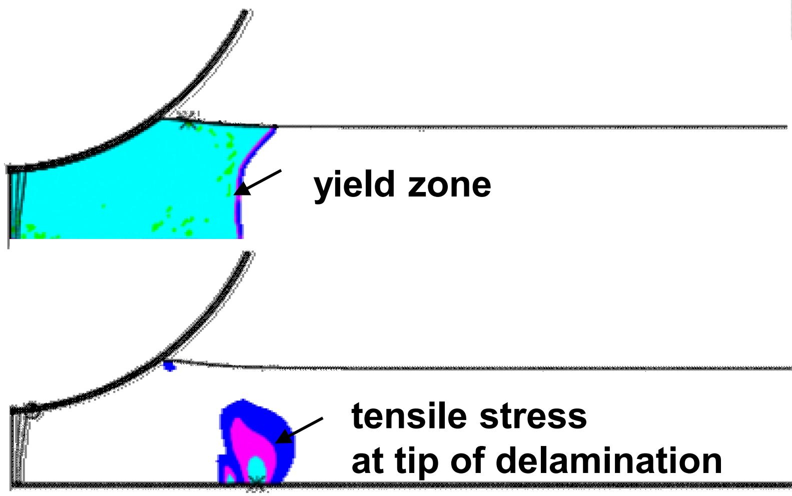


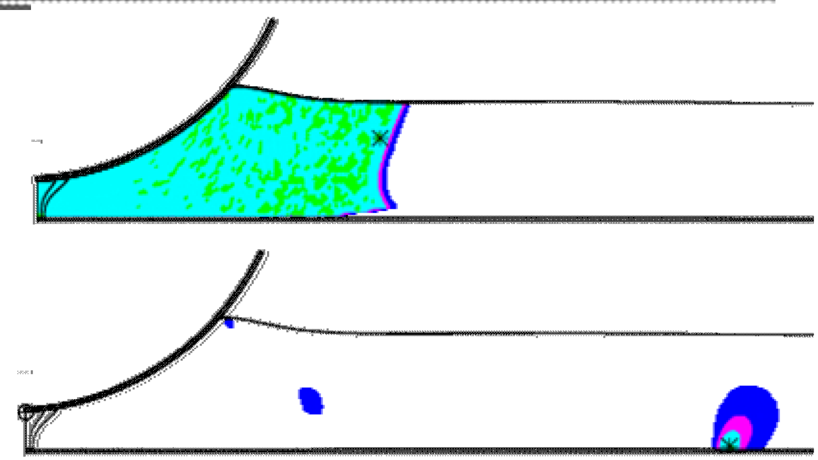


Initial Sierra/SM indentation test analysis results

Results for Γ = 20 J/m^2 (scotch-brite roughened surface)





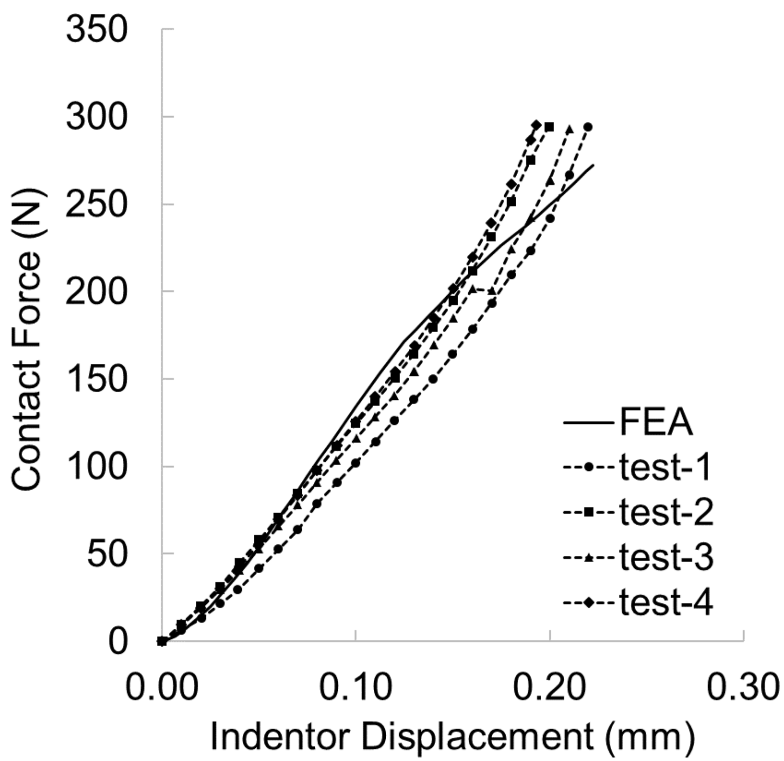


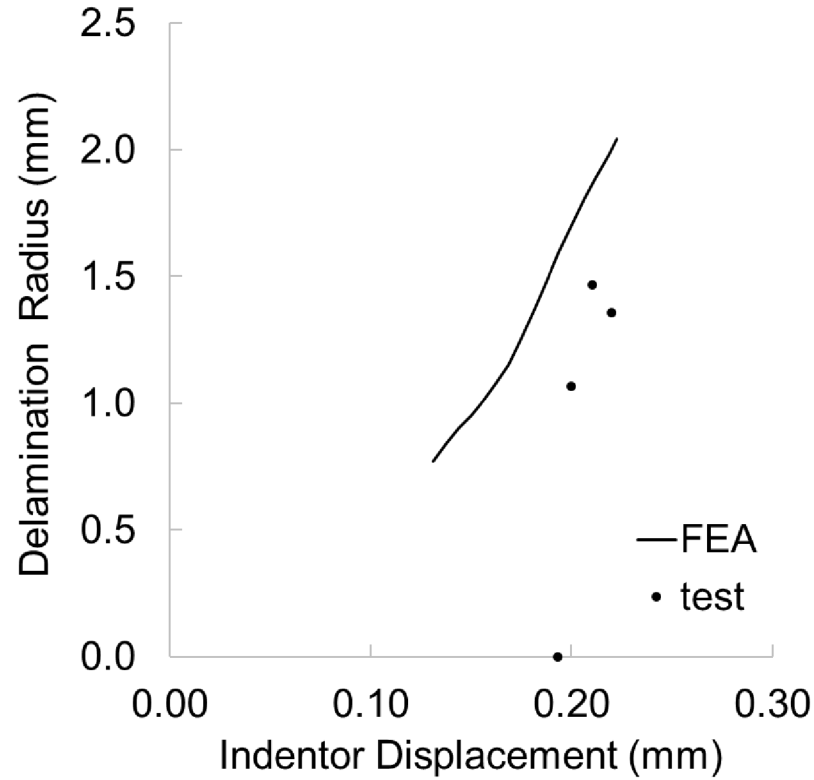
$\delta/h = 0.388$
delamination begins to grow

$\delta/h = 0.673$
delamination ~ 2 mm in diameter

- **Large-scale yielding occurs in film beyond contact radius**

Comparison of Indentation Test and Analysis





- **There is "fair" agreement between analysis and indenter tests**
  - **analysis may benefit if a higher fidelity polymer constitutive model is used.**
  - **additional testing with better characterization of delamination event may prove useful (e.g., delamination radius vs load; does the epoxy crack?)**

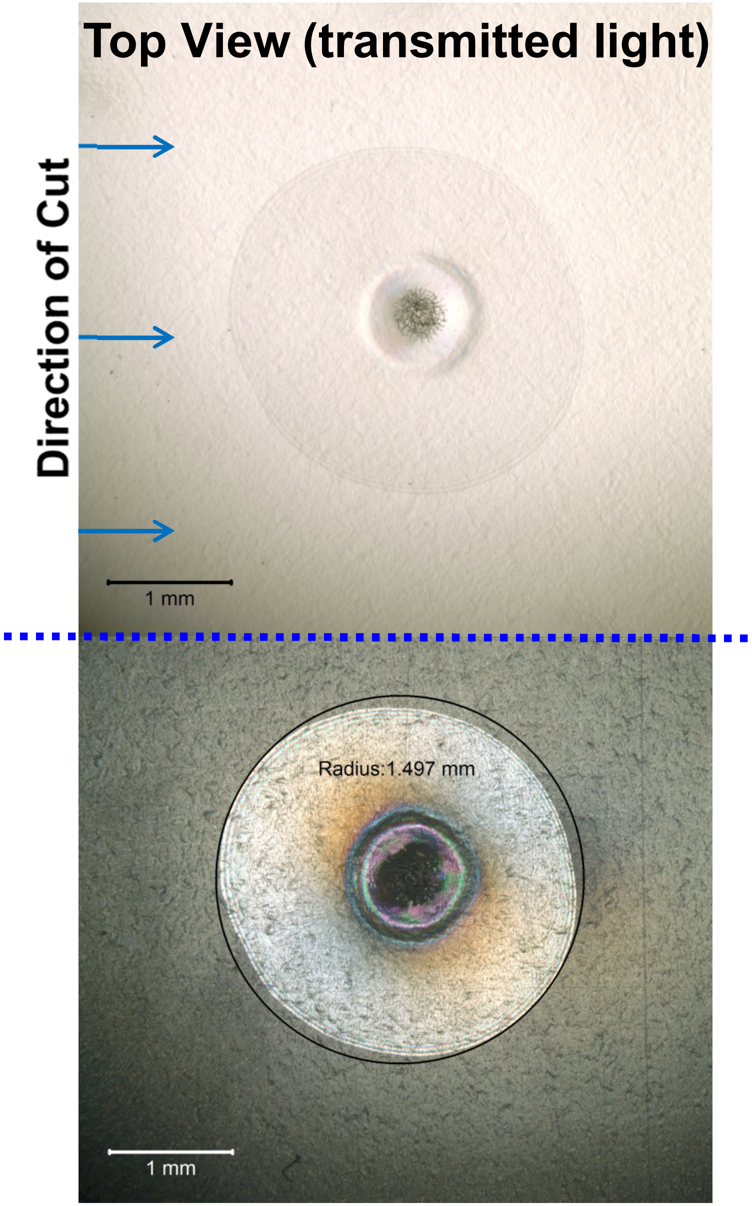
Conclusions

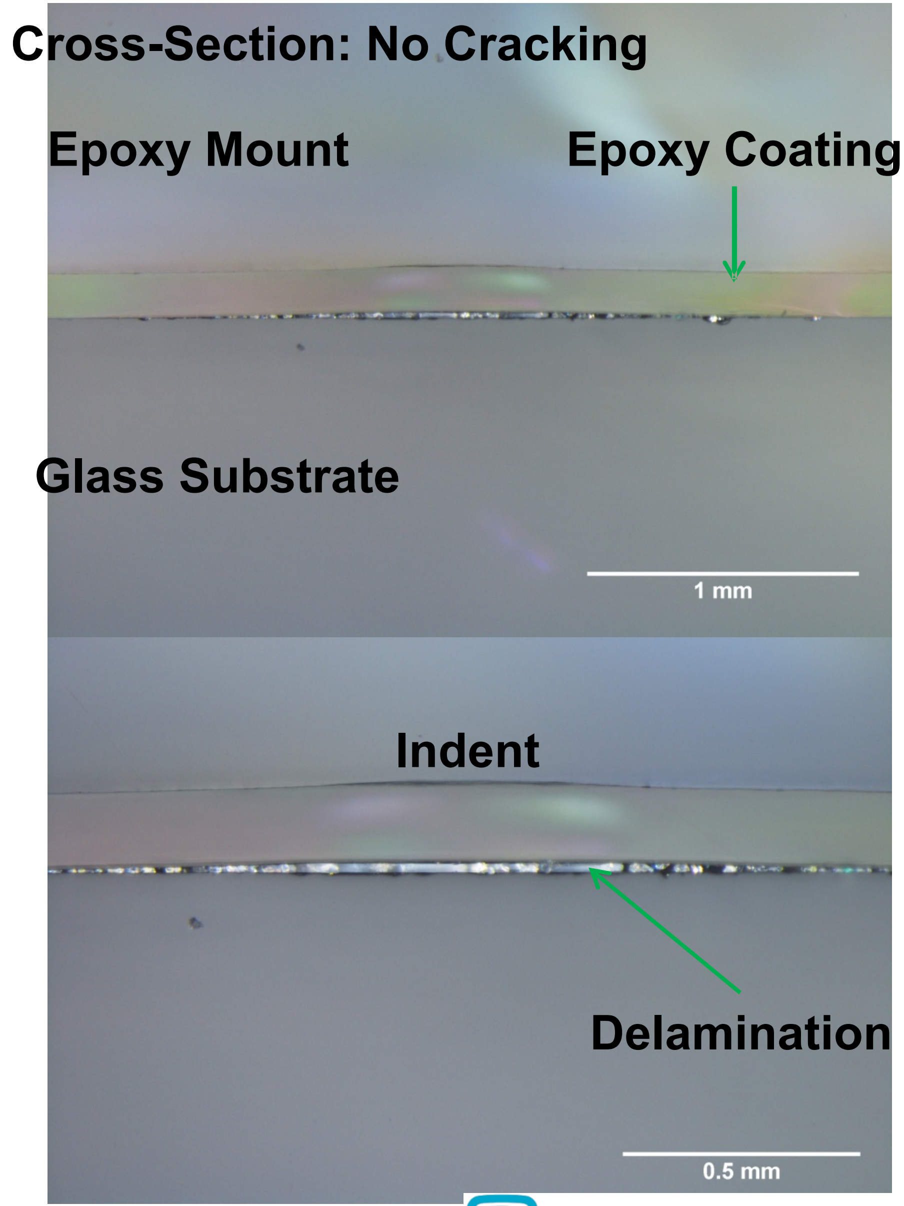
Epoxy on rigid substrates: Hard to initiate delaminations at RT

Residual stress increase must aid in delamination

Modifications of alumina surfaces significantly increase interfacial shear strength (results shown), and interfacial fracture toughness

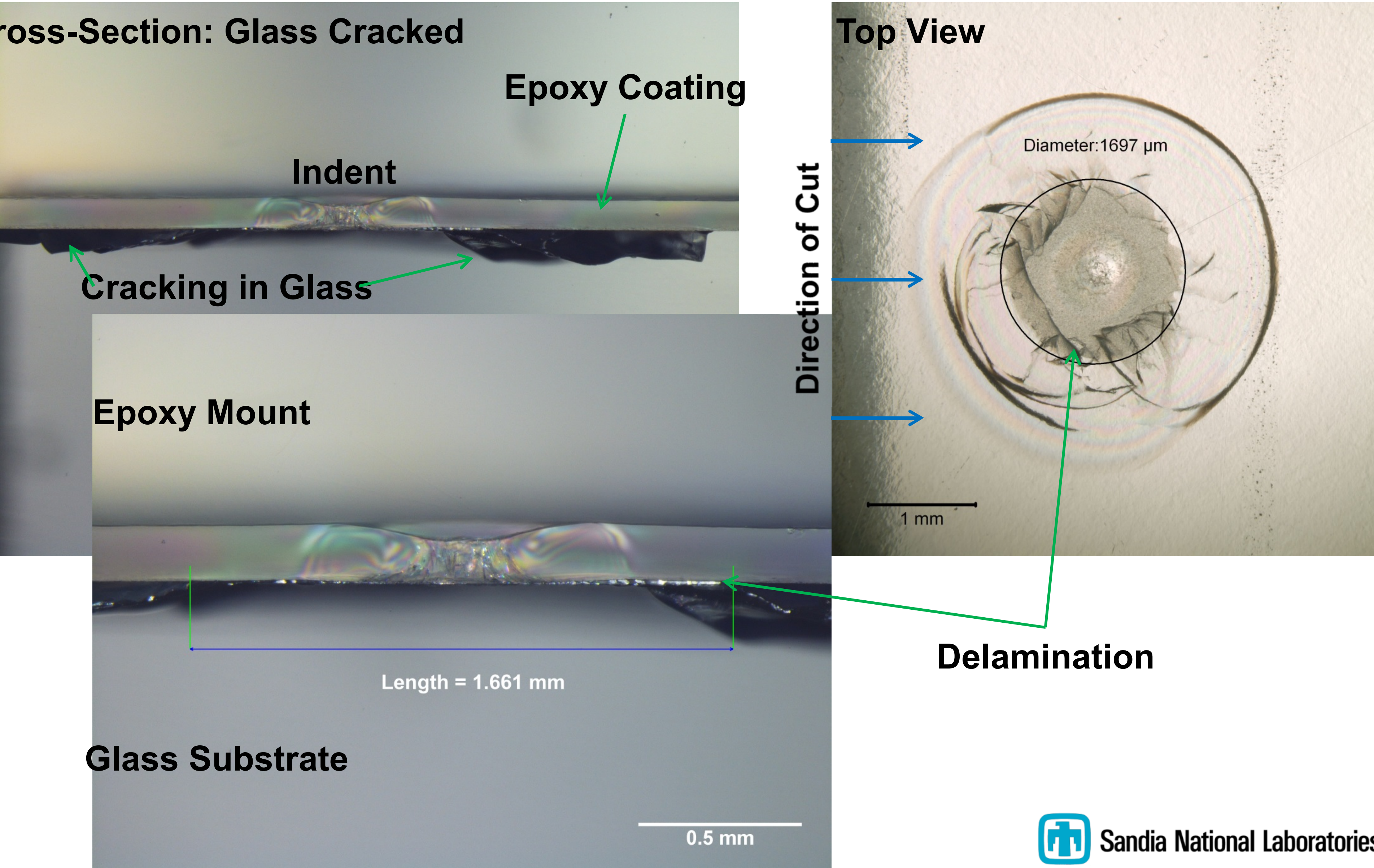
As-rcvd. Glass Surface



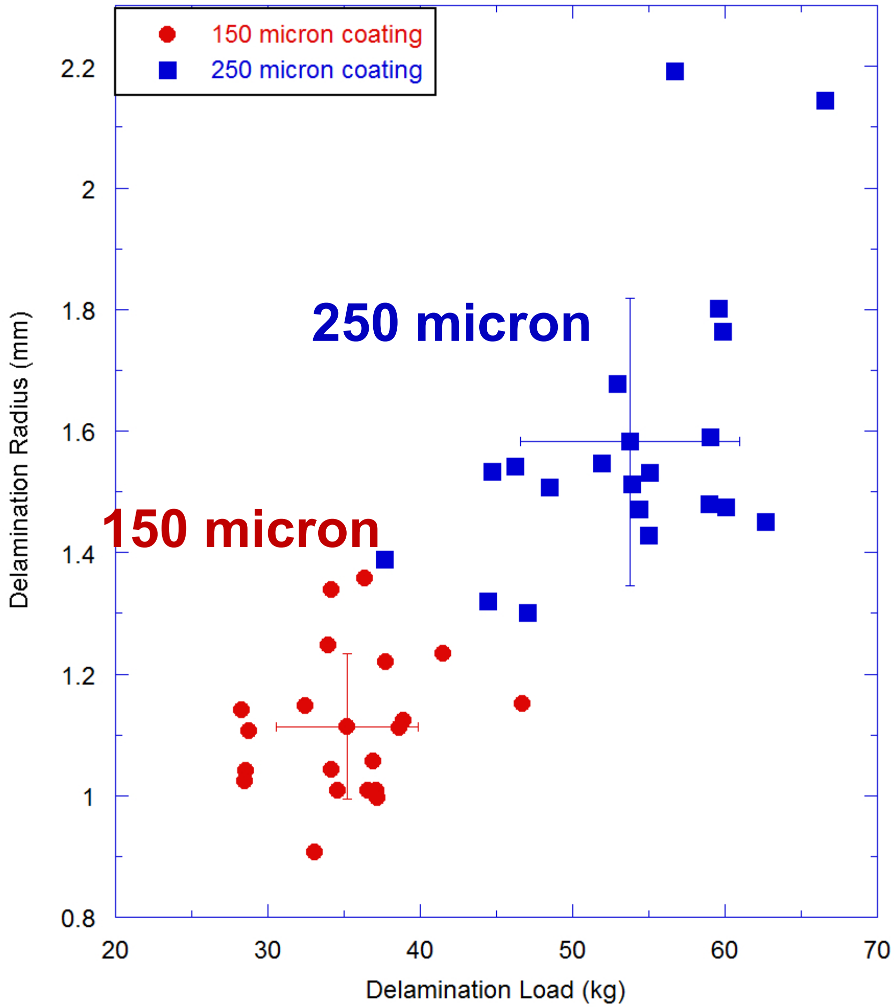


Sandia National Laboratories

Roughened Glass Surface



Effect of Coating Thickness on Delamination



Higher load is required to delaminate the thicker coating

Epoxy Thickness	Delam. Load	Delam. Radius
150 μm	35± 5 kg	1.1 ± 0.1 mm
250 μm	54 ± 7 kg	1.6 ± 0.2 mm

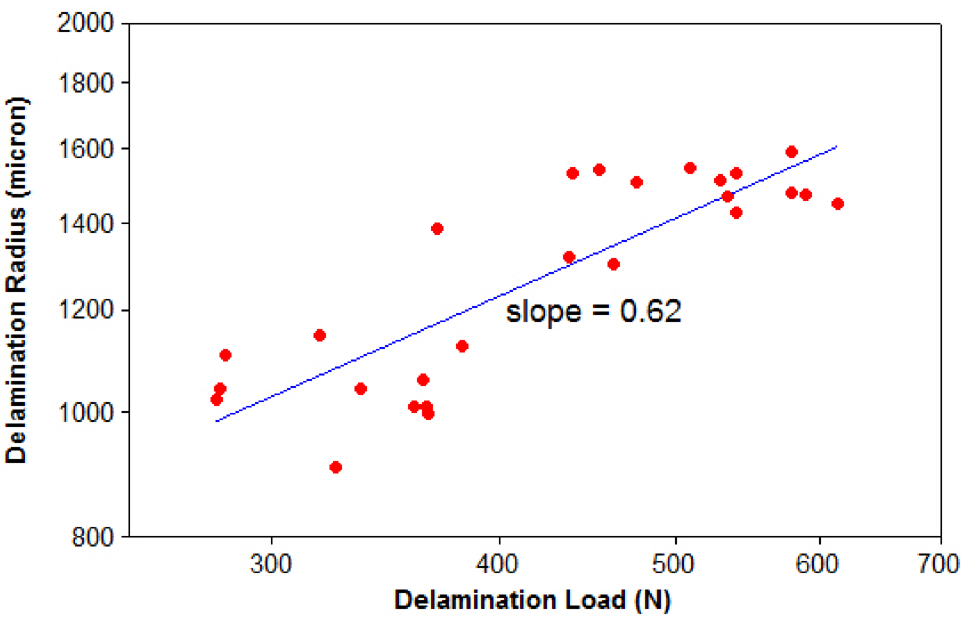
Interfacial fracture energy (glass-epoxy)

$$G = \frac{0.627 H^2 h \left(1-\nu_c^{\,2}\right)}{E_c} \frac{1}{\left[1+\nu_c+2\left(1-\nu_c\right) {Hc^2}/{P}\right]^2}$$

Rosenfeld, et al., J. Appl. Phys. p. 3291, 1990

$$c \propto P^{1/2}$$

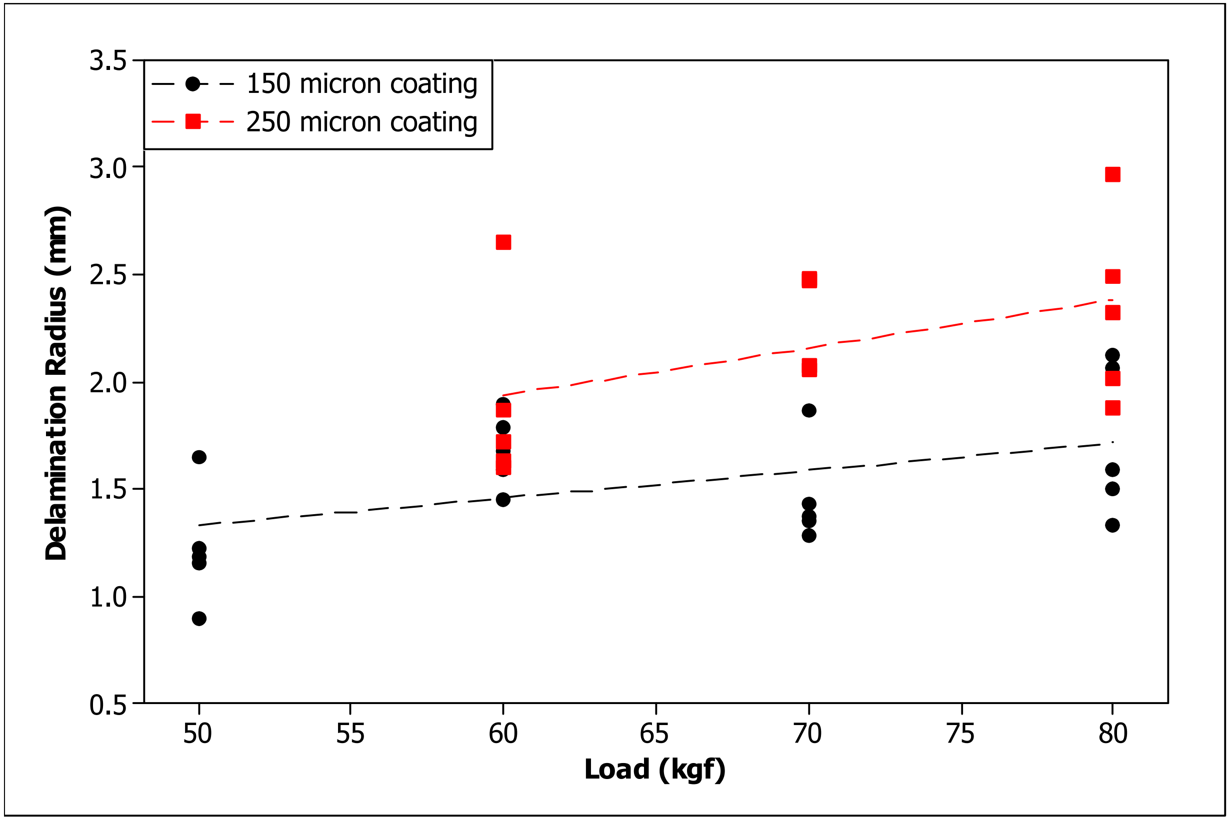
G = strain energy release rate
P = indenter load
c = crack/delamination radius
h = Epoxy thickness, ~100 μm
H = Epoxy hardness, 600 MPa
E_c = Epoxy modulus, 3600 MPa
v_c = Epoxy Poisson's ratio, 0.38



G_c = 514 ± 144 J/m² for 150 μm
G_c = 595 ± 174 J/m² for 250 μm

The interfacial fracture energy appears to be independent of film thickness in range investigated

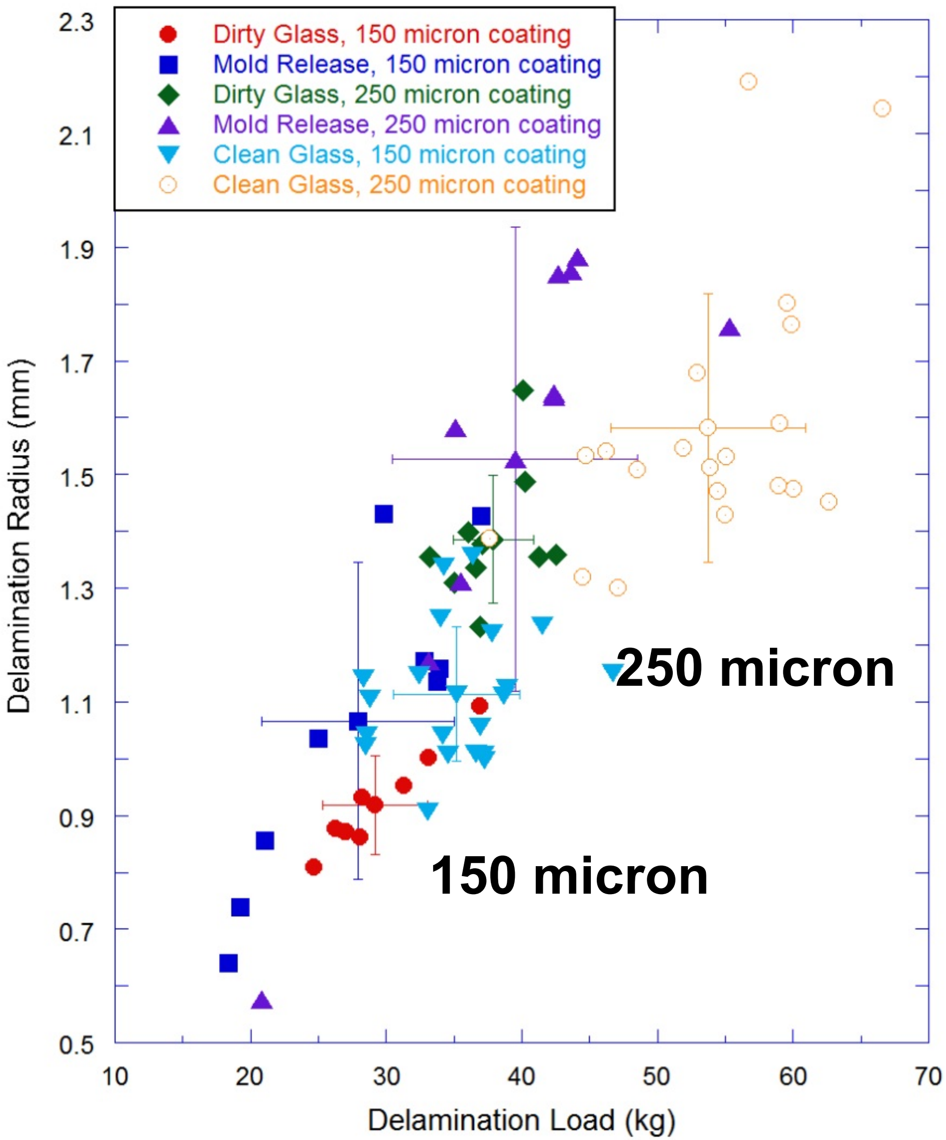
Effect of Load on Delamination Size



- Loading continued after delamination was initiated
- Delamination size increases with increasing load
- Crack sizes are larger for the thicker coating

Higher strain energy associated with the thicker coating

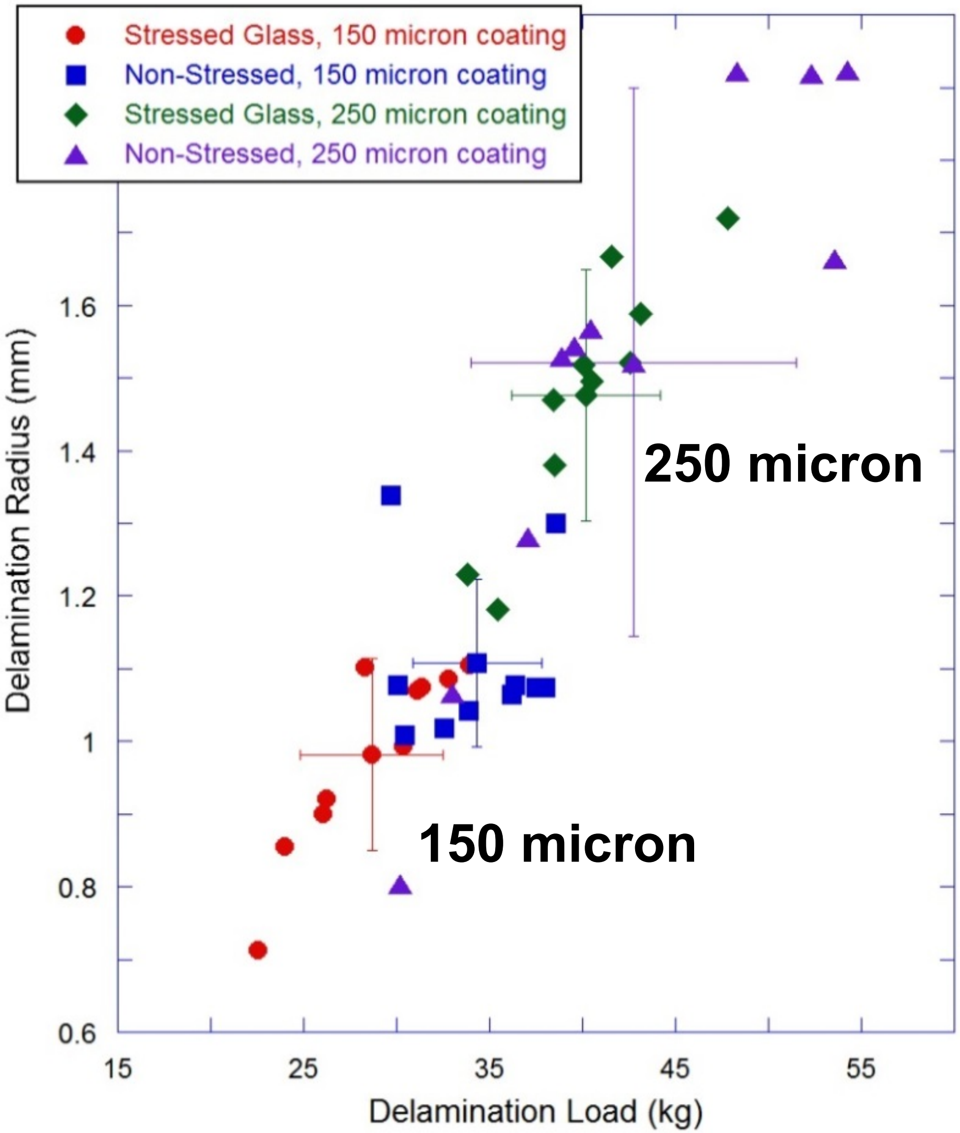
Effect of Surface Cleanliness on Adhesion



- Some samples were handled and had fingerprints placed on them prior to coating
- Other samples were contaminated by spraying a mold release on the surface of the glass prior to coating

- On average, delamination occurs at lower loads for the dirty and contaminated glass than the clean glass
- The samples that were contaminated with mold release show much more variability than the other samples

Does stress in the glass affect delamination?



- Stressed (tempered) glass was tested to see if the compressive stress already present in the glass would interact with the shear stresses imparted by the indenter loading and influence delamination loads
- On average, delamination occurs at lower loads for the stressed glass than the non-stressed glass. However, it is not statistically significant due to the scatter in the data.