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Measurement of Adhesion in Alumina/Glass-Epoxy System Using Spherical Indentation

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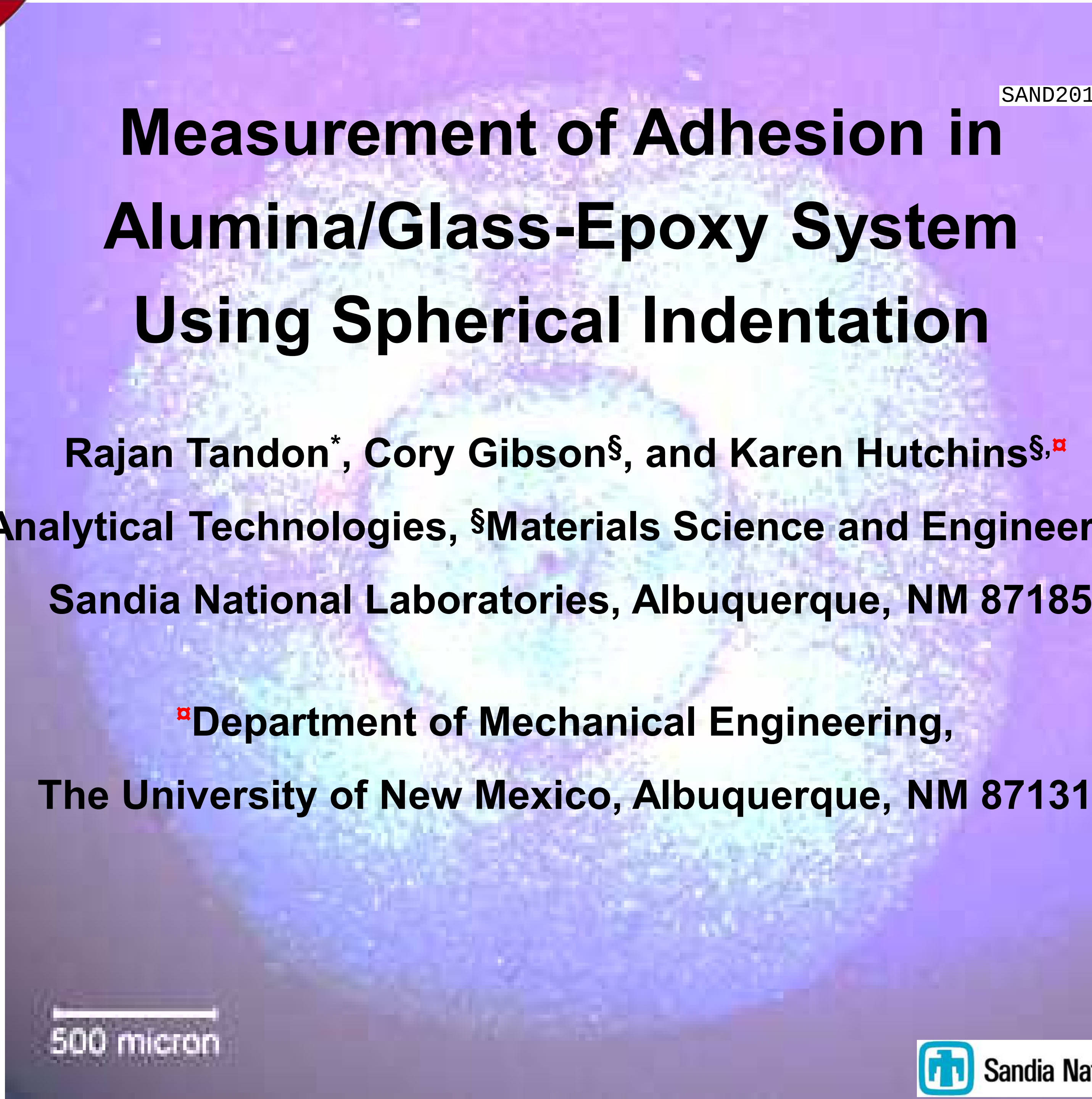

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Outline

- Test Method and Mode-mixity

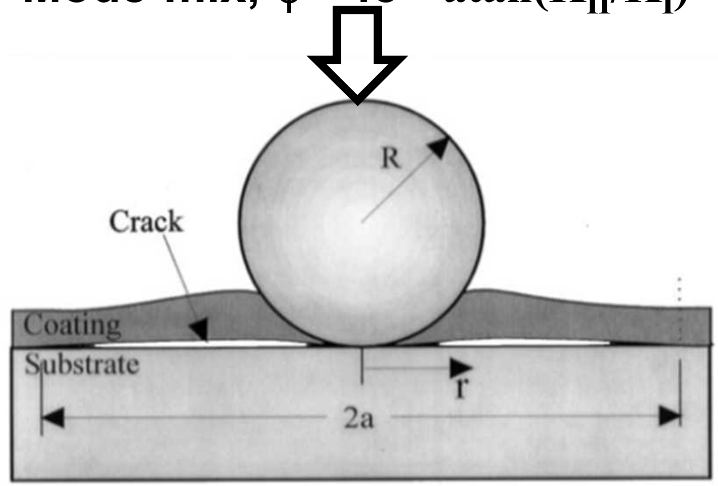
- Initial Room Temperature Results: Alumina-epoxy and glass-epoxy

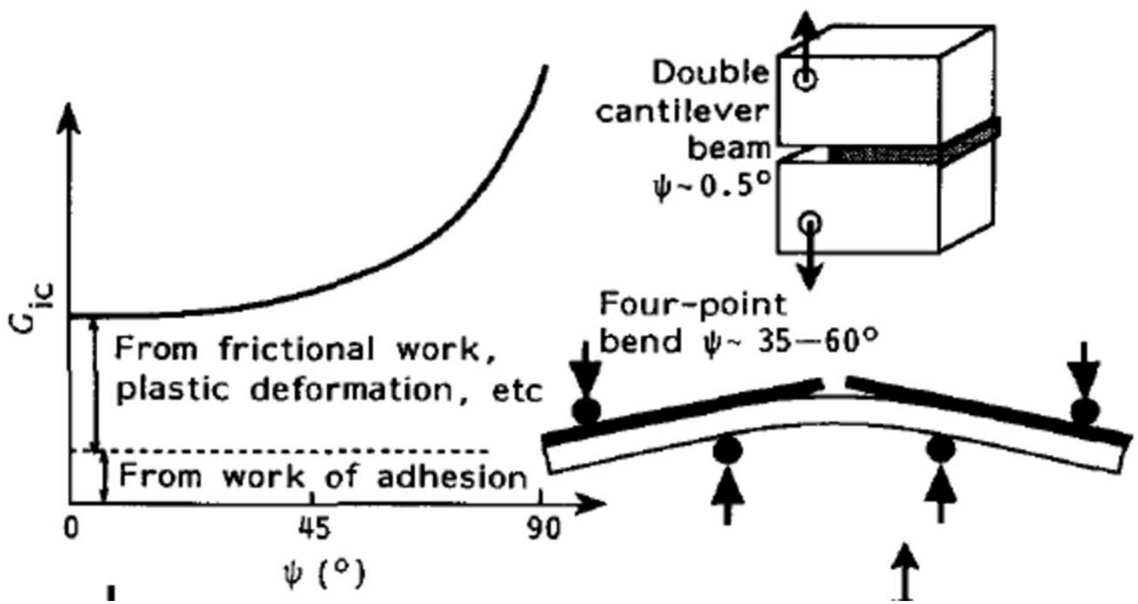
- Stress Distributions and Cold Temperature Results
    - Alumina (temperature, load, and cleanliness)
    - Glass (thickness, cleanliness, stress)

- Calculations of interfacial fracture energy

Characterization of interfacial adhesion

Mode-mix, ψ ~45°=atan(K_{II}/K_I)

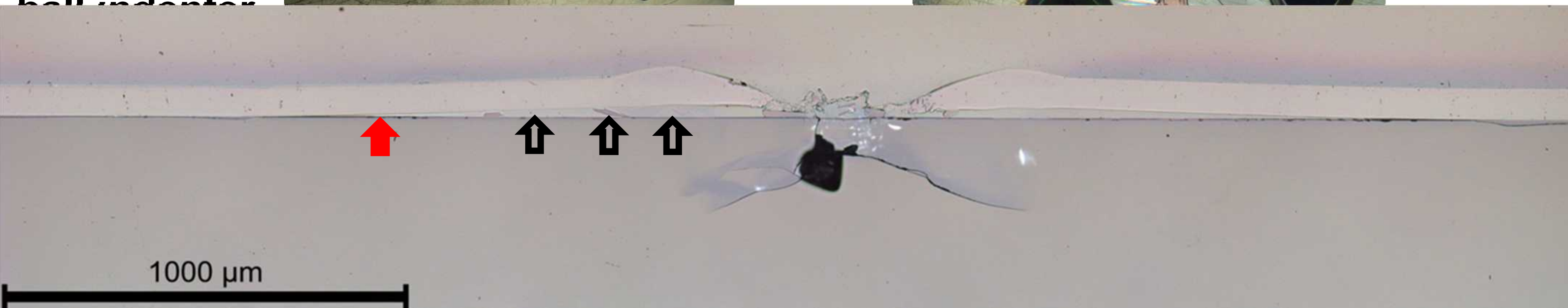




Focus on indentation work as both interfacial adhesion, and shear strength can be obtained

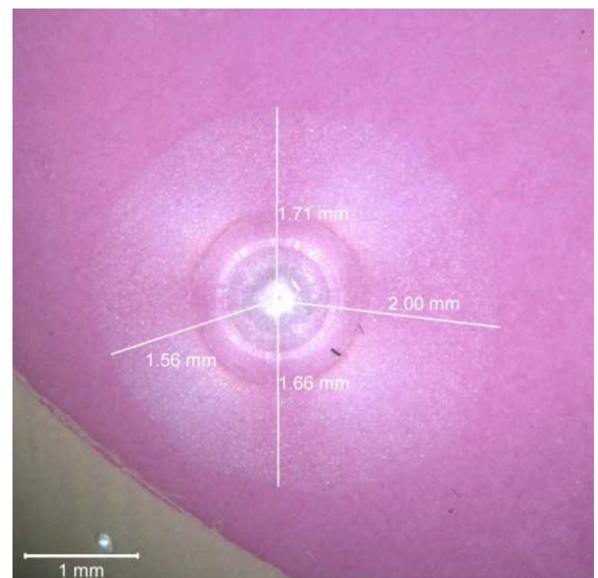
Room Temperature Results

1/8” dia. WC



observed.

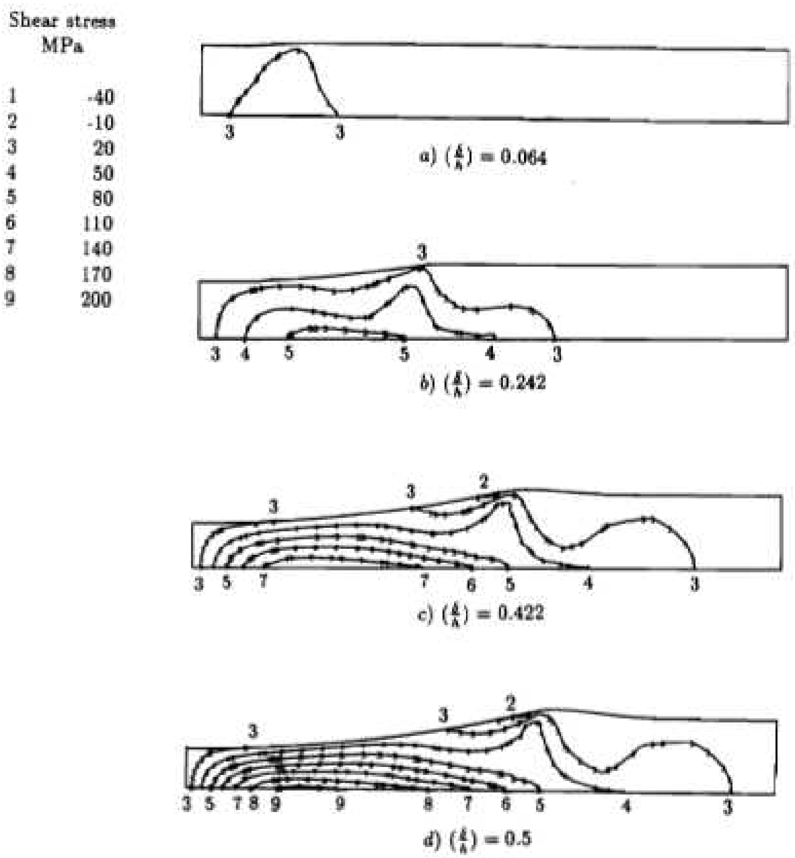
A 1/16” diameter spherical WC indenter used

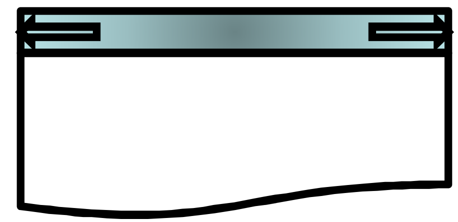


Coating thickness ~100 μm (?)

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.





$\alpha_{Epon} >> \alpha_{Alumina}$

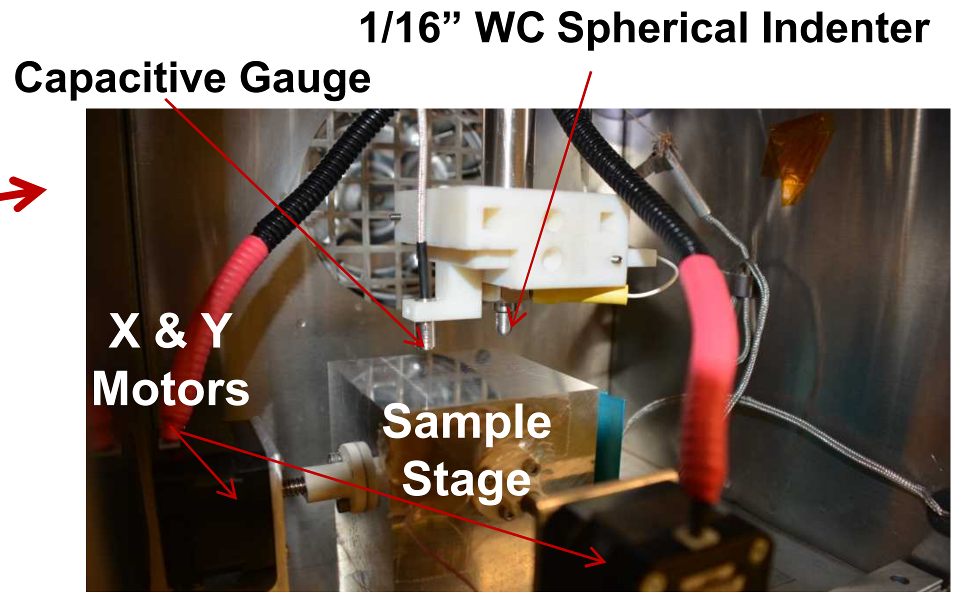
Epon cure at 71^{o}C

Epon 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

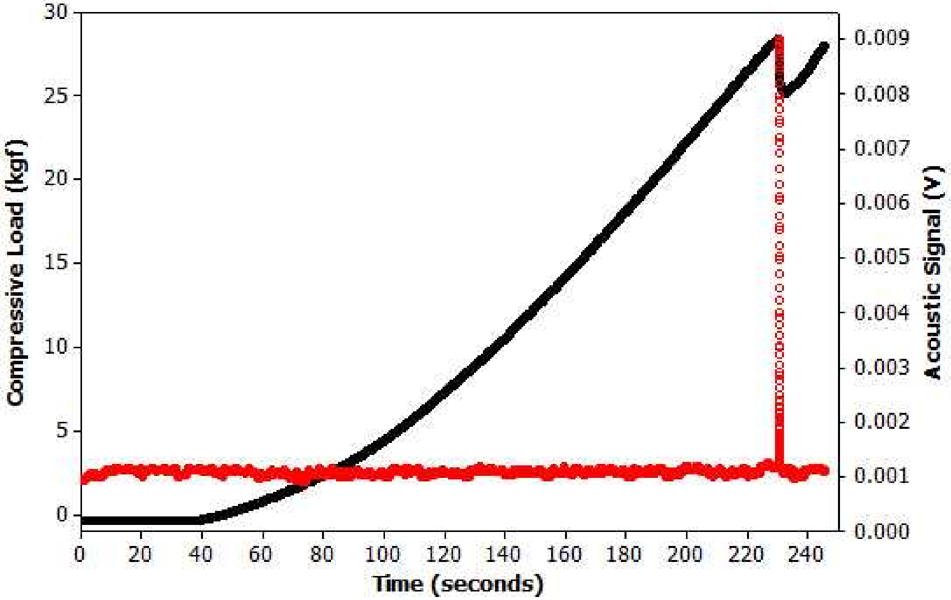
Experimental Setup



Environmental Chamber (LN_2 chilled)

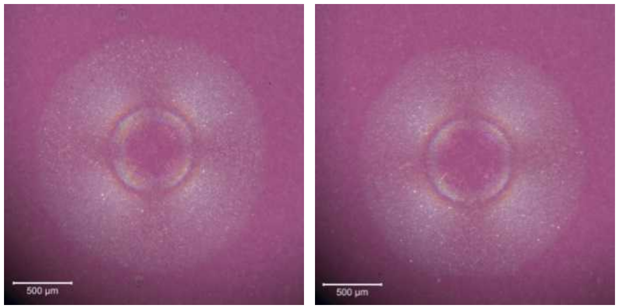
- Indenter attached to load cell bolts to bottom of crosshead.
- Crosshead rate 0.05 mm/min.

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



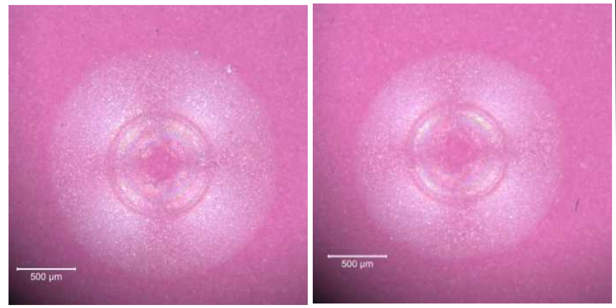
Contaminated vs. Clean surfaces

Contaminated 25 kg, -30°C

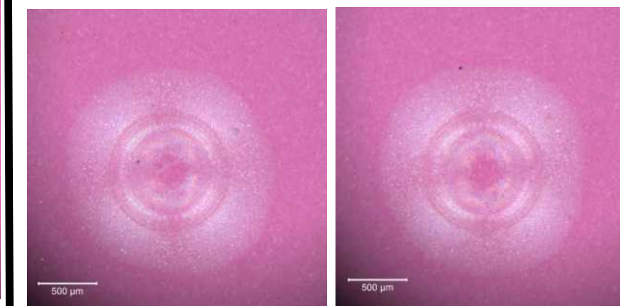


R=1000 micron, 880 micron

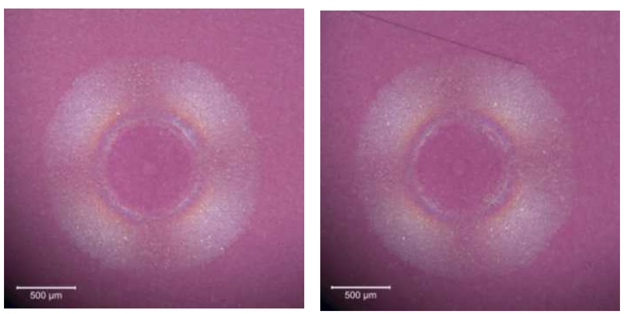
0°C



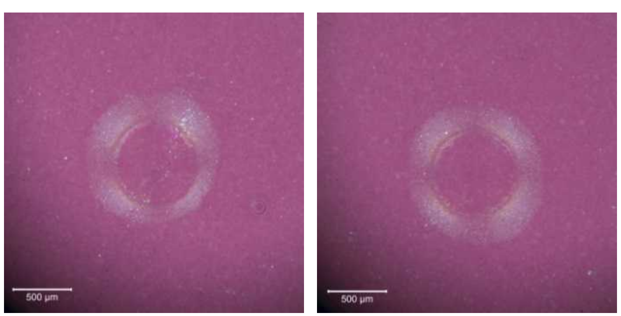
Room temperature

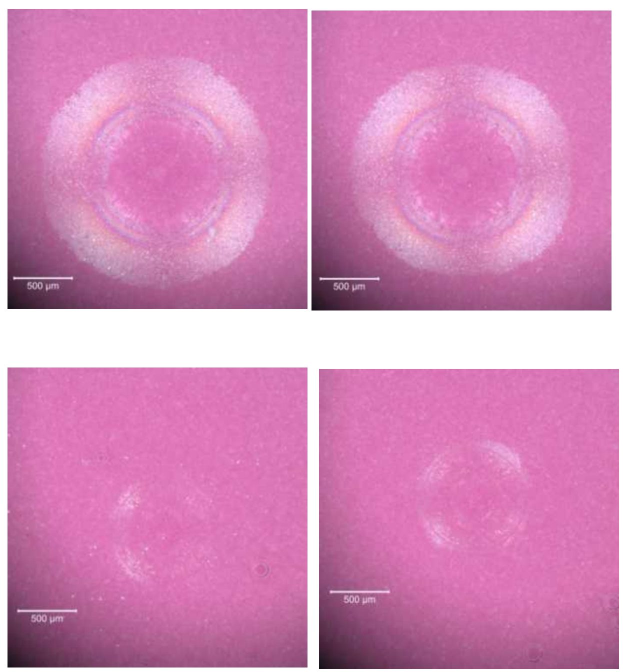


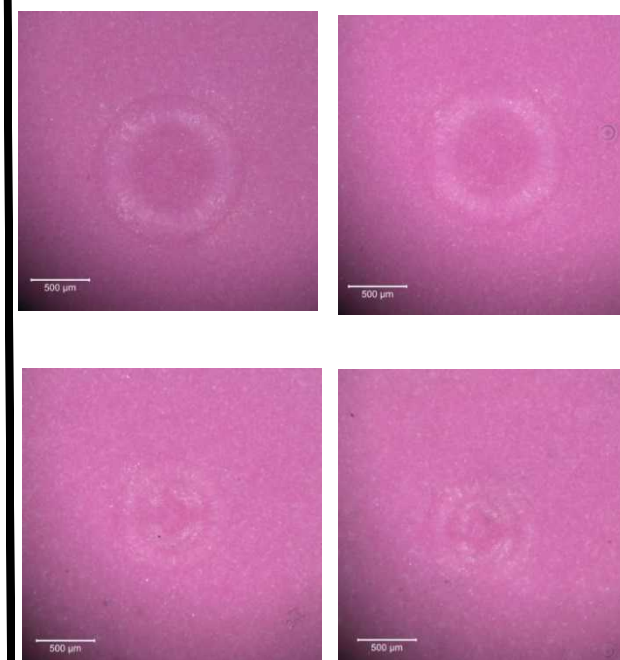
Identical Cleans (?)



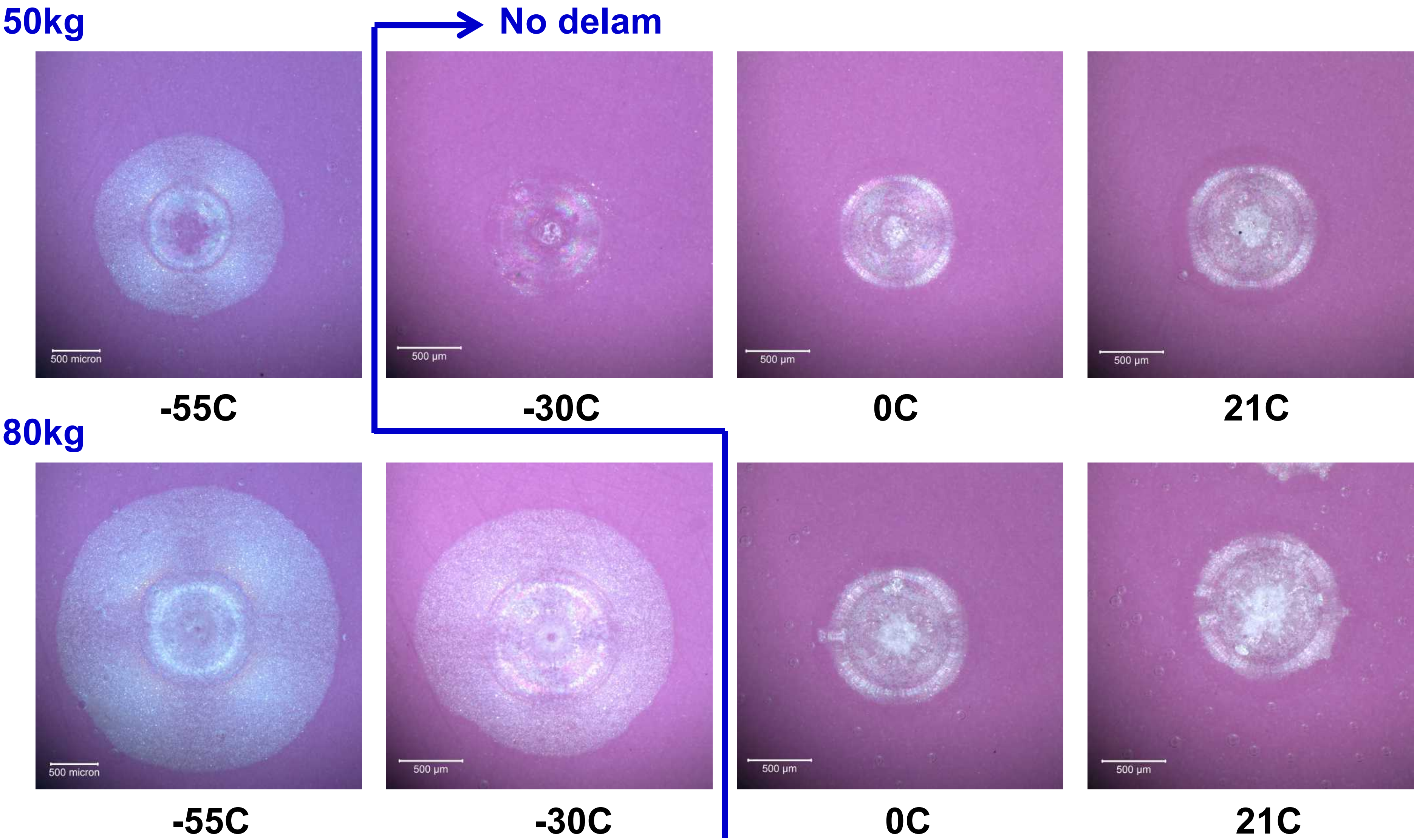
R=950 micron







Alumina Sample – Temp./load effects



Increased delam size: Lower temperature and increase load

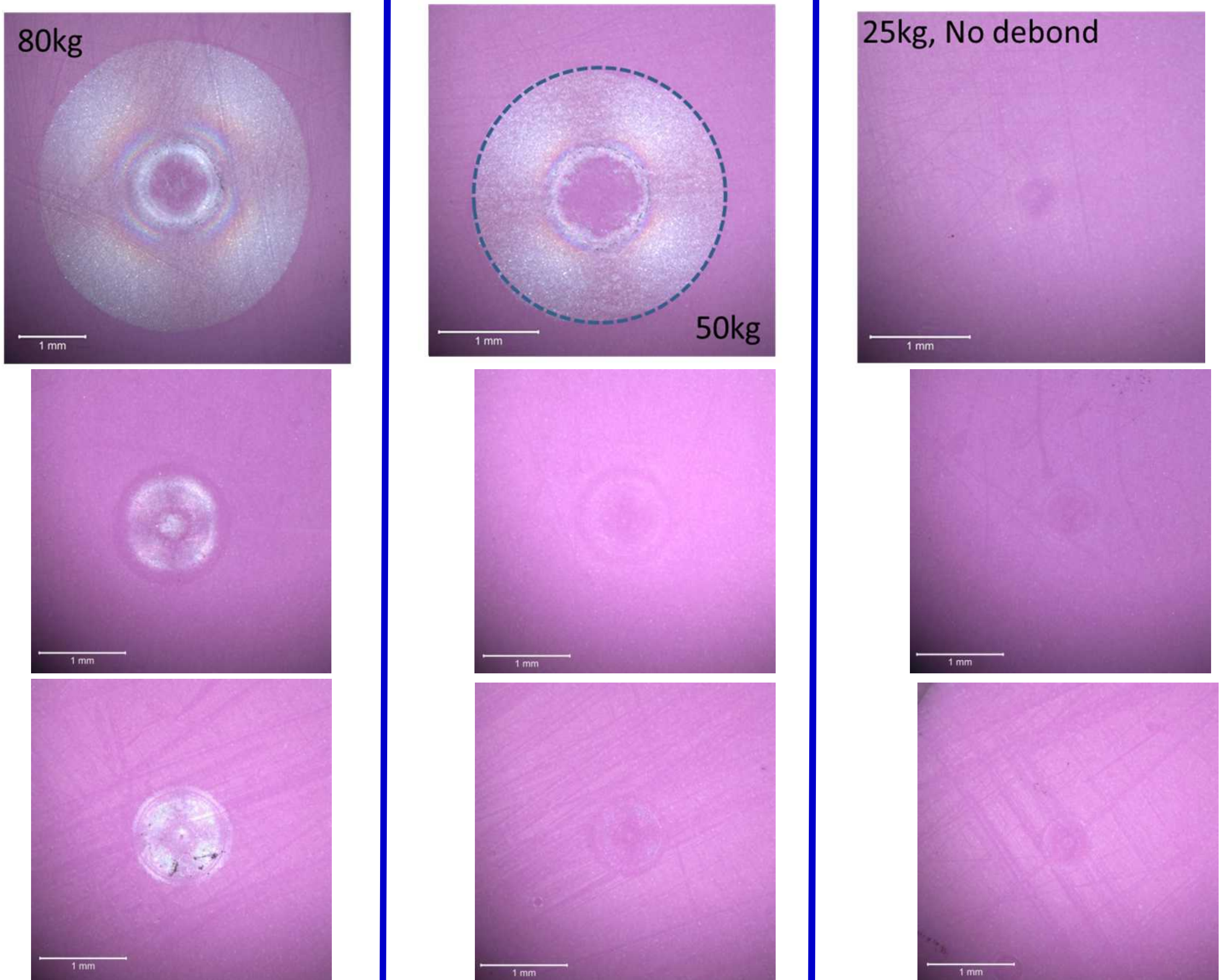
Alumina surface modification

Control

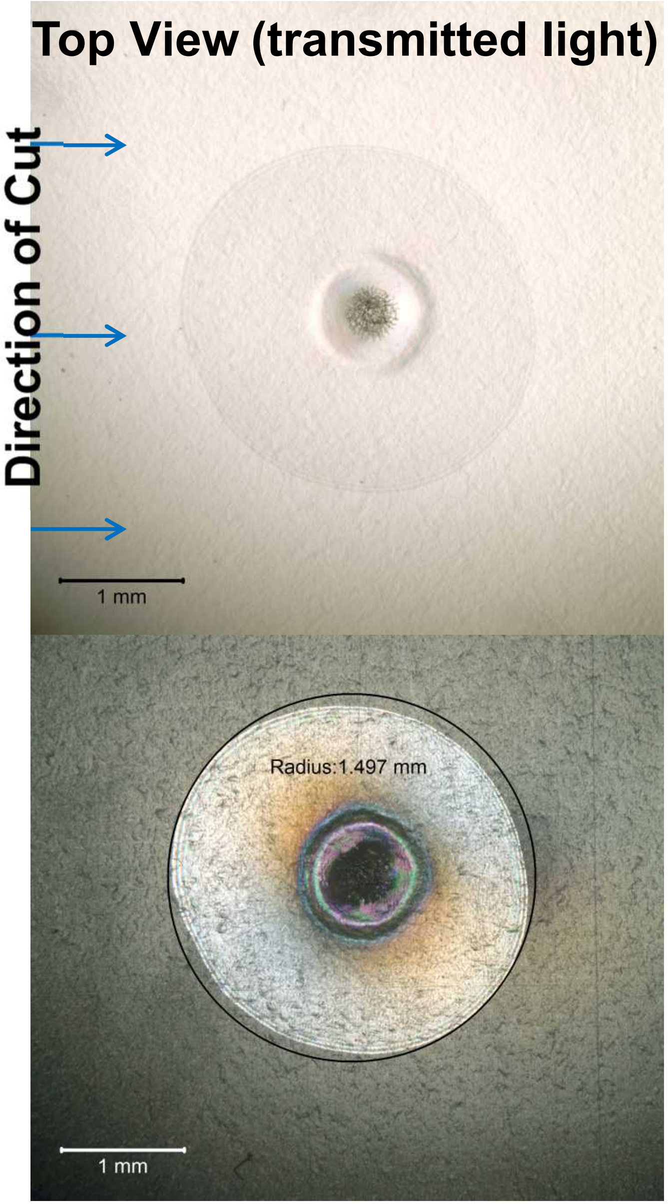
Test at -55°C

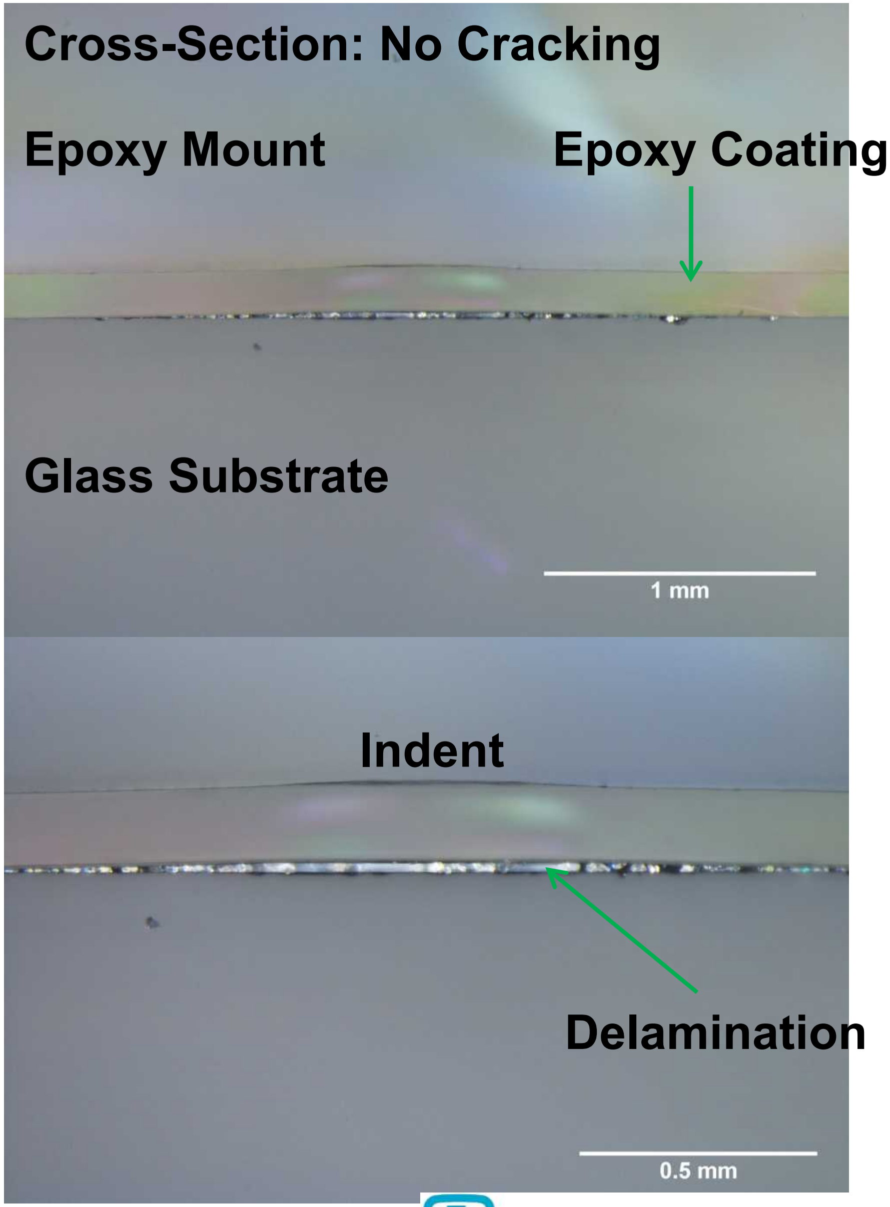
Surface Mod. 1

Surface Mod. 2



As-rcvd. Glass Surface





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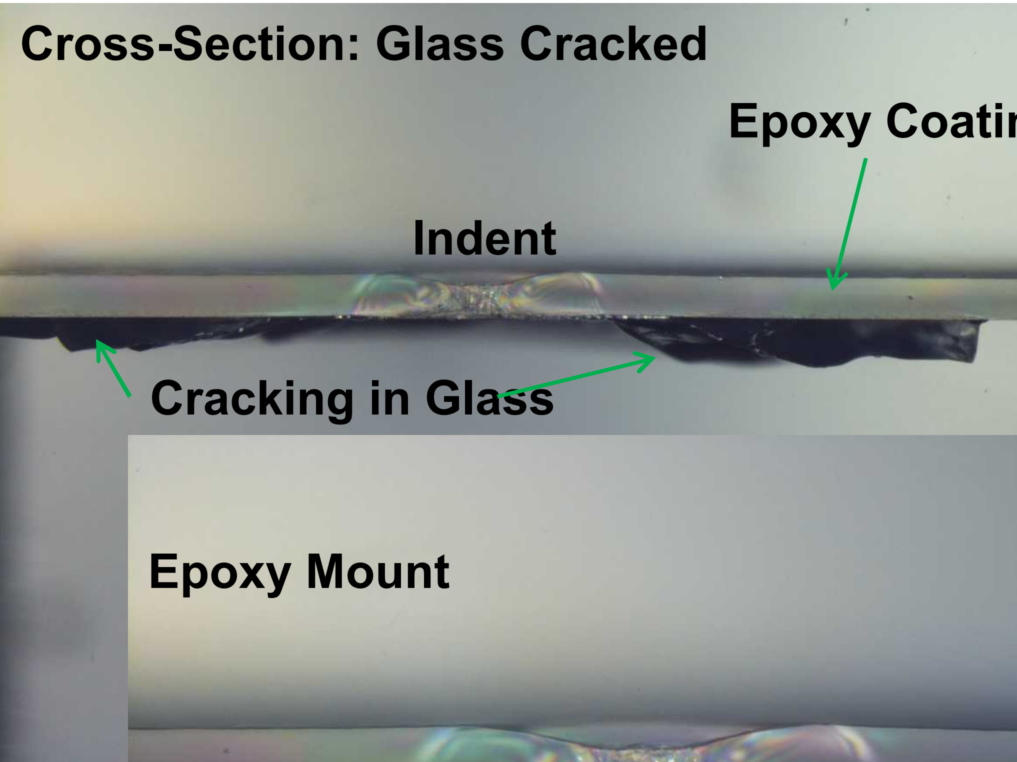
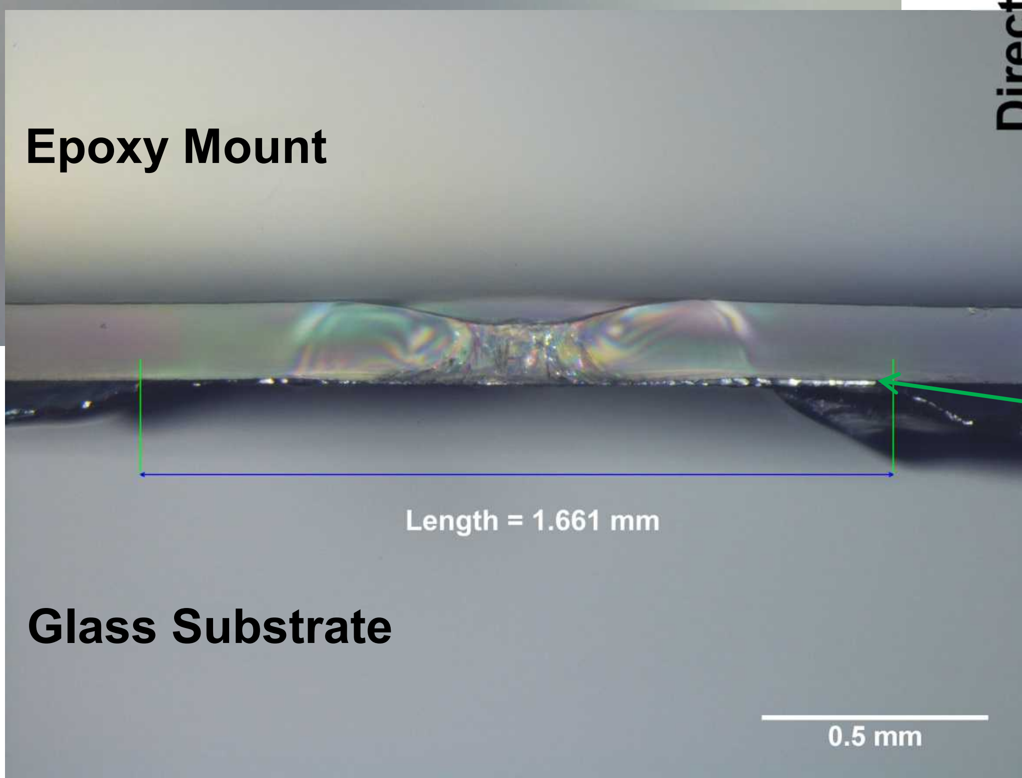
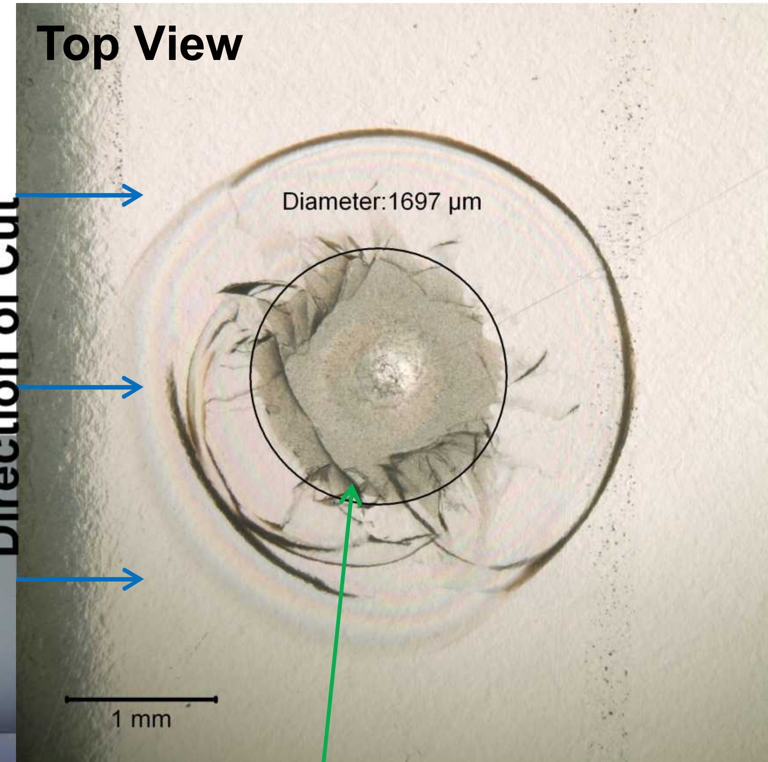
Roughened Glass Surface

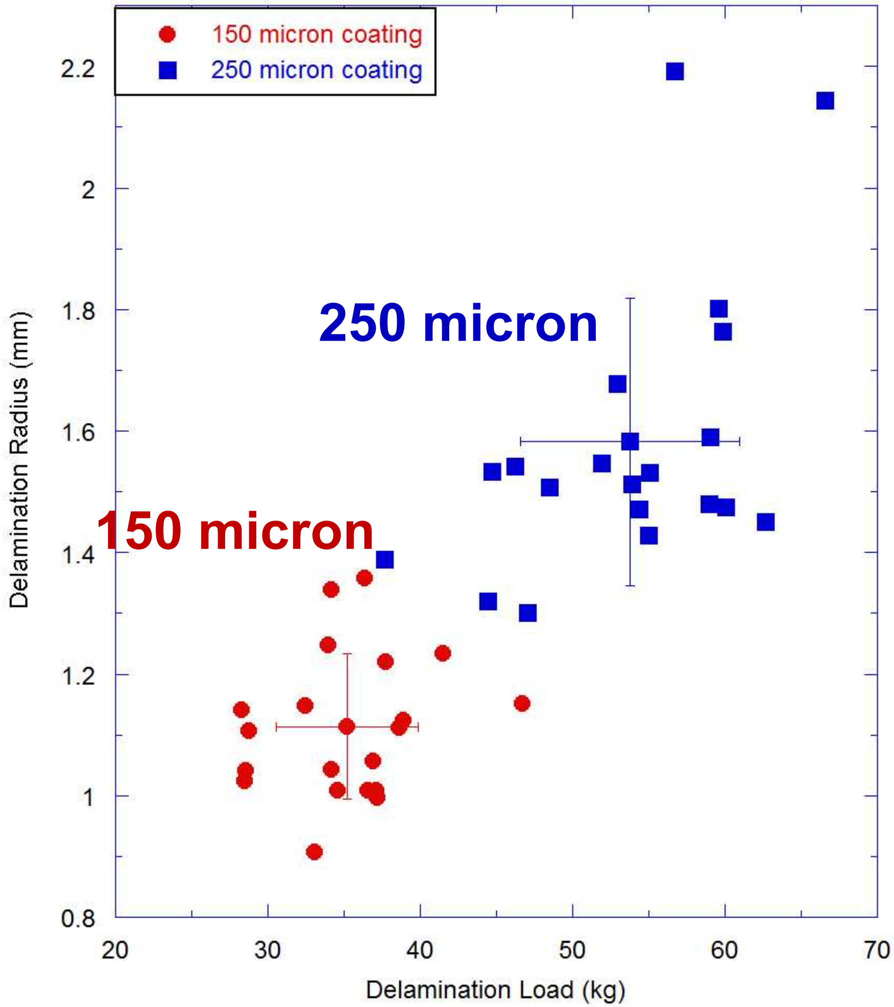






Delamination

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

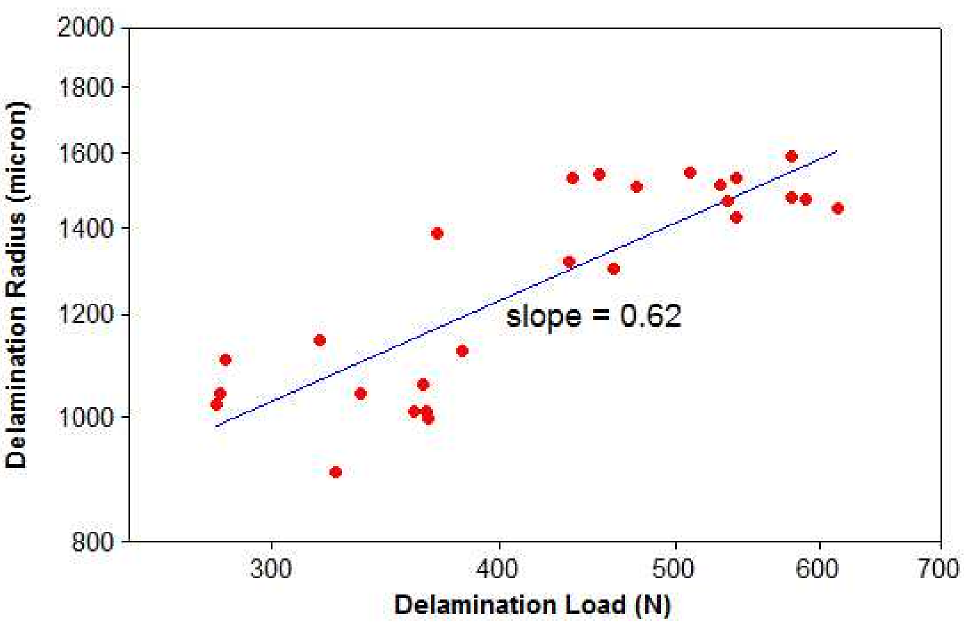
Interfacial fracture energy (glass-epoxy)

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

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

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

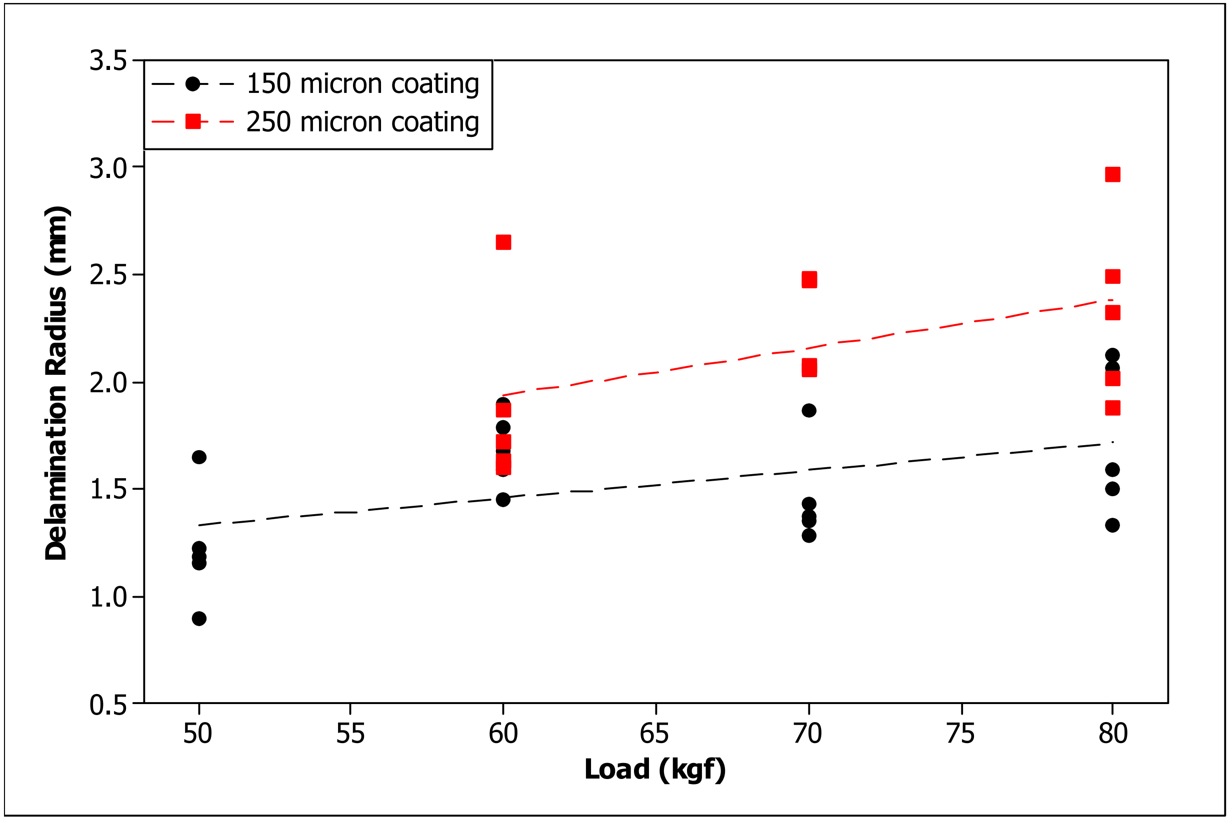
G = strain energy release rate
P = indenter load
c = crack/delamination radius
h = Epoxy thickness, ~100 μm
H = Epoxy hardness, 220 MPa
E_c = Epoxy modulus, 3600 MPa
v_c = Epoxy Poisson's ratio, 0.38



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

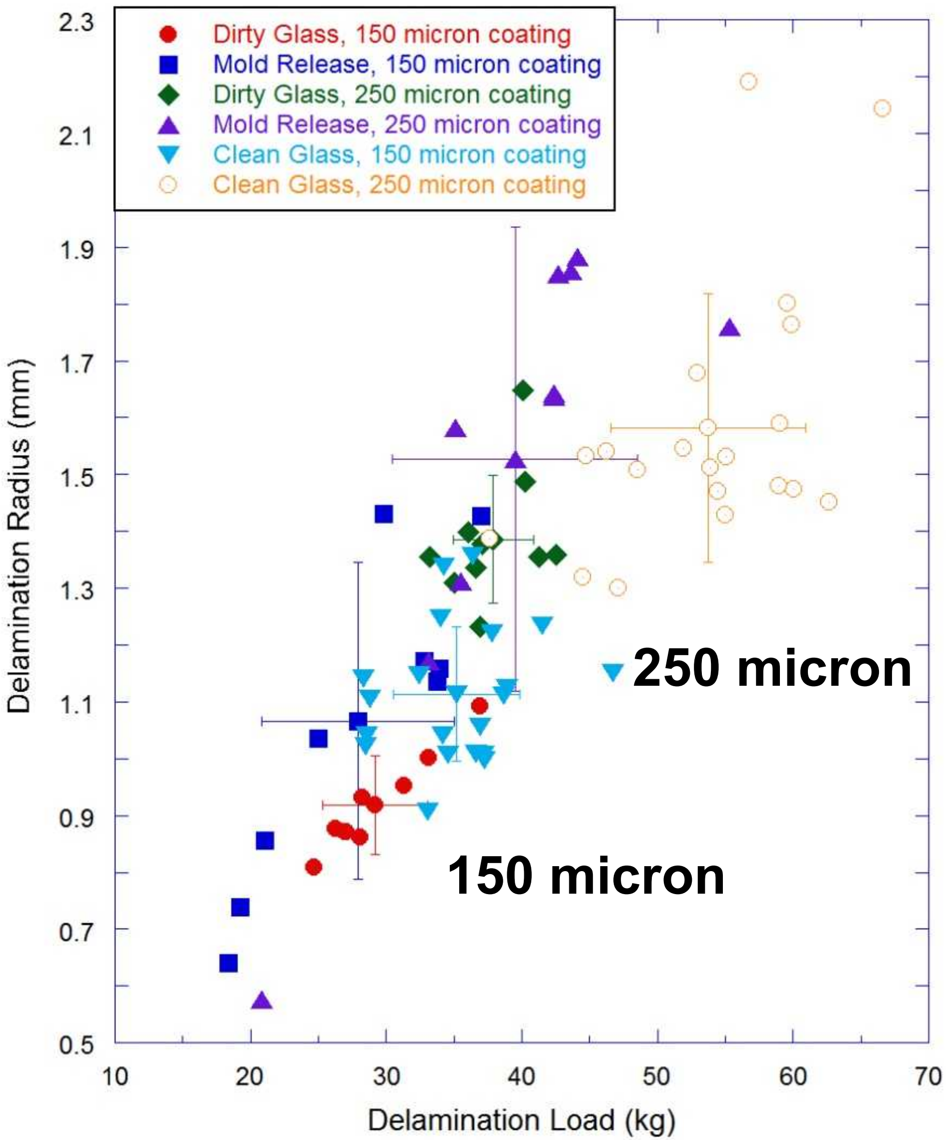
The interfacial fracture energy appears to be independent of film thickness in range investigated

Effect of Load on Delamination Size



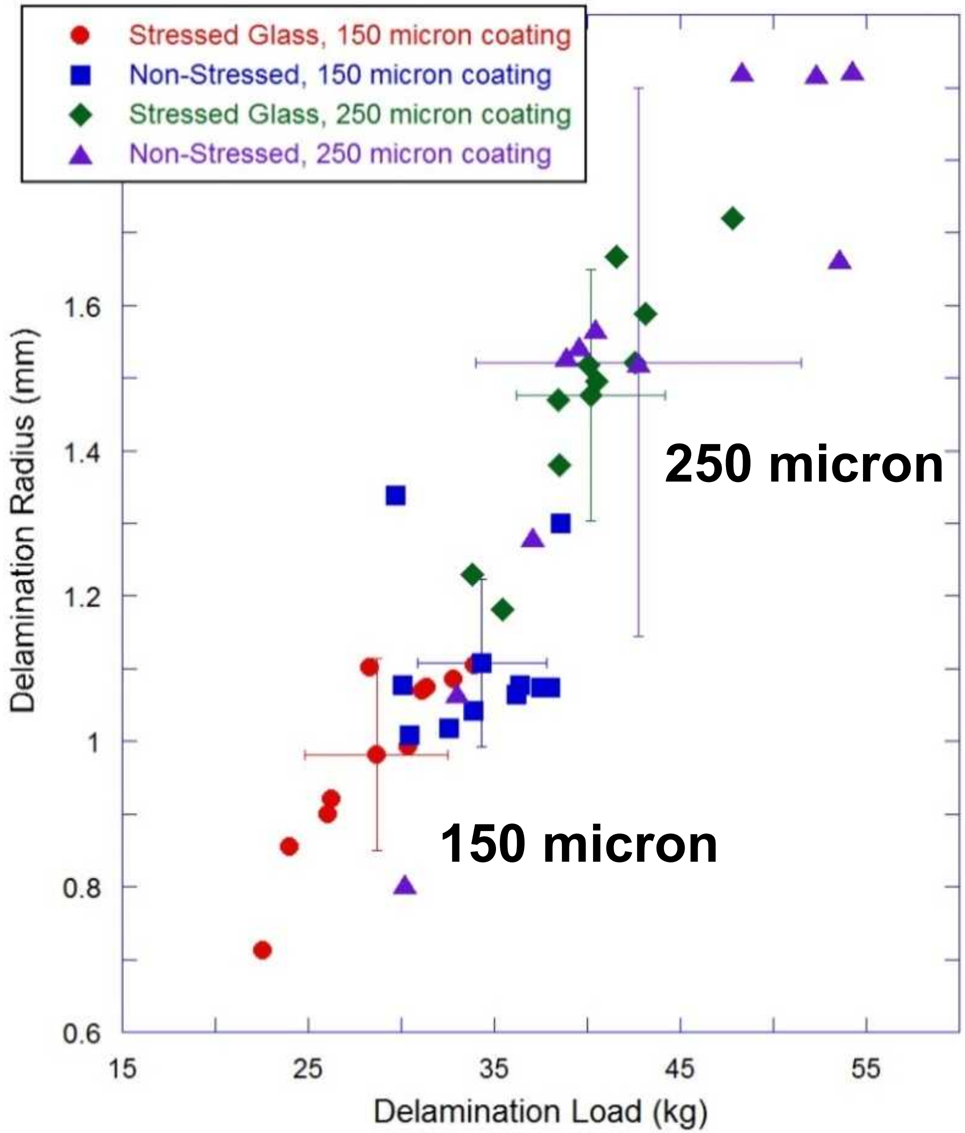
- Loading continued after delam was initiated
- Delamination size increases with increasing load
- For delaminations at the same load, the crack sizes are larger for the thicker coating
  - Higher strain energy associated with the thicker coating

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 the 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.

Conclusions

Epoxy on rigid substrates: Hard to initiate delaminations at RT

Residual stress increase must aid in delamination

Higher loads needed to delaminate thicker coatings

Load transfer to interface

Crack sizes are larger for the thicker coating

Higher strain energy associated with the thicker coating

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

Abstract

Measurement of Adhesion in Alumina/Glass-Epoxy System Using Spherical Indentation

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Bonded systems between different material families (metals, ceramics, and polymers), and within each family are widely used, e.g., polymer coated dielectrics, encapsulated electronic packages, brazed and soldered assemblies, glass to metal seals, plated and coated electrical contacts, coatings on tribological materials, and arc-sprayed components. The performance of such systems is often limited by the adhesion between the layers. Experimental observations and measurements of interfacial properties in glass-epoxy and alumina-epoxy system are described. Spherical indenters were used to induce delaminations at the interface. The load for initial delamination was used to measure the interfacial strength, while the load-crack length relationships are used to estimate interfacial toughness. Surface modifications of the alumina and glass surfaces and their effects on interface adhesion are also described. Fracture surface observations and cross-sectional views of the delaminated regions were used to understand the physical processes occurring at the delamination site.

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Parameter Estimation

What is shear strength of the unmodified interface?

Elasticity analysis provides (plastic zone treated as a pressurized cylinder)

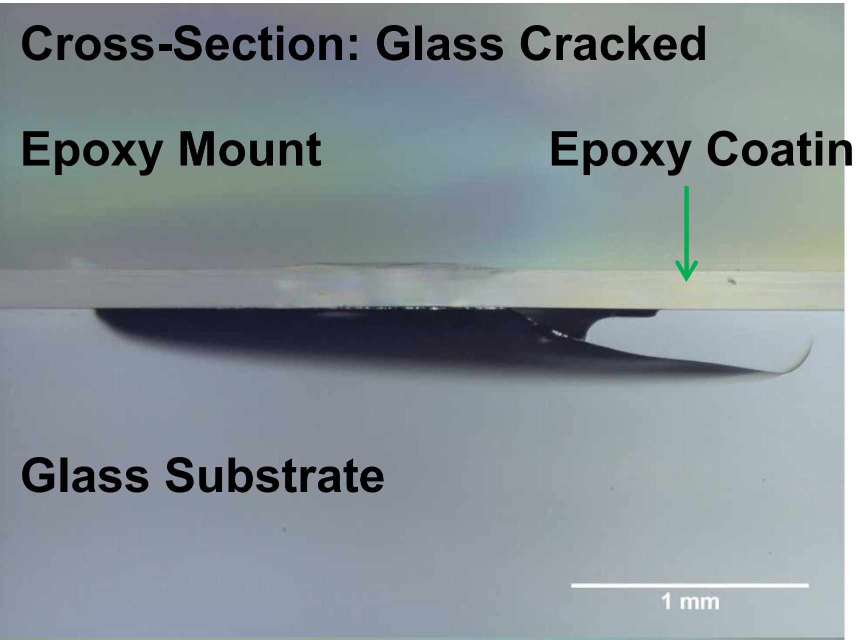
$$\tau_{critical} = \frac{-0.56H}{\dfrac{K_1'(\phi b_{crit}/h)}{\phi K_1(\phi b_{crit}/h)} + \dfrac{\nu_c h}{\phi^2 b_{crit}}} + \sigma_R \qquad \phi = \sqrt{6(1-\nu_c)/(4+\nu_c)}$$

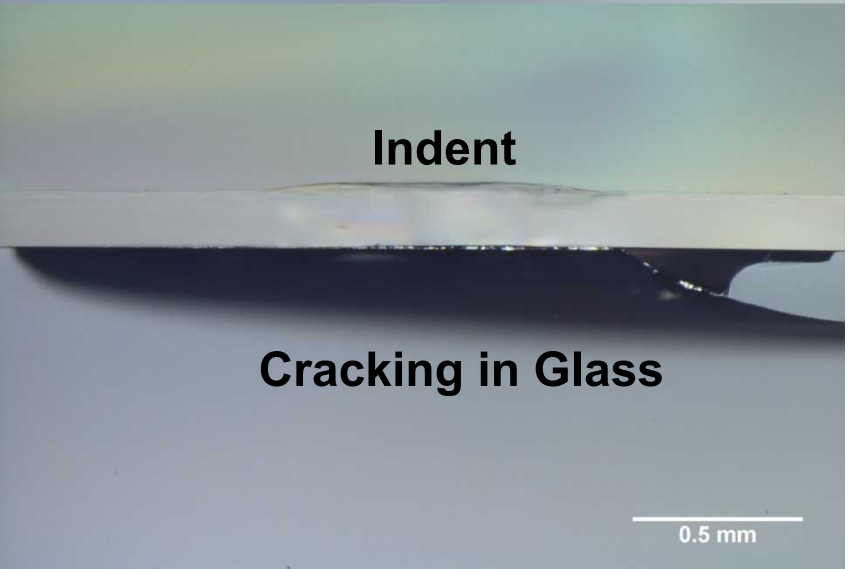
K_1 is modified Bessel function of the second kind, and K_1' its derivative

Ignoring residual stress:

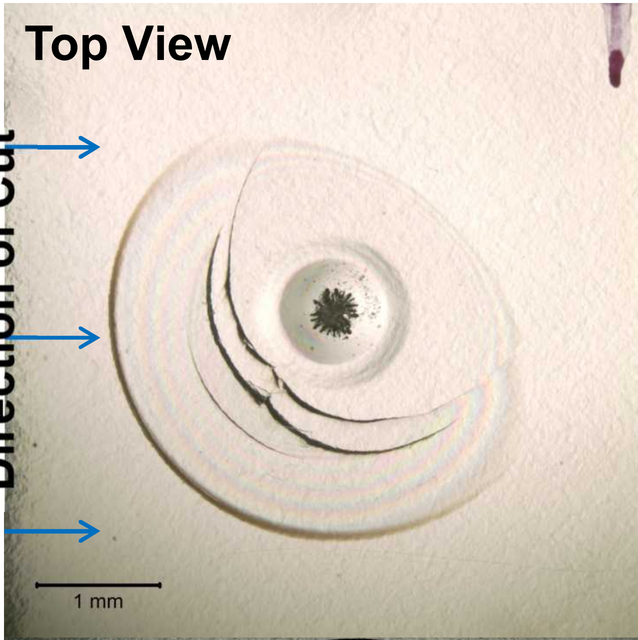
For 7a, delamination mean load at 582 N, while it is 440 N for 16B.
Model caution !

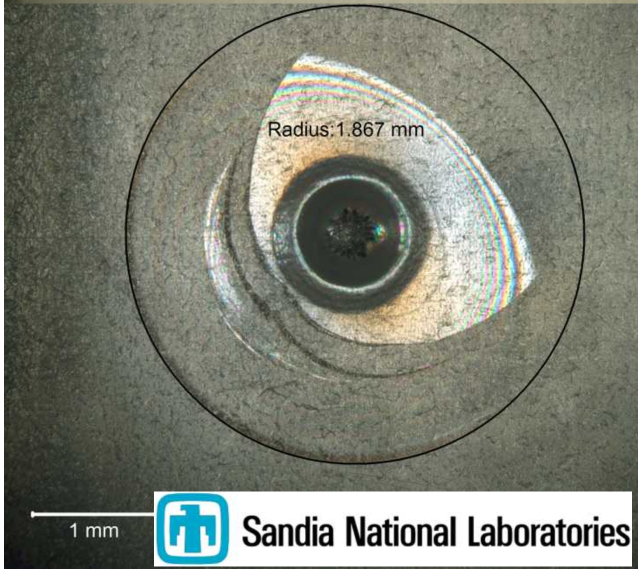
Smooth Glass- Partial Cone crack





Partial
Cone-Cracking





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