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Measurement of Interfacial Adhesion in Brittle Materials Using Indentation Fracture

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Sandia National Laboratories/NM, U.S. Department of Energy
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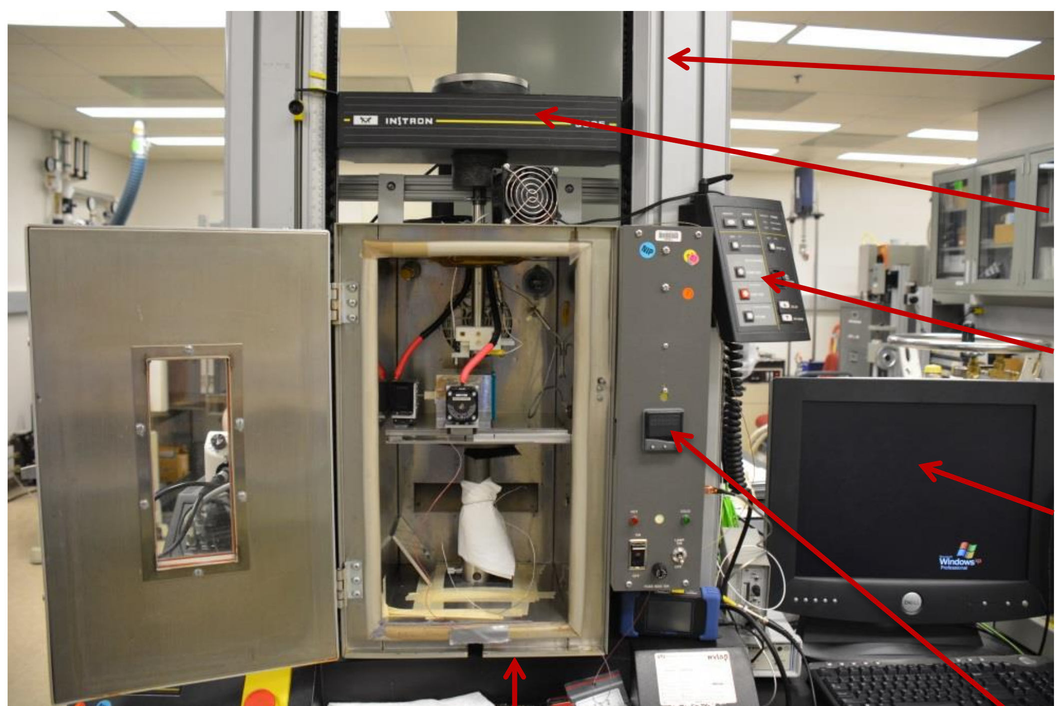
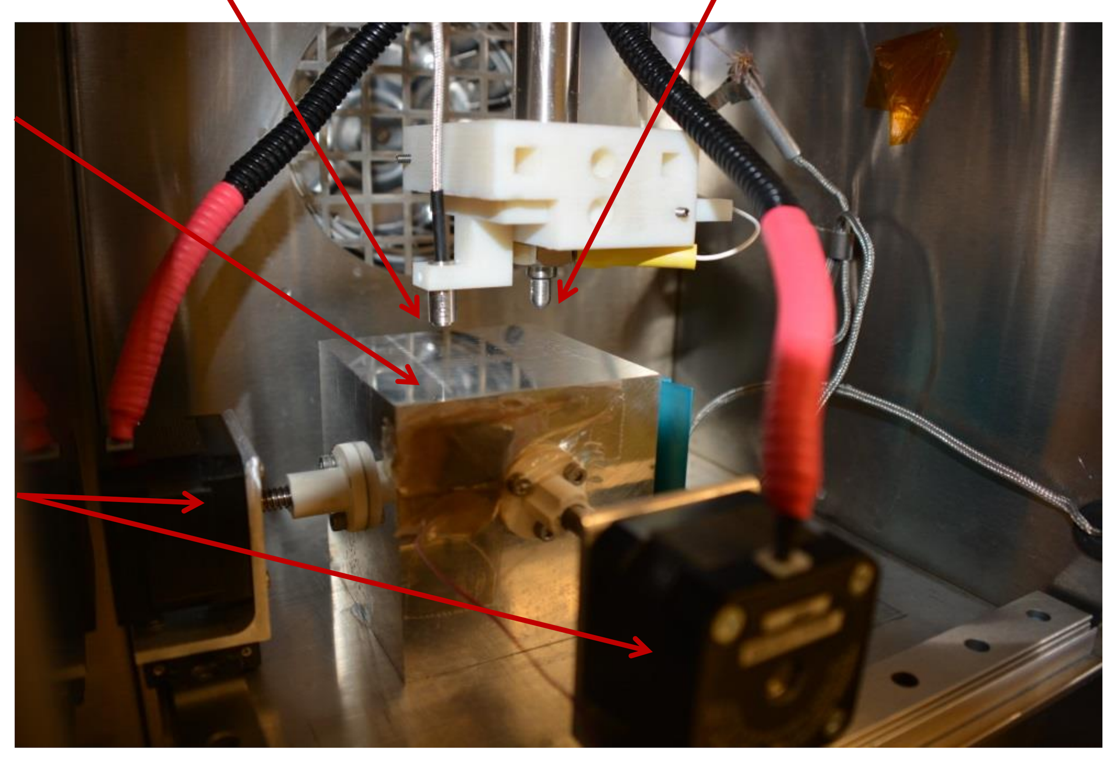
Objective: Calculate interfacial fracture energy of EPON 828 epoxy coatings on soda-lime glass substrates.

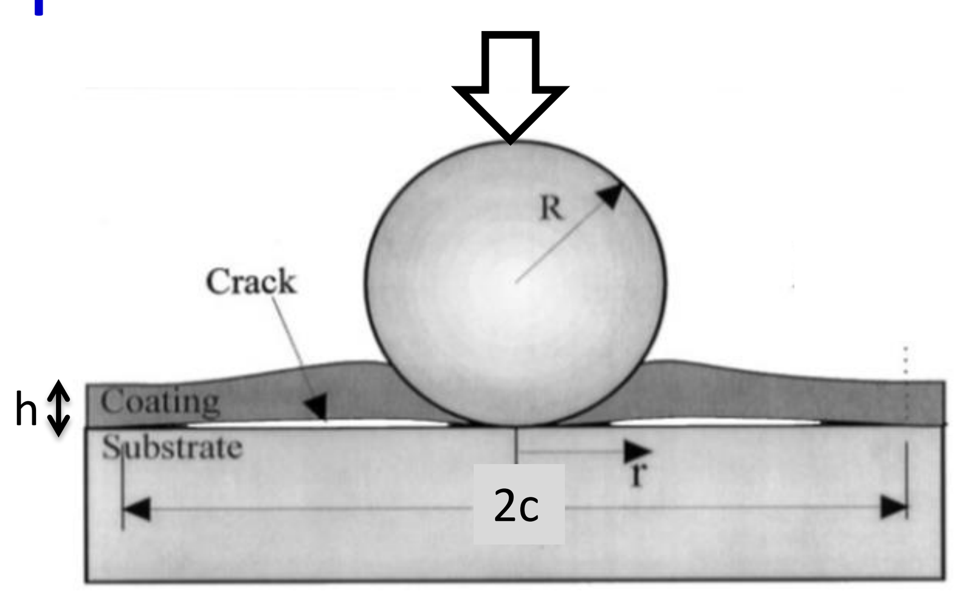
Instron 5565 with Environmental Chamber





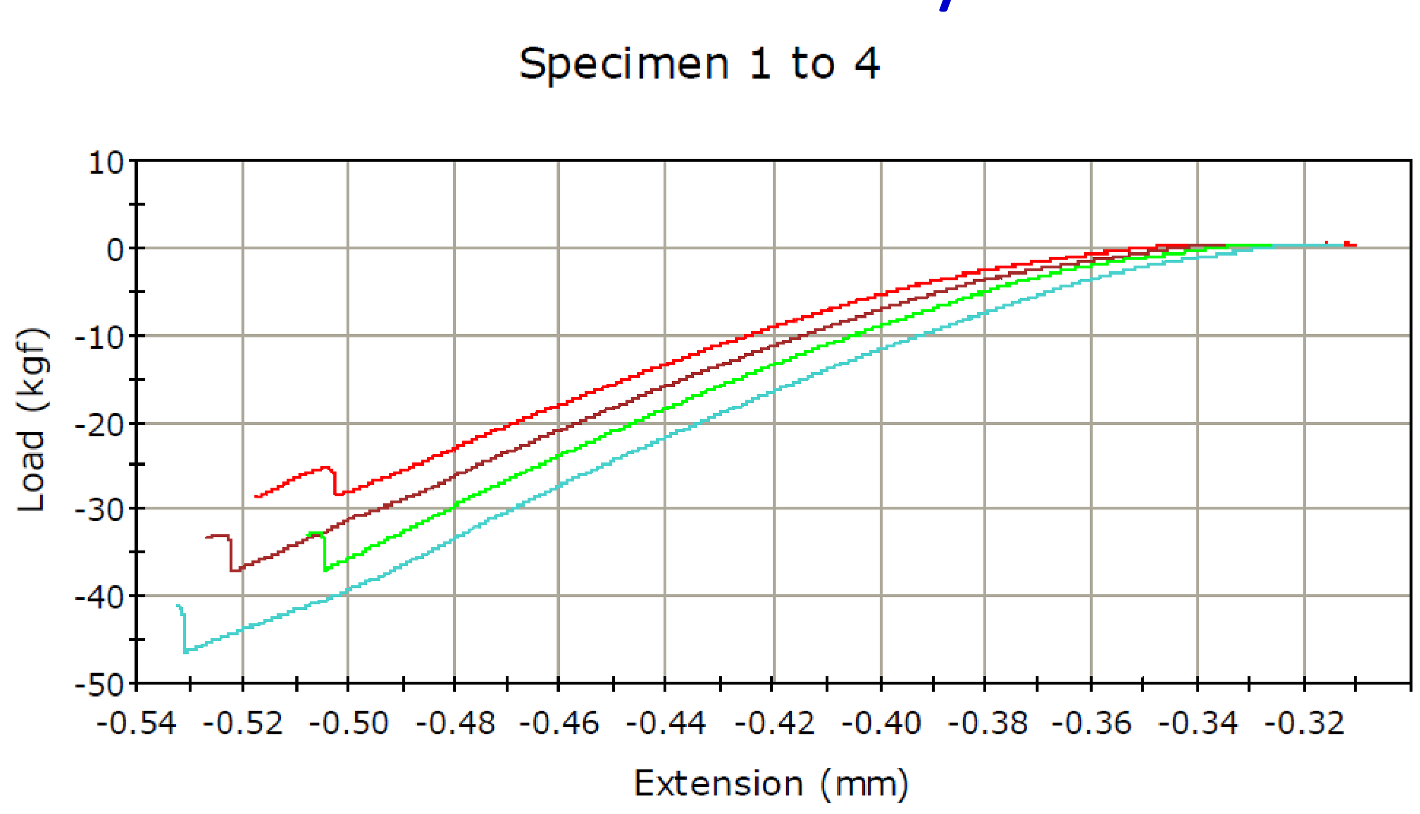
Indentations were performed with an Instron 5565. The indenter was attached to a load cell that was bolted to the bottom of the crosshead. A motor moved the crosshead down at a rate of 0.05 mm/min. An attached computer recorded the indenter load and crosshead displacement every 2 ms.

Spherical Indentation as a Method for Quantifying Interfacial Adhesion



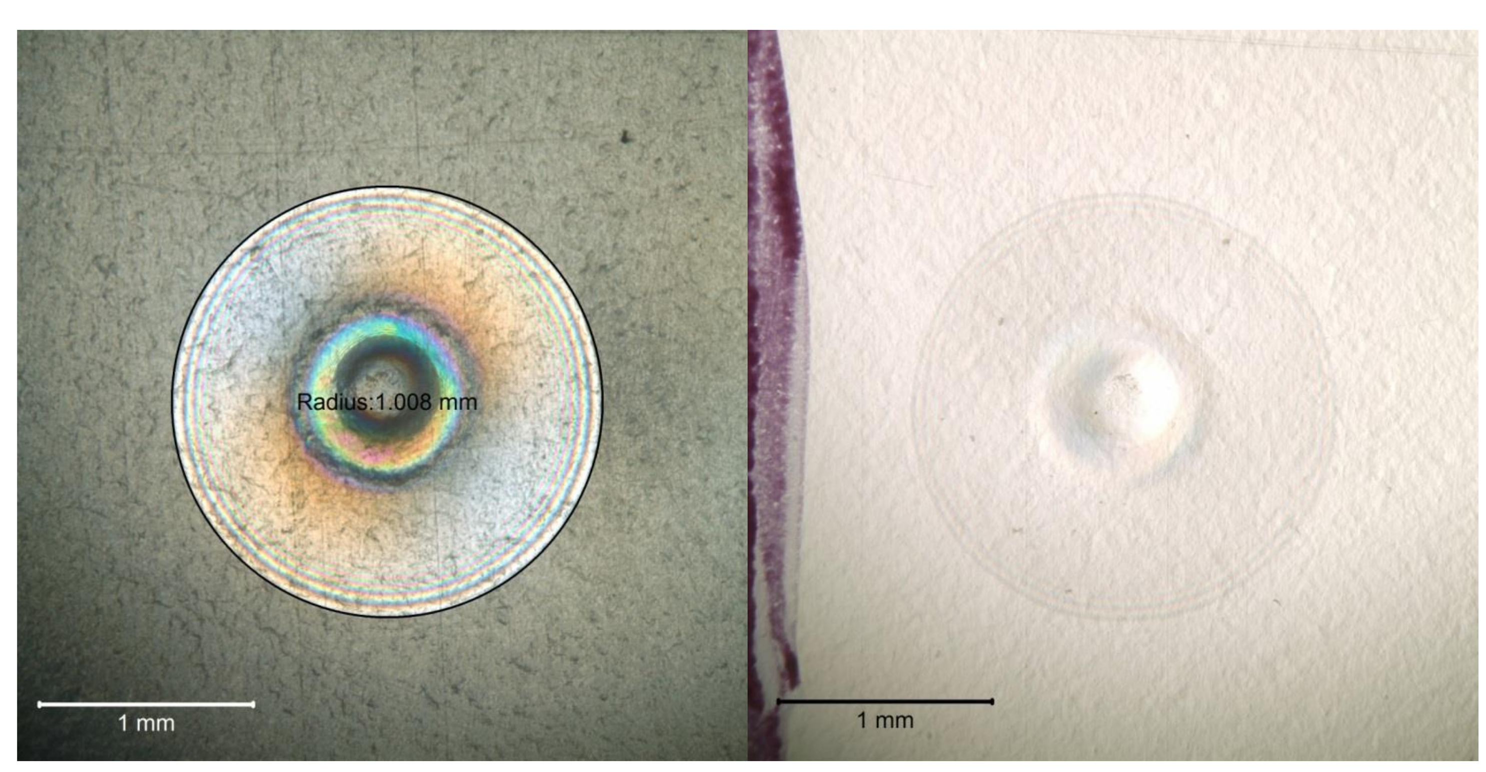
The coated surface of the sample was loaded with a WC spherical indenter. The coating was deformed and displaced laterally by the indenter. This lateral displacement produced a shear stress across the glass-epoxy interface and resulted in delamination of the epoxy coating from the glass substrate at sufficiently high loads. Indentations took place within an environmental chamber at -55°C in order to increase the residual tensile stress in the epoxy coating due to a thermal expansion mismatch and promote delamination.

Measurement of Crack/Delamination Size



When delamination occurs, there is a small and sudden drop in load. The sample is then unloaded and moved to perform the next indent.

Note: The load is compressive, therefore negative, and increases downward.

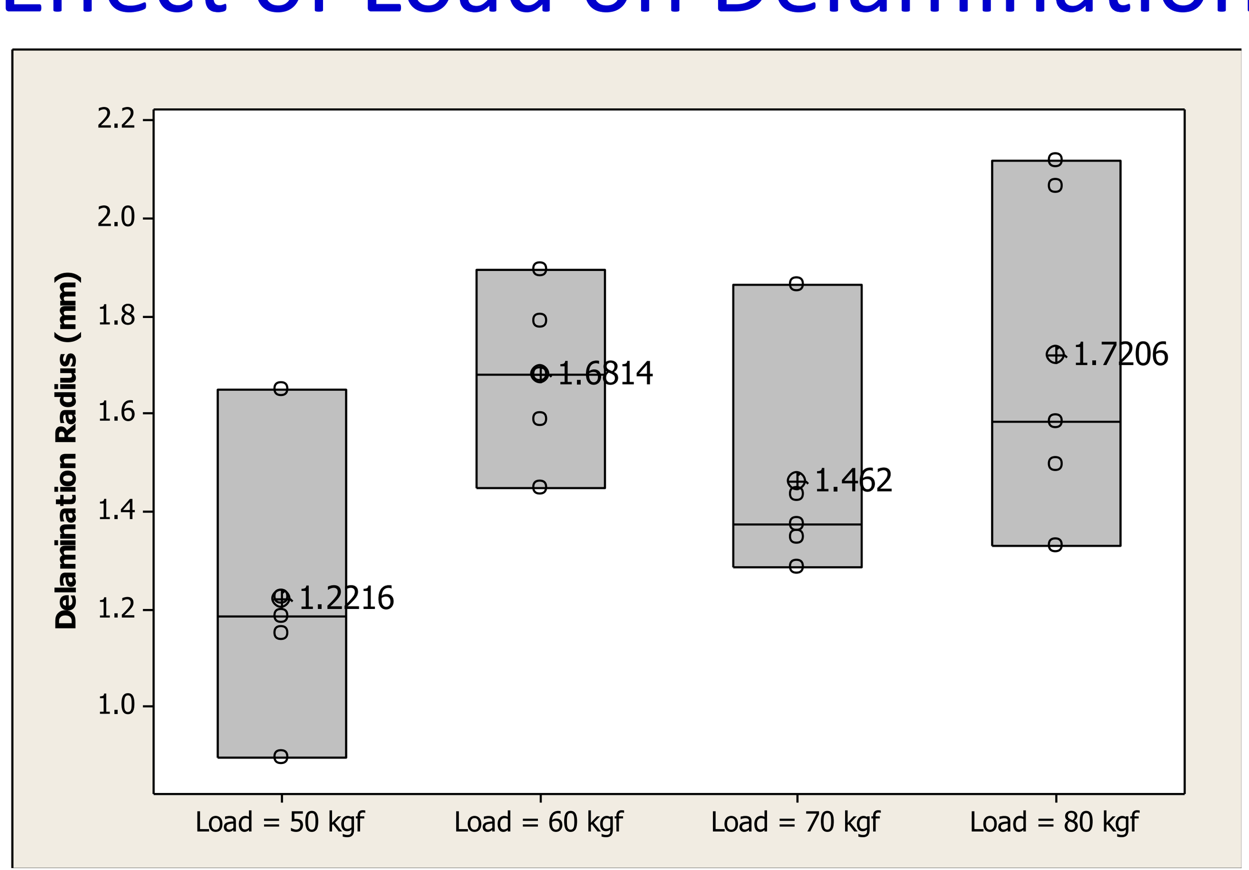


Reflected Light Transmitted Light

Delamination of epoxy coating from glass substrate.

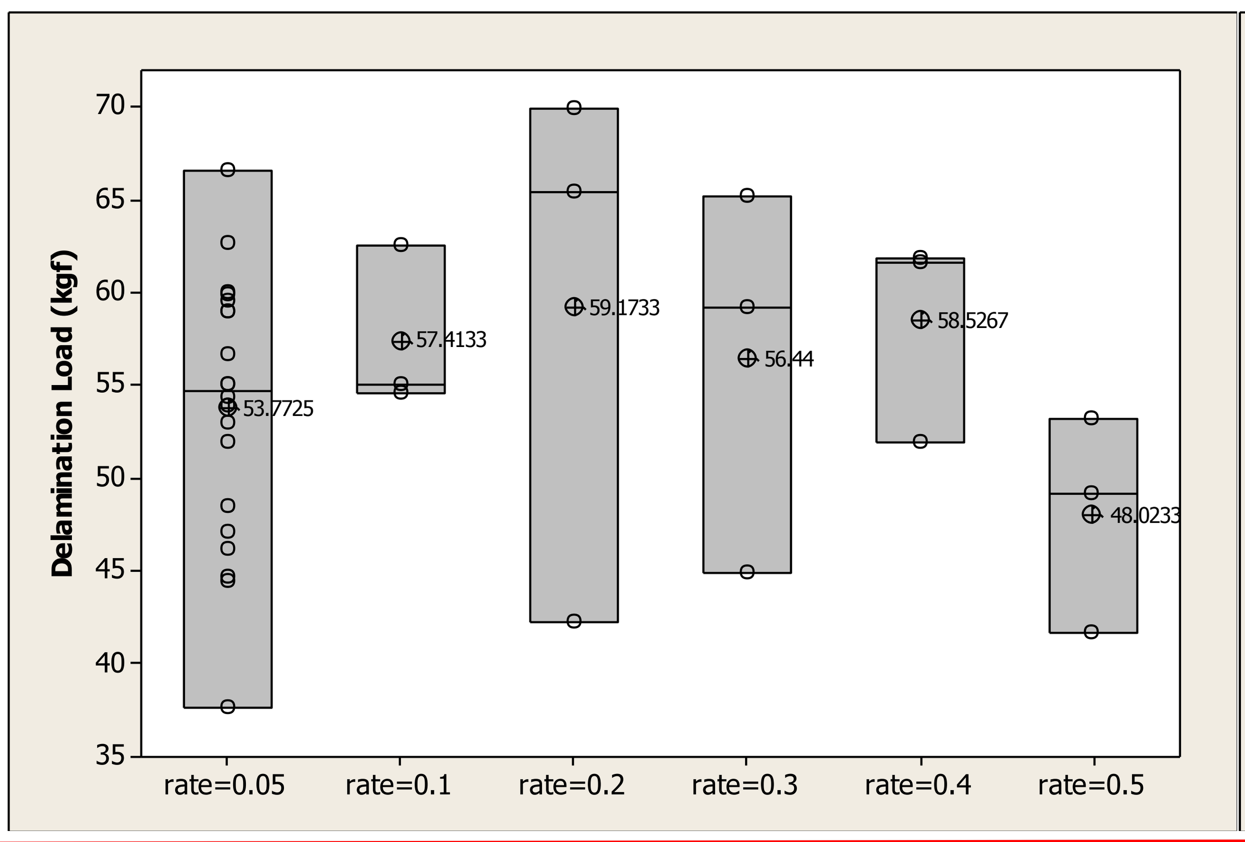
- 150 micron epoxy coating: average delamination load = 34 kg, average delamination radius = 1.042 mm
- 250 micron epoxy coating: average delamination load = 52 kg, average delamination radius = 1.471 mm

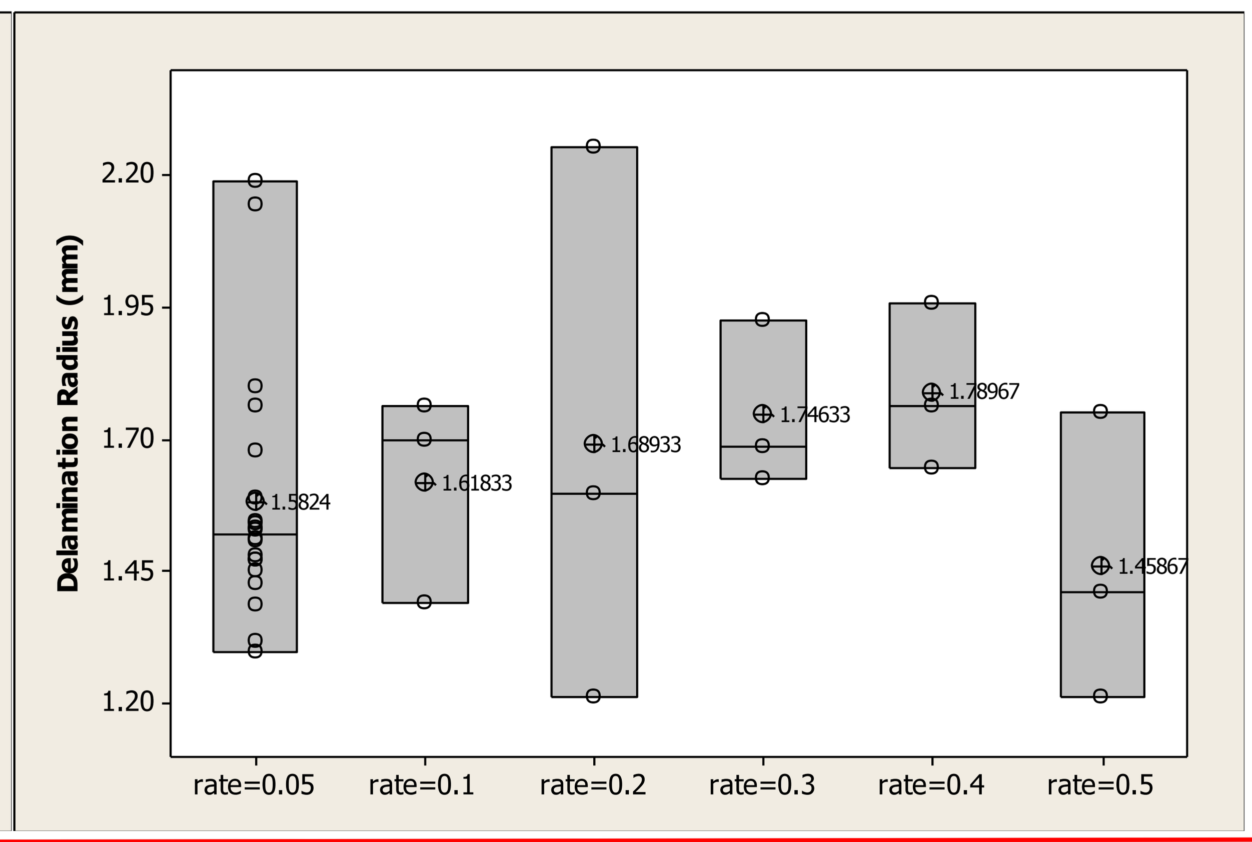
Effect of Load on Delamination Size



In general, delamination size increases with load.

Does crosshead/indenter speed have an effect on delamination?





Based on these data, indenter speed does not appear to have an effect on delamination.

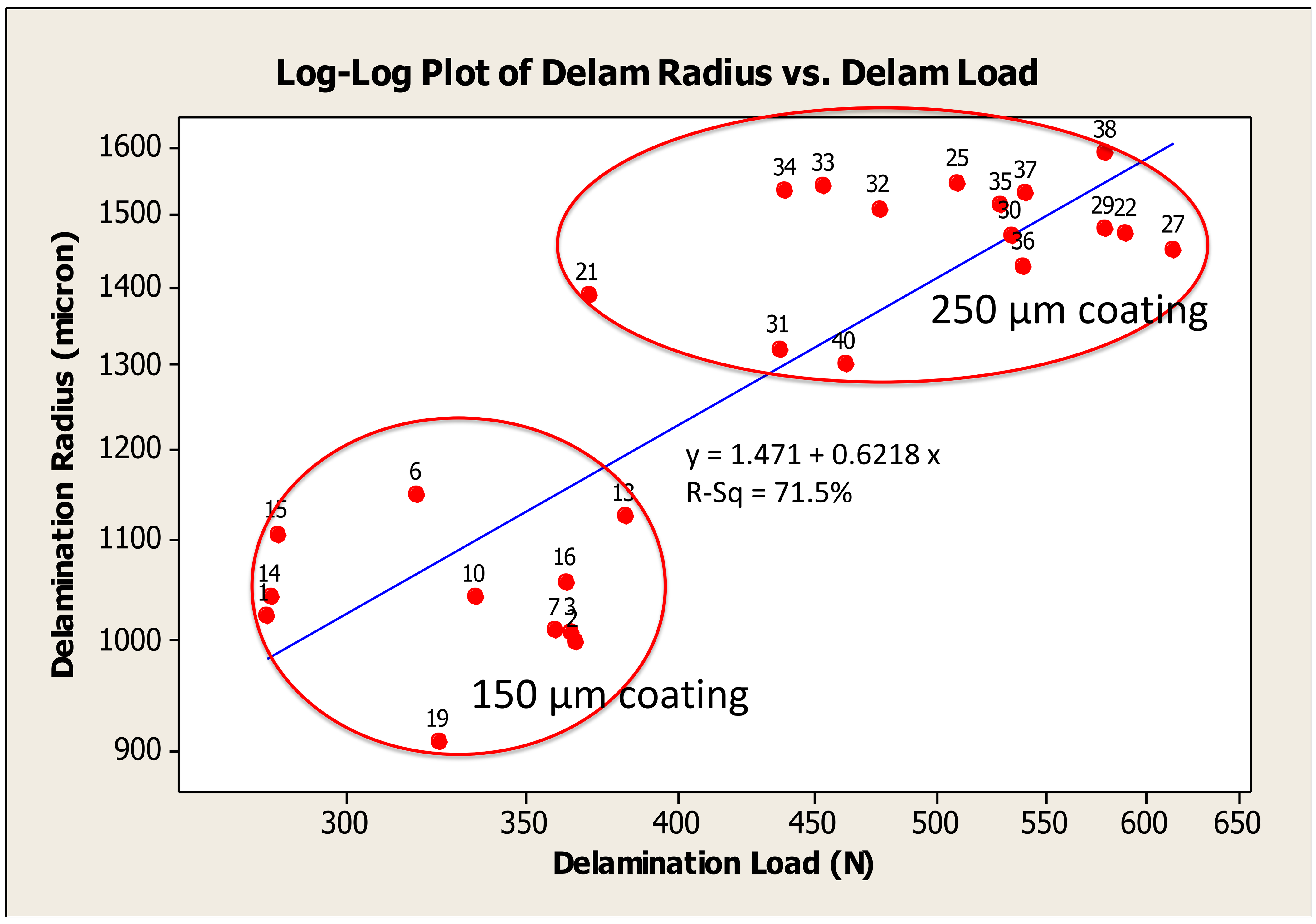
Use of microindentation technique for determining interfacial fracture energy. L.G. Rosenfeld, et al., J. Appl. Phys. 67, 3291 (1990).

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

- They calculate an average G = 25.2 ± 8.7 J/m^2
- They propose H and G are independent of P and c → $c \approx P^{1/2}$
- Slope = ½ on a log-log plot

G = interfacial fracture energy
P = indenter load
c = crack/delamination radius
h = thickness of epoxy coating
H = hardness of epoxy coating
E_c = elastic modulus of epoxy coating
ν_c = Poisson's ratio of epoxy coating

Results



- Slope of 0.6, not 0.5, but there is a lot of scatter in the data.
- Interfacial fracture energy: 877 J/m^2 for 150 µm coating and 946 J/m^2 for 250 µm coating…our adhesion is an order of magnitude greater than theirs! Our samples are better cleaned and/or our epoxy has much better adhesion? Test dirty samples?
- The understanding from these tests can be applied to mission critical work (alumina-epoxy adhesion studies). Scatter in data could be due to variability in technique and/or variability in sample surface. Glass is smooth → no variability in surface → variability is inherent in technique.

Future Work

- Perform indentation experiments on rough glass and compare results with smooth glass. Surface roughness should hinder delamination. Will the epoxy coating delaminate? Will the indenter penetrate through the epoxy coating and into the glass?
- Perform indentation experiments on stressed (tempered) glass and compare results with unstressed glass.
- Perform double cantilever beam and 4 point flexure beam experiments to calculate interfacial fracture energy and compare results with indentation method.

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NNSA National Nuclear Security Administration

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