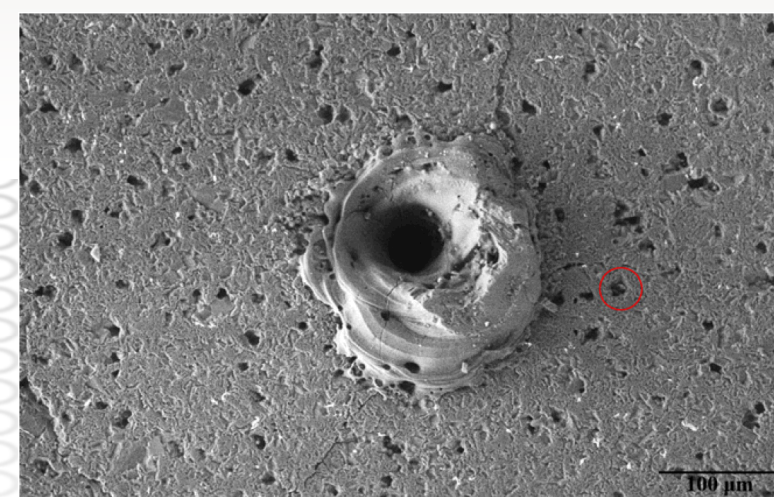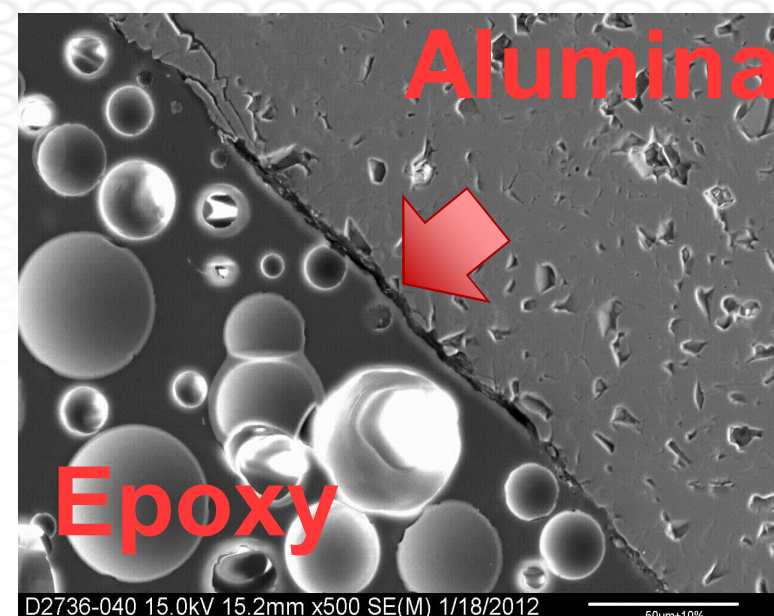# Measurement of Shear Strength and Interfacial Adhesion of Alumina-Epoxy Interfaces Using Indentation

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Exceptional service in the national interest

## Dielectric Breakdown in the Alumina-Epoxy System

Penetration of alumina due to dielectric breakdown is mitigated by the use of an epoxy coating, however delamination of the epoxy can lead to breakdown.

Methods for measurement of interfacial toughness & shear strength will help in:

-Identifying promising processing and materials modification that increase breakdown strength

-Quantification of Margins

Alumina
Epoxy

Spherical Indentation as a Method to Test Adhesion
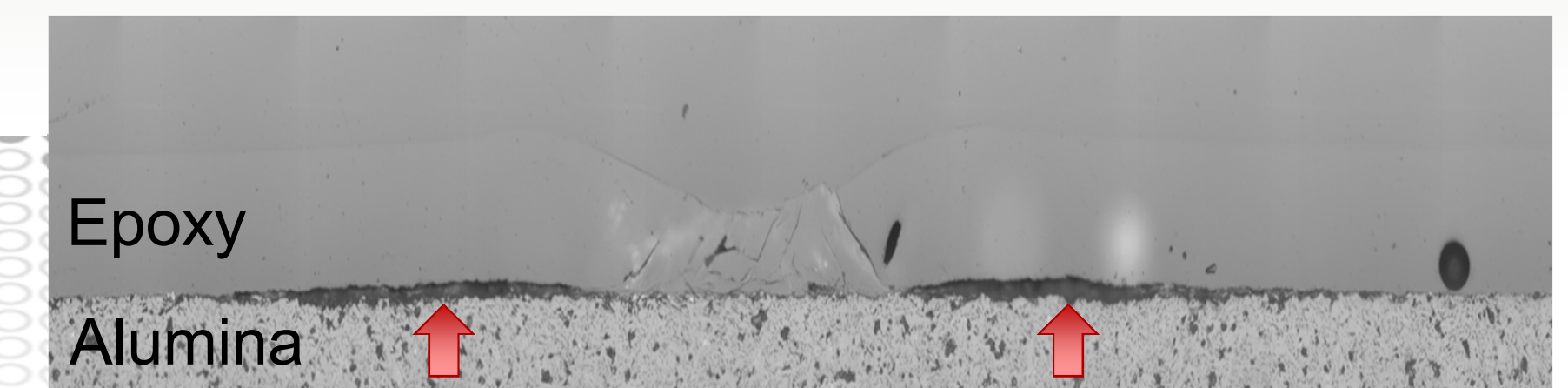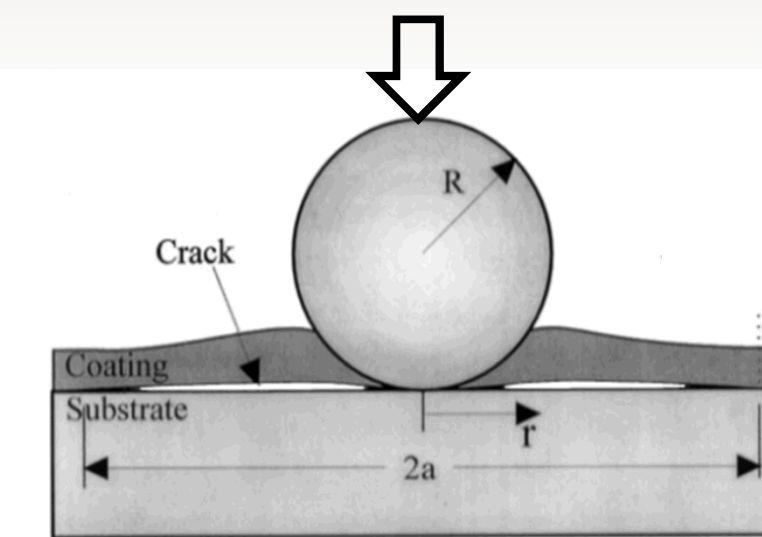
Epoxy
Alumina

Spherical indentation has previously been used to determine interfacial properties in glass-polymer systems. This method can also be applied to alumina-epoxy system. Indentation of a glass-epoxy system was performed as proof of concept. Delamination was observed at very high loads.
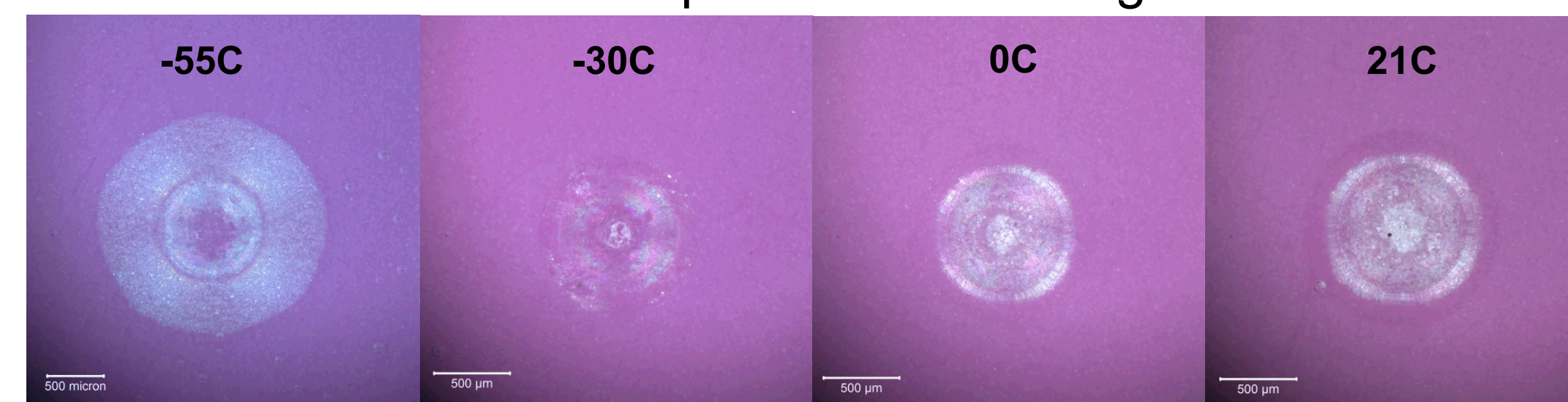
Indentation on Alumina-Epoxy
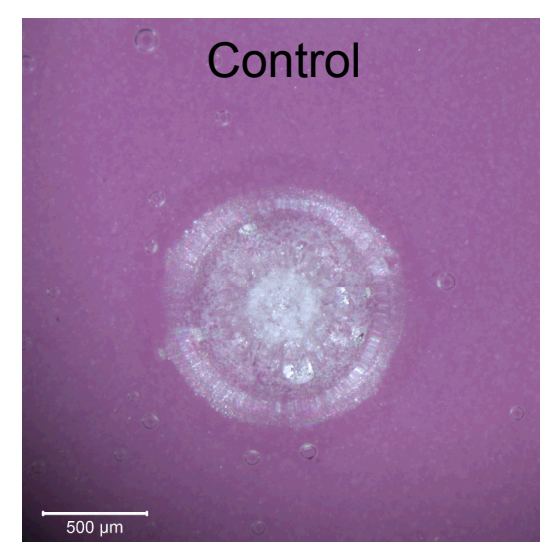
Control

Initial indentation on epoxy coated alumina at room temperature was unsuccessful in initiating delaminations due to the high interfacial toughness.

Lowering Temperature Enables Delamination

Control Sample Loaded to 50kg

-55C   -30C   0C   21C

Low temperatures increase residual stress in coating due to thermal expansion mismatch. This stress coupled with shear stress induced by the indenter leads to delamination.

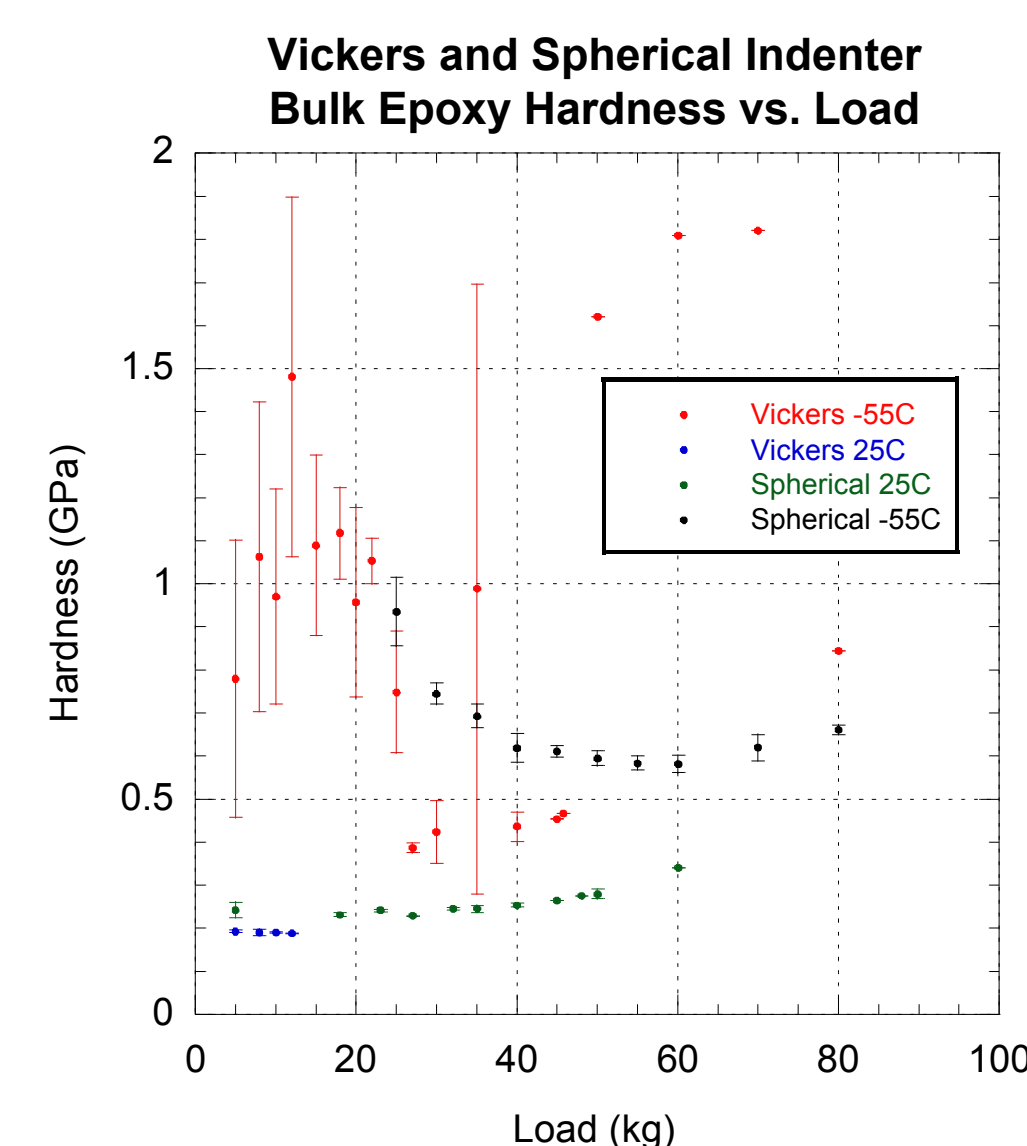## Hardness Testing For Interfacial Parameter Estimation

Vickers indenter:
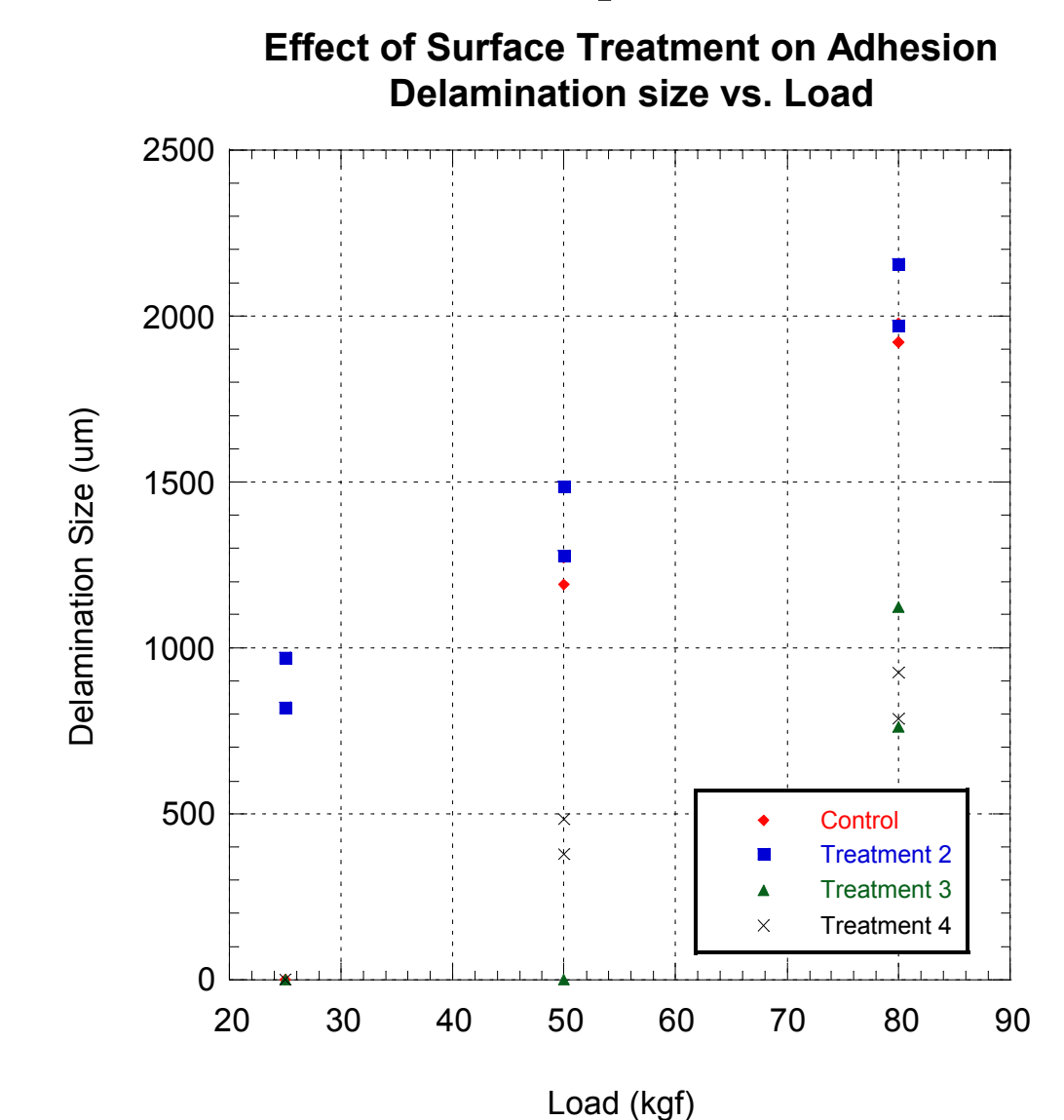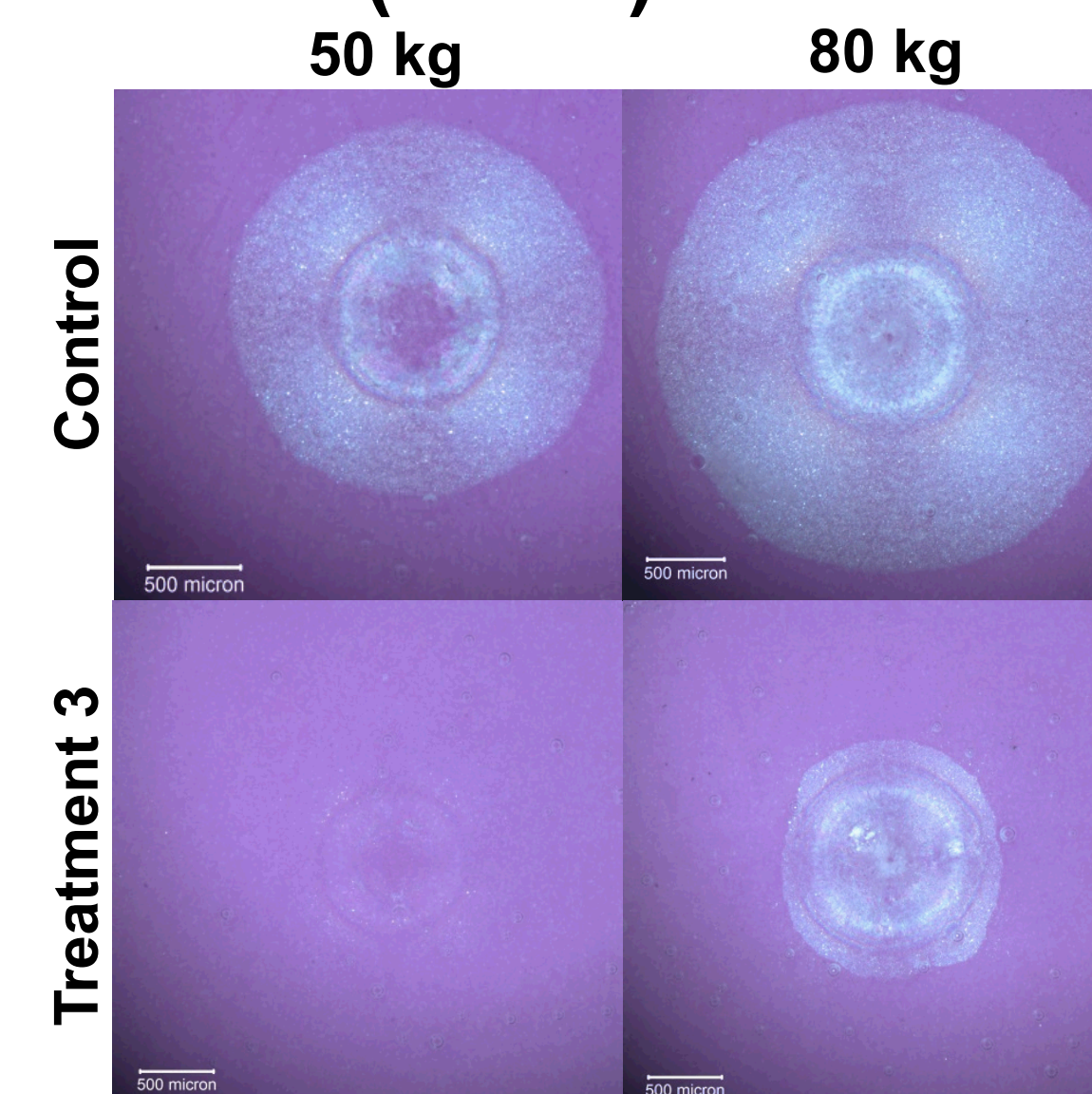
$$VDH = 1.86 \frac{P}{d^2}$$

For Spherical indenter:

$$H = \frac{P}{\pi R_c^2}$$

$$R_c = \sqrt{2 \cdot R_i \cdot h_p - h_p^2}$$

Vickers and Spherical Indenter
Bulk Epoxy Hardness vs. Load

Hardness (GPa) vs. Load (kg)

- Vickers -55C
- Vickers 25C
- Spherical 25C
- Spherical -55C

Accurate hardness of the epoxy must be obtained to calculate interfacial parameters from our work. Experiments were conducted in order to determine the hardness of epoxy in bulk and coated state.

Cold (-55°C) Indentation on Treated Samples

50 kg   80 kg

Control

Treatment 3

Effect of Surface Treatment on Adhesion
Delamination size vs. Load

Delamination Size (um) vs. Load (kgf)

- Control
- Treatment 2
- Treatment 3
- Treatment 4

Treatments 3 and 4: samples have high interfacial shear strength, and interfacial fracture toughness
The control and treatment 2 samples have poor interfacial characteristics.

Illustrative Calculations

Interfacial Fracture Toughness

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

G_c=interfacial fracture energy
~ 180-220 J/m²

Interfacial fracture energy » glass-epoxy energy (10-40 J/m²), and
adhesion energy (~1J/m²) obtained from wetting angle

Shear Strength

$$\tau_{critical} = \frac{-0.56H}{\dfrac{K_1'(\phi b_{crit}/h)}{\phi K_1(\phi b_{crit}/h)} + \dfrac{v_c h}{\phi^2 b_{crit}}} + \sigma_R$$

For unmodified surface, delamination at 50 kg, and none at 25 kg.
So shear strength is between 110 MPa-104 MPa

Future Work

1. Quantification of spherical indentation method results with micro-mechanical characterization, FEM

2. Alternate method utilizing the 4 point bend test confirm results obtained through indentation

3. Crack Deflection at interface may also be used to estimate interfacial toughness

P/2b   Notch   P/2b
Crack

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National Nuclear Security Administration