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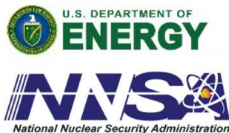
Interfacial Toughness: Effect of Surface Roughness and Test Temperature

Dave Reedy and Mark Stavig

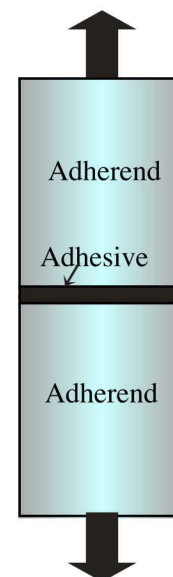
edreedy@sandia.gov

Sandia National Laboratories
Albuquerque, NM

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- It is well known that interfacial roughness can and test temperature can effect adhesively bonded joint strengths.
- For example, in previous tests the strength of adhesively bonded, cylindrical butt joints.
 - increased 40-80% increase as R_q increased from 1 to 5 μm .
 - increased $\sim 30\%$ as test T was decreased from RT to -60C .
 - tests performed far below the adhesive's T_g , and failure initiated at the interface along a segment of the specimen periphery.
- Significant testing burden if one wants to characterize the effect of R_q and T on interfacial Γ .
 - can one identify any useful scaling laws that might mitigate this burden?



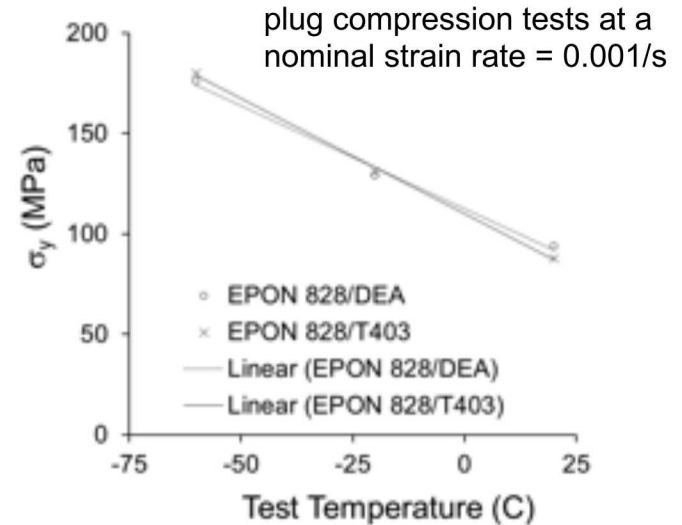
Materials: two epoxy materials

EPON® Resin 828 cured with DEA (diethanolamine)

- 100:12 pbw mix ratio.
- $T_g = 70\text{ }^\circ\text{C}$

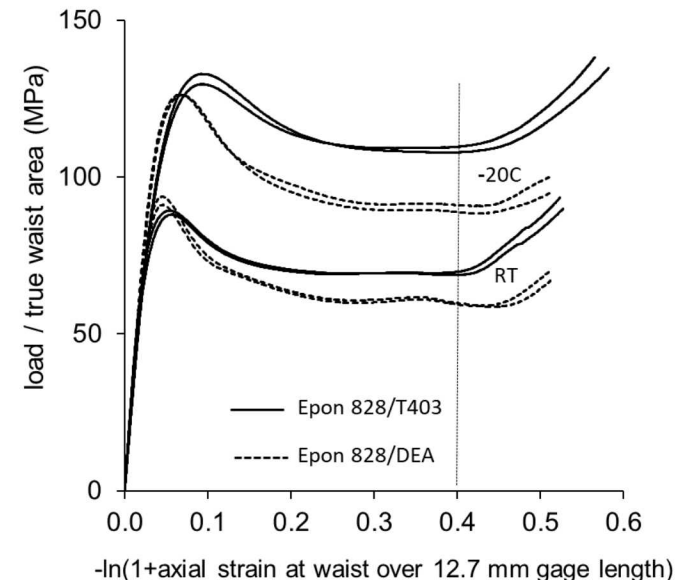
EPON® Resin 828 cured with Jeffamine® T-403 (polyetheramine)

- 100:43 pbw mix ratio.
- $T_g = 85\text{ }^\circ\text{C}$
- Can converted test temperature to epoxy yield strength (linear relationship between σ_y and T)

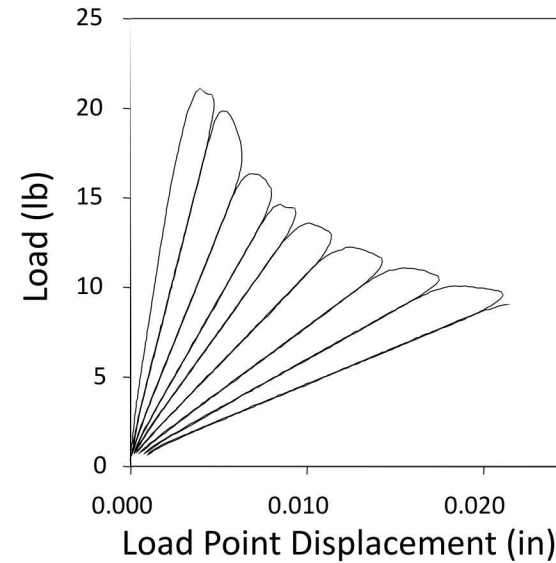
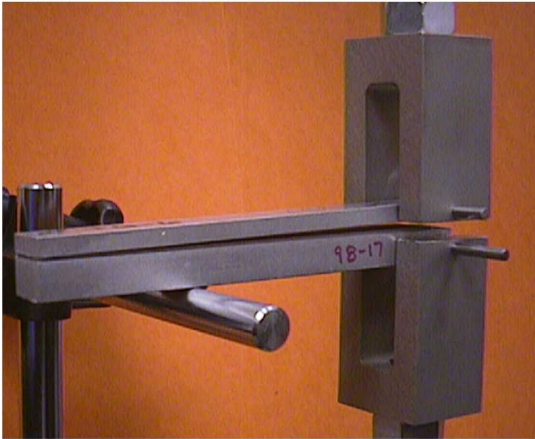


Also measured large strain compression data

- strain hardening begins at $\sim \epsilon = \epsilon_h > 0.4$, and is not strongly dependent on σ_y .
- Intrinsic strain softening can generate strain localization that appears similar to the Luder's bands generated in annealed mild steel.



Asymmetric Double Cantilevered Beam Sandwich specimen



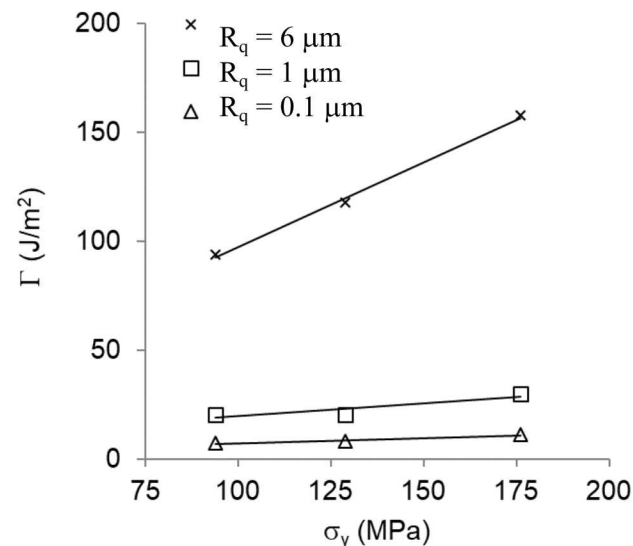
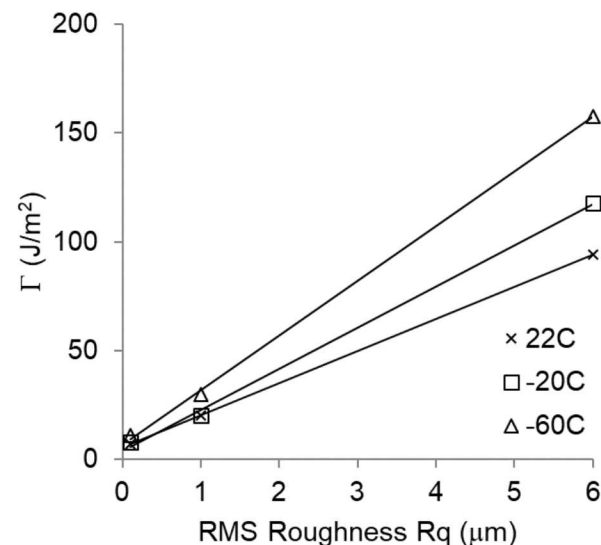
- Can make multiple Γ measurements per specimen (crack propagates stably).
- Use unloading compliance to determine crack length.
- Produces a predominantly Mode I crack-tip loading with a slight tendency to push the crack towards the interface ($\Psi_{l=10\mu\text{m}} = -15^\circ$ for a 0.5 mm bond).

Dependence on surface roughness and test temperature

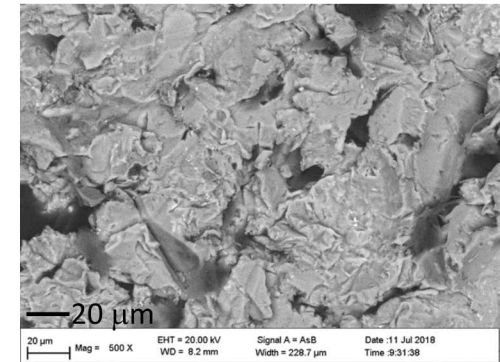
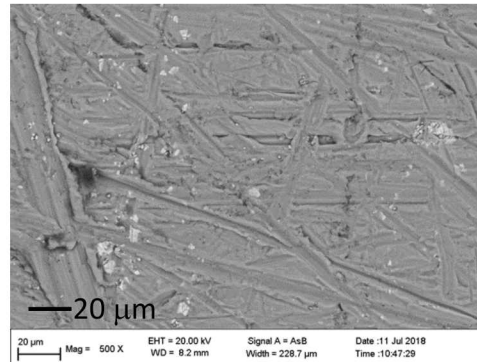
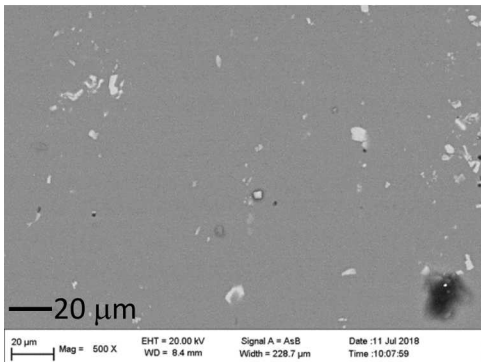
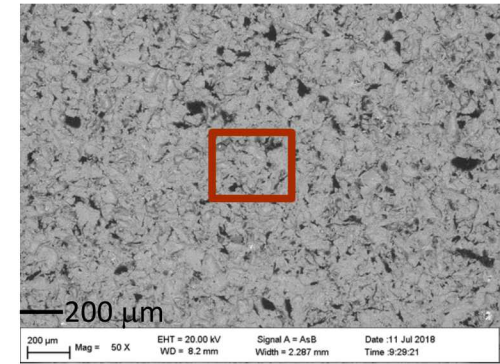
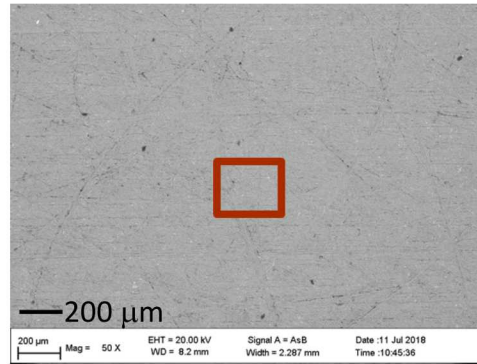
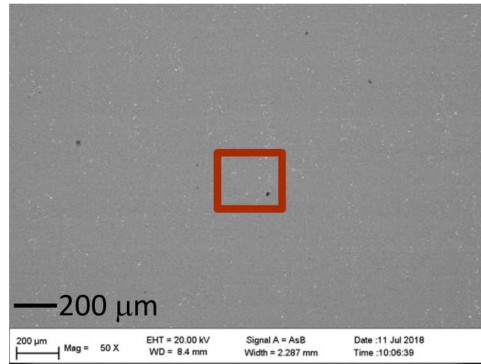
EPON 828/DEA epoxy-to-6061 aluminum interface

T (°C)	σ_y (MPa)	R_q (μm)	$\sigma_y R_q$ (N/mm)	Γ (J/m ²)	St Dev Γ (J/m ²)	# data pts
23	94	0.1	0.01	7.3	0.6	7
23	94	1	0.09	20.5	1.4	7
23	94	6	0.56	94	4.3	5
-20	129	0.1	0.01	7.9	0.7	8
-20	129	1	0.13	20.1	1.0	10
-20	129	6	0.77	117.6	1.4	5
-60	176	0.1	0.02	11.1	1.3	14
-60	176	1	0.18	29.8	3.1	10
-60	176	6	1.06	157.7	10.3	15

- Γ varies \sim linearly with σ_y when R_q is fixed and $\Gamma \sim$ linearly with R_q when σ_y is fixed.
- > factor of 10 increase in Γ when R_q is increased from 0.1 μm to 6 μm .
- > 40 % increase in Γ when test temperature is decreased from 22°C to -60°C.



EPON 828/DEA epoxy-to-6061 aluminum fracture surfaces



Note: backscatter SEM images. Used EDS to identify al/epoxy regions. Then image thresholding where black=> carbon

Polished

- $R_q < 0.1 \mu\text{m}$
- <1% carbon on aluminum fracture surface

Hand roughened using a Scotch-Brite™ pad

- $R_q \sim 1.0 \mu\text{m}$
- ~2-6% carbon on aluminum fracture surface

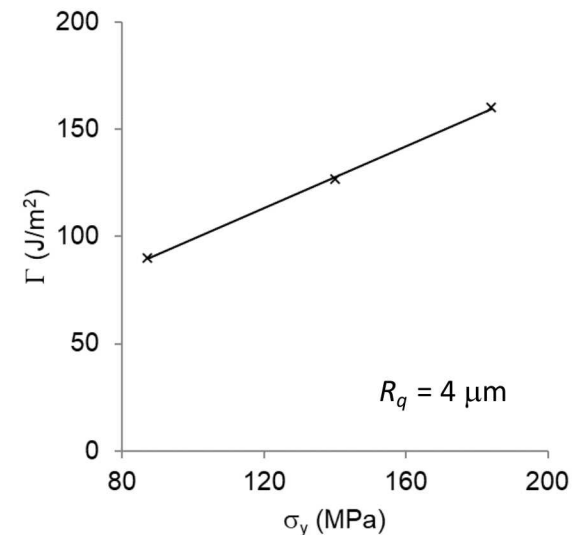
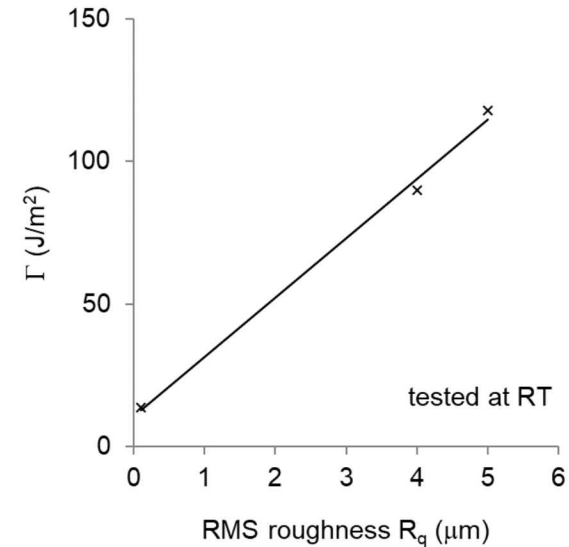
Grit blasted

- $R_q \sim 6.0 \mu\text{m}$
- ~7-8% carbon on aluminum fracture surface

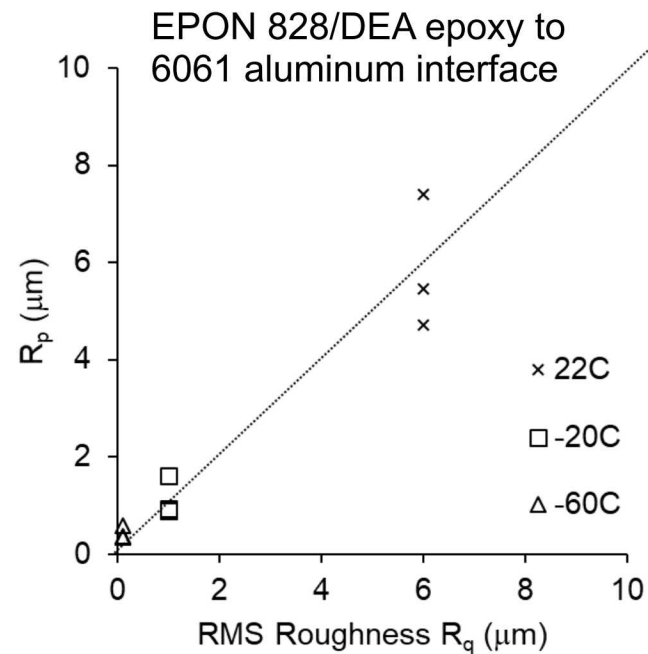
Dependence on surface roughness and test temperature EPON 828/T403 epoxy-to-6061 aluminum interface

T (°C)	σ_y (MPa)	R_q (μm)	$\sigma_y R_q$ (N/mm)	Γ (J/m ²)	St Dev Γ (J/m ²)	# data pts
23	87	0.1	0.01	13.7	0.9	13
23	87	4	0.35	89.9	4.6	8
23	87	5	0.44	117.9	7.3	15
-25	140	4	0.56	126.8	4.4	10
-65	184	4	0.74	160.3	7.6	12

- Dependence similar to that measured for the EPON 828/DEA epoxy to 6061 aluminum interface.
- > factor of 9 increase in Γ when R_q is increased from 0.1 μm to 5 μm .
- > 80 % increase in Γ when test temperature is decreased from 22°C to -65°C.



- Small Scale Yielding



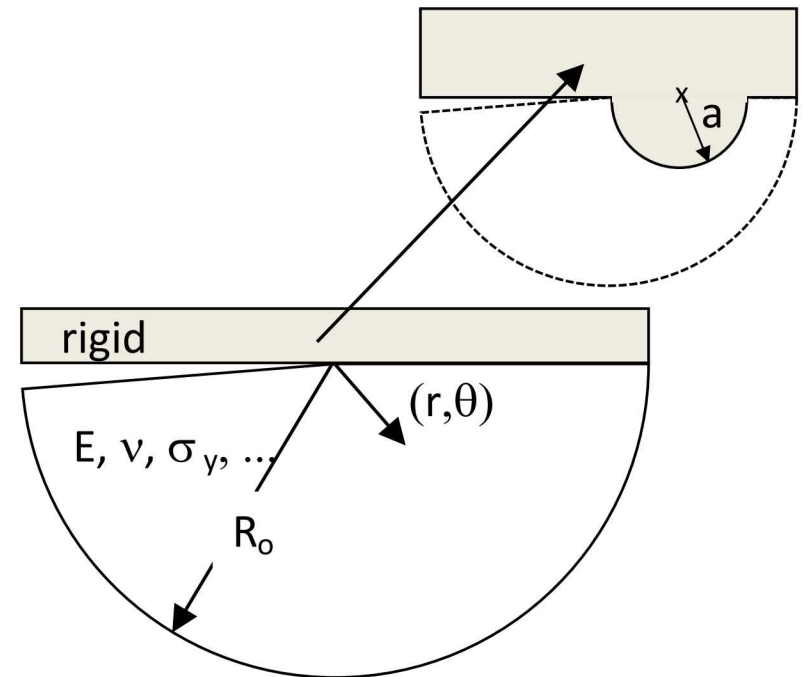
- Estimated plane strain crack-tip plastic zone size R_p for case of a rigid

$$R_p = \frac{1}{3\pi} \left[\frac{2\bar{E}\Gamma}{(1-\beta^2)\sigma_y^2} \right] \quad \text{where} \quad \beta = \frac{(1-2\nu)}{2(1-\nu)} \quad \text{and} \quad \bar{E} = \frac{E}{(1-\nu^2)}$$

- R_p is roughly commensurate with surface roughness R_q and much smaller than the 500 μm ADCB bond thickness, etc. (i.e., SSY applies).

Is there a scaling that relates Γ , R_q , and σ_y ?

- To gain insights, identified a simple illustrative problem.
- Upper material is rigid while lower material is elastic-plastic.
 - plastic yielding depends on the yield strength σ_y and other nondimensionalized properties.
- Includes a crack-tip “bump” of radius a that represents surface roughness (length scale).



Loading at $r = R_o$ is defined by the known linear elastic asymptotic crack tip fields so that it is consistent with a prescribed energy release rate G and crack-tip mode mixity $\psi_{r=R_o}$.

Is there a scaling that relates Γ , R_q , and σ_y ?

- Stevens published coarse-grained MD results for a cross-linked epoxy network bonded to a solid interface.
 - polymer strands between cross-links are not initially taut.
 - become taut only after relatively high average network strains.
 - interfacial bond failures occur only after the polymer strands between cross-links are pulled taut and the epoxy is hardening.

Stevens Macromolecules, Vol. 34, No. 8, 2001

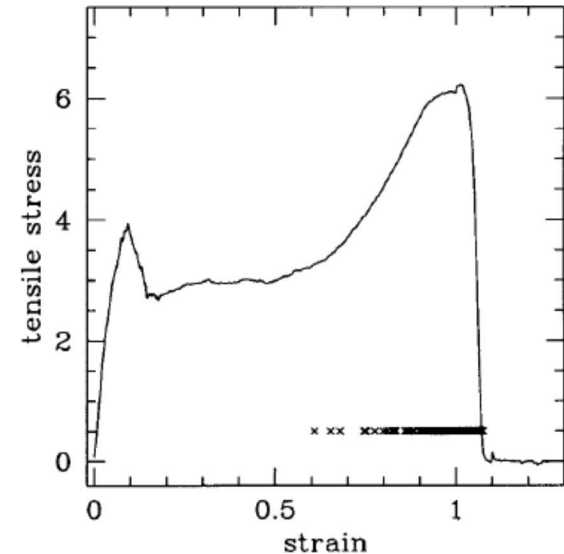


Figure 1. Tensile stress–strain curve for the fully bonded case of system 2. The points mark the strains at which bond breaking occurs. Stress is in LJ units.

Hypothesize interfacial separation initiates at the tip of an arrested interfacial crack when the plastic strain at the bump/epoxy interface exceeds the critical strain associated with rapid polymer hardening at large strains ($\varepsilon = \varepsilon_h \sim 0.5$, independent of σ_y).

Is there a scaling that relates Γ , R_q , and σ_y ?

- This idealized problem is defined by a small set of parameters.
 - based on dimensional considerations, strain $\hat{\varepsilon}$ at position \hat{r} along the epoxy/bump interface can be expressed as

$$\hat{\varepsilon} = f\left(\frac{\sigma_y}{E}, \frac{G}{\sigma_y a}, \frac{\hat{r}}{a}, \theta, \psi_{r=R_0}, \nu, \dots\right)$$

- if loading and material parameters are fixed and assume within the zone of very large plastic strains there is little dependence on σ_y/E then

$$\hat{\varepsilon} = f\left(\frac{G}{\sigma_y a}\right) \text{ along the bump/epoxy interface}$$

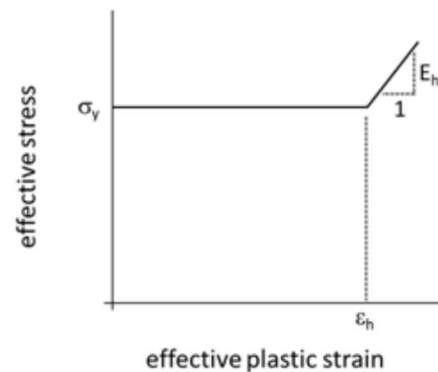
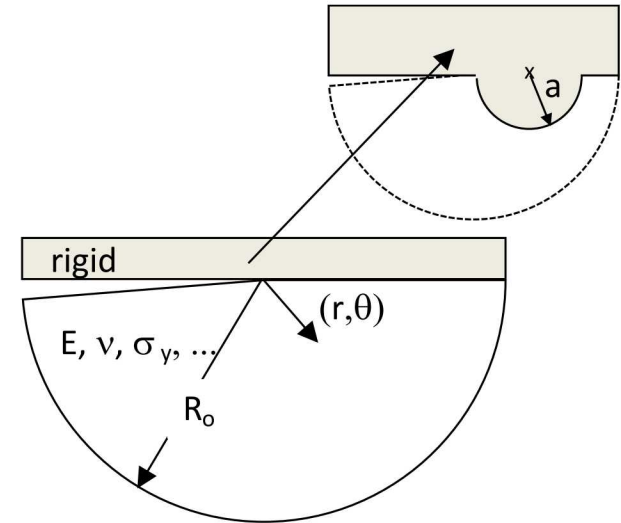
- the proposed criterion is $G = \Gamma$ and the crack propagates when $\hat{\varepsilon} = \varepsilon_h$
 - since ε_h is a constant (does not depend on σ_y) this implies that

$$\frac{\Gamma}{\sigma_y a} = a \text{ constant} \Rightarrow \Gamma \propto \sigma_y a$$

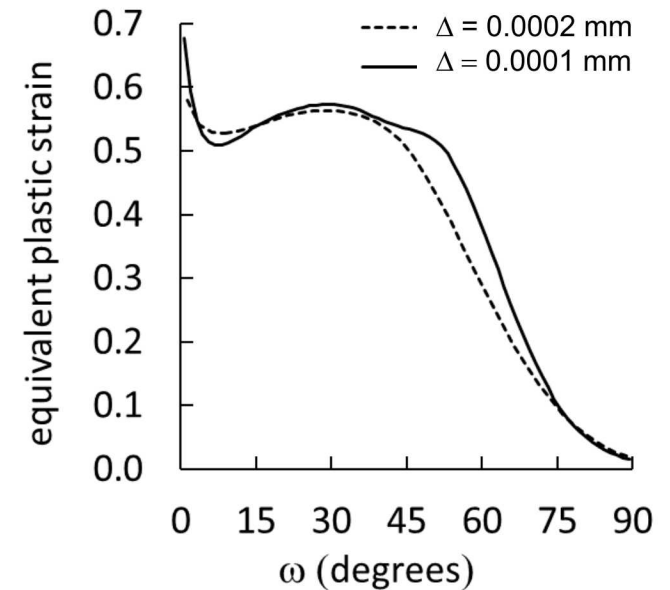
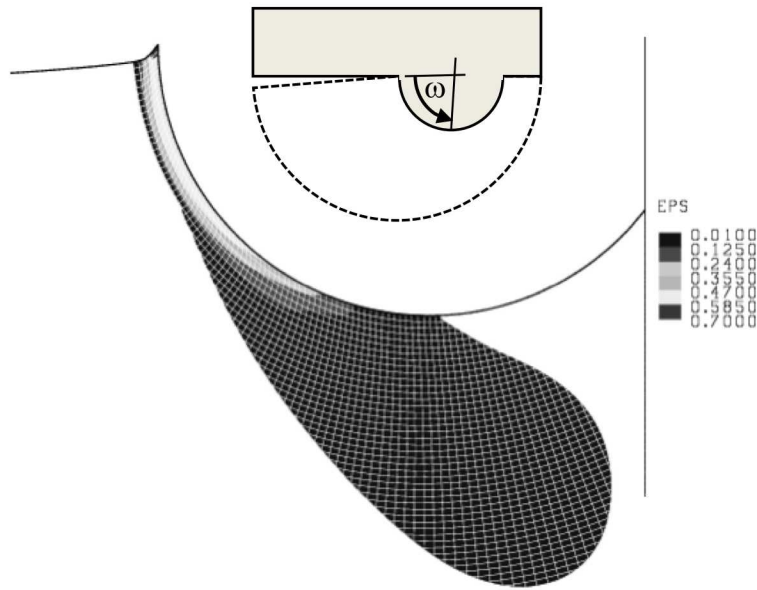
- Note the SSY Dugdale-Barenblatt strip yield zone model predicts $\Gamma \sim \sigma_y \delta_t$
 - a loose interpretation of the proposed scaling law is that the arrested interfacial crack propagates at a critical COD that is commensurate with roughness R_q .

Supporting FEA analysis to verify $\hat{\varepsilon} = f\left(\frac{G}{\sigma_y a}\right)$ relationship

- Analyzed the illustrative small-scale yielding problem.
 - loading at $r = R_o$ generates a G of either 50 or 100 J/m² and crack-tip mode mixity $\psi_{r=R_o=1mm} = -30^\circ$ (ADCB)
 - used a J2-plasticity model that includes hardening at large strain.
- $E = 3$ GPa, $\nu = 0.38$, $\sigma_y = 90$ or 180 MPa, $\varepsilon_h = 0.5$, $E_h = E/10$.



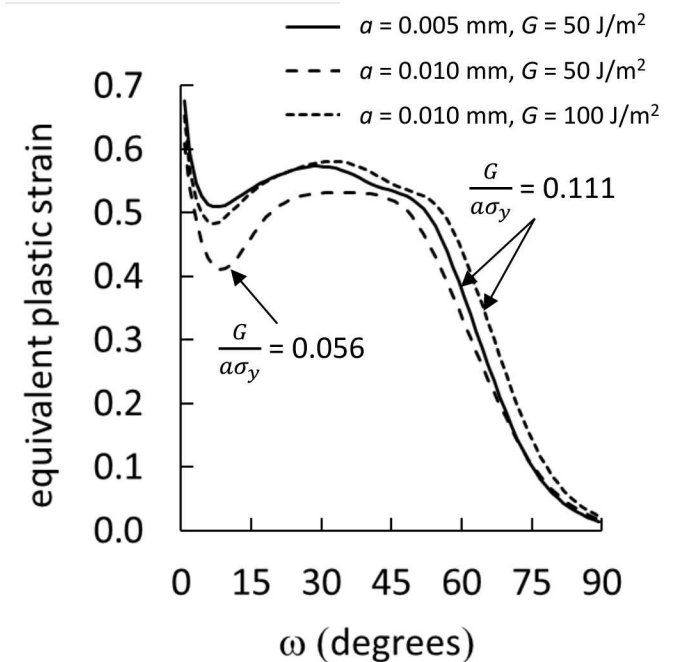
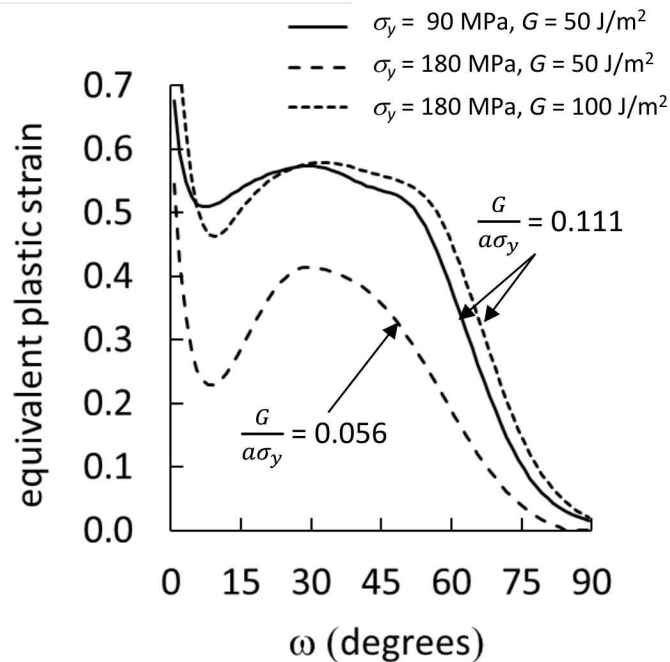
Supporting FEA analysis to verify $\hat{\epsilon} = f\left(\frac{G}{\sigma_y a}\right)$ relationship



- Equivalent plastic strain when $a = 0.005$ mm, $\sigma_y = 90$, $G = 50$ J/m²
- High equivalent plastic strain with strains $> 40\%$ is concentrated in a narrow band near the interface over a segment with $\omega < 60^\circ$.

- Plot equivalent plastic strain adjacent to the interface vs. arc length in degrees.
- Varied characteristic element size Δ to examine mesh sensitivity.
 - $a = 0.005$ mm, $\sigma_y = 90$, $G = 50$ J/m²
- FEA results show good agreement at high equivalent plastic strain levels with mesh refinement.

Supporting FEA analysis to verify $\hat{\epsilon} = f\left(\frac{G}{\sigma_y a}\right)$ relationship



- Varied σ_y and G .
 - $a = 0.005$ mm
- FEA results show good agreement at high equivalent plastic strain when $\frac{\Gamma}{a\sigma_y}$ is fixed.
 - consistent with proposed scaling.

- Varied a and G .
 - $\sigma_y = 90$ MPa
- FEA results show good agreement at high equivalent plastic strain when $\frac{\Gamma}{a\sigma_y}$ is fixed.
 - consistent with proposed scaling.

Is there a scaling that relates Γ , R_q , and σ_y ?

- Dimensional analysis of simple illustrative problem with $\varepsilon = \varepsilon_h$ criterion suggests

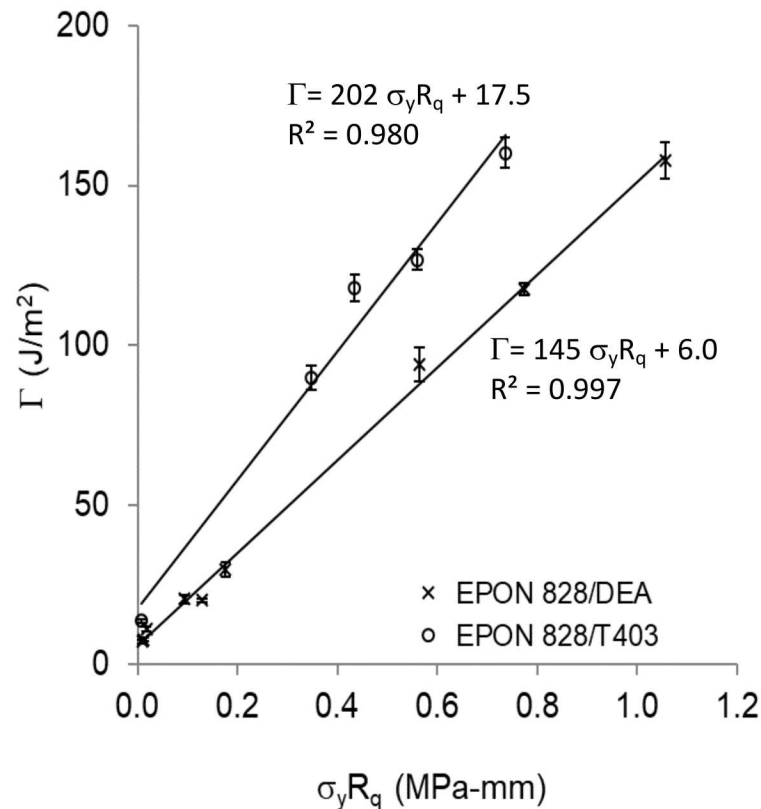
$$\Gamma \propto \sigma_y a$$

- Based on this result, propose the scaling relationship

$$\Gamma = \Gamma_0 + C \sigma_y R_q$$

- replace a with the measure of surface roughness R_q used in our experiments.
- C is a constant (i.e., independent of σ_y and R_q) that depends on the nature of the particular epoxy/solid interface.
- add Γ_0 since at zero roughness there will be an intrinsic toughness associated with a perfectly smooth interface.
- if this scaling relationship holds, my toughness data should plot as a linear relationship between Γ and the compound variable $\sigma_y R_q$.

Comparison of EPON828/DEA or EPON828/T403 epoxy data with proposed scaling relationship



- Measured interfacial toughness data is in excellent agreement with the proposed scaling.
 - bars indicating their 95% confidence intervals
 - slope of the Γ vs. $\sigma_y R_q$ line differs with the choice of epoxy adhesive.

- The proposed scaling $\Gamma = \Gamma_0 + C\sigma_y R_q$ suggests that one could perform tests that measure extreme values of $(\Gamma, \sigma_y R_q)$ and estimate intermediate values. Thus reducing the testing burden.
- Anticipate Γ will depend on loading rate in a similar way as observed for T .
- Under what conditions does the proposed scaling apply?
 - is it applicable to an epoxy that does not display post-yield softening followed by rapid hardening at large strains?
 - under what conditions does the observed dependency change? (e.g., transition to cohesive failure at high applied mode mixity?).