SAND2014-19813PE

Adhesive Joint Failure and the Aging of Adhesive Joints

TCG-XIV – Munitions Reliability and Lifecycle Technology

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All-TCG Technology Review: Rodeo 2014

Aberdeen Proving Grounds, MD

November 17-20, 2014

Sandia National Laboratories

National Nuclear Security Administration

Our Vision: Validated Model-Based Lifecycle Engineering for Packaging Design

J.M. Caruthers, et al., *Polymer*, 2004, 45, 4577
D.B. Adolf, et al., *Polymer*, 2004, 45, 4599
D.B. Adolf, et al., *Polymer*, 2009, 50, 4257

Polymer Nonlinear Viscoelastic (NLVE) Model

research physics → **development** tools → **analysis** predictions

Tuesday's Talk **Predict Stress/Strain and Understand Impact on Performance**

manufacturing
cure chemistry

How do we make it?
(Cure Chemistry)

thermal cycling

mechanical loading / aging

high rate

Current talk

Adhesive

failure

What can go wrong?
(Failure Metrics)

How does it perform?
(Constitutive Eqns)

Current Focus Areas

Recent Transitions

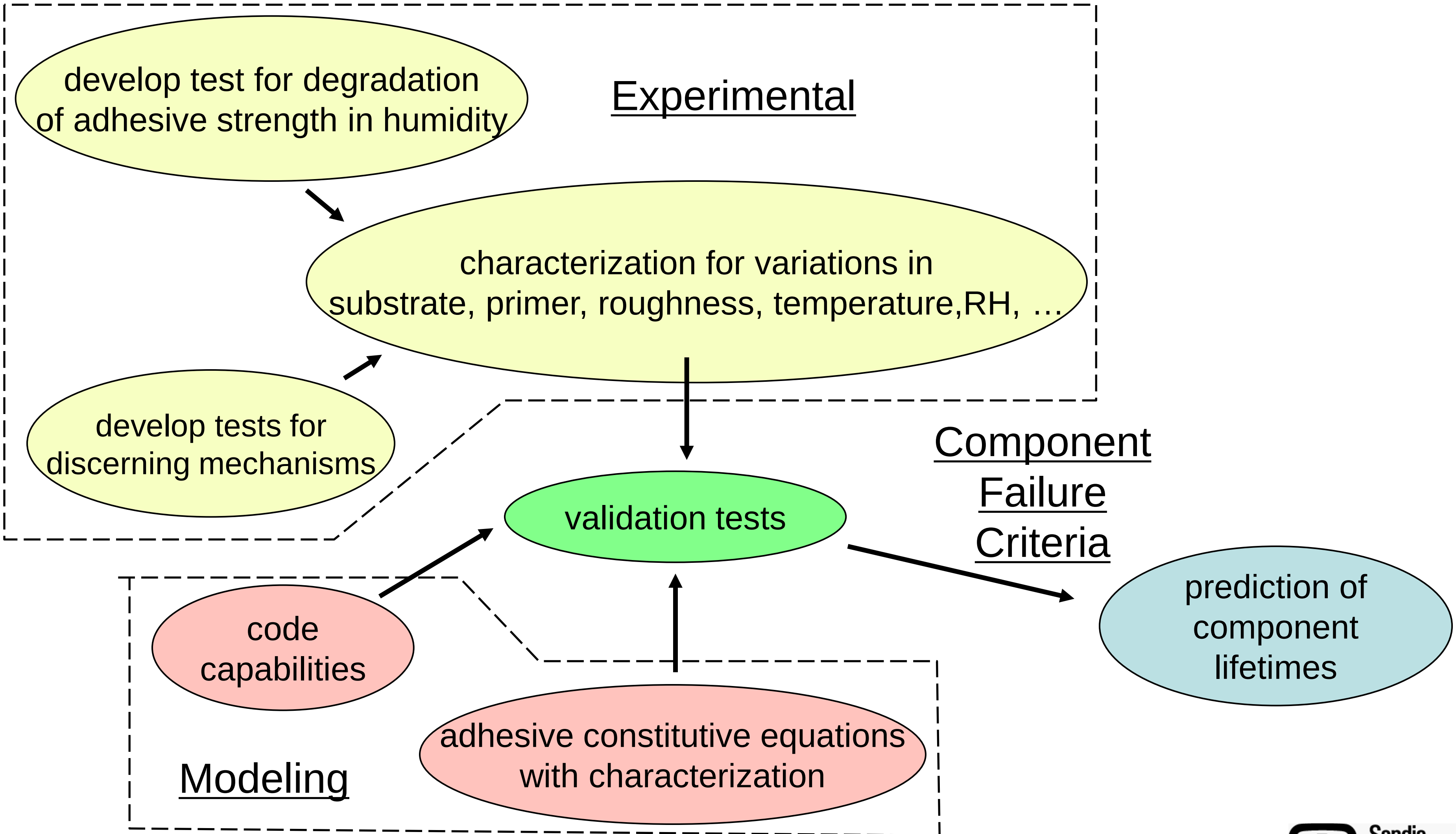
FY	Transition	Recipient
14	Technical exchange seminar on materials (adhesives/encapsulants) issues in Neutron Generators	Shawn Dirk/Deidre Hirschfeld (SNL, 2735/1832)
14	Assessment of contaminant effect on adhesive bond strength	Allen Roach/Shawn Dirk (SNL, 2735)
14	SAND2014-2920 report documenting work on HELLFIRE missile cradle railpad adhesive	Megan Shumate (Redstone Arsenal-AMRDEC)
14	Packaging design review for multiple NW components (ELNG, ISL/TSL, Connectors, Junction Box and Adapters)	Respective Product Realization Team Leads (SNL)
14	Demonstrated methodologies and specific cure schedules to reduce stress developed during encapsulation	Gary Pressly (SNL, 2732)
14	Assessment of adhesive material used in radar antenna in order to better understand design margins	Allison Routson (SNL, 5343)
14	Die attach material analyses to aid packaging materials choices for ball grid arrays	Steve Garrett/Ken Peterson (SNL, 1718/1833)

Discussion Topics

- Review of humid environment adhesive joint aging results

- Role of residual stress on strength of adhesive joints

Adhesive Joint Aging in Humid Environments: Big Picture



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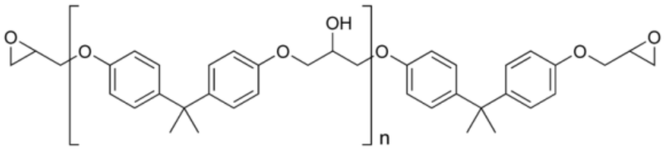
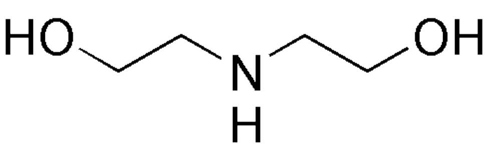
Adhesive Joints

<u>Adhesive: 828/DEA</u>

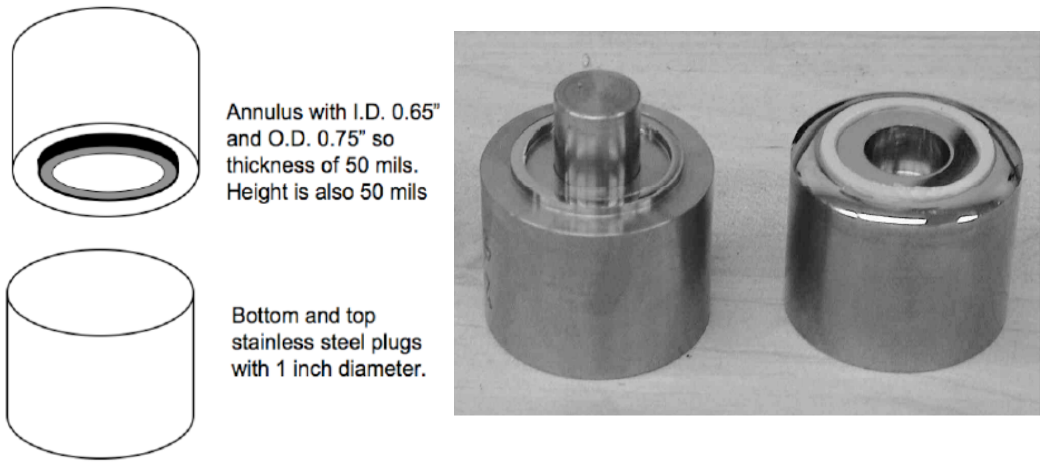
EPON® Resin 828
Diglycidylether of Bisphenol-A



Diethanolamine



<u>Test Geometry: Napkin-Ring</u>

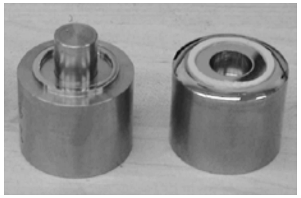


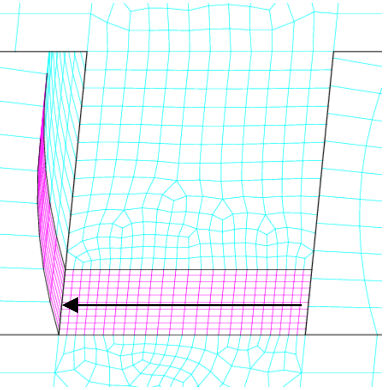
de Bruyne and Houwink (eds) *Adhesion and Adhesives*, Elsevier, London p.92 (1951)

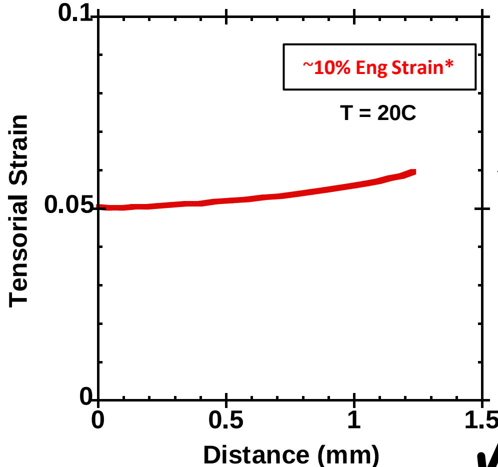
http://www.sandia.gov/polymer-properties/828_DEA.html

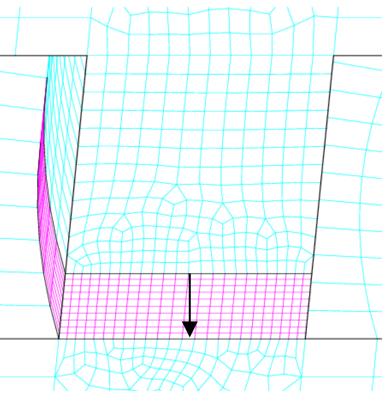
Develop a Straight-Forward Experimental Test

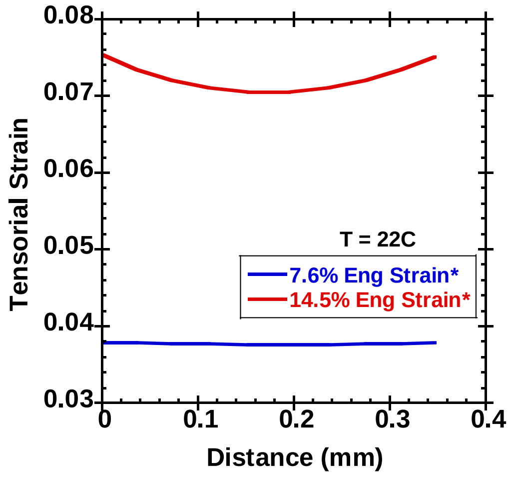
Napkin-Ring Geometry





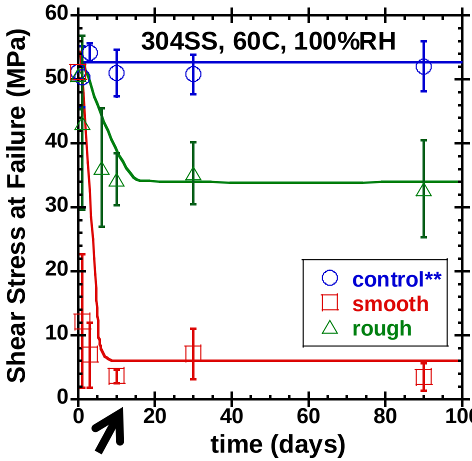






Advantages:

1. **The absence of severe strain gradients enables computational assessment of the joint using the NLVE polymer model**
2. **Simplicity of the joint allows for a mechanistic interpretation of failure**
3. **Short diffusion path allows for effects of small molecule (e.g., water vapor) absorption to be a assessed in a short time period**

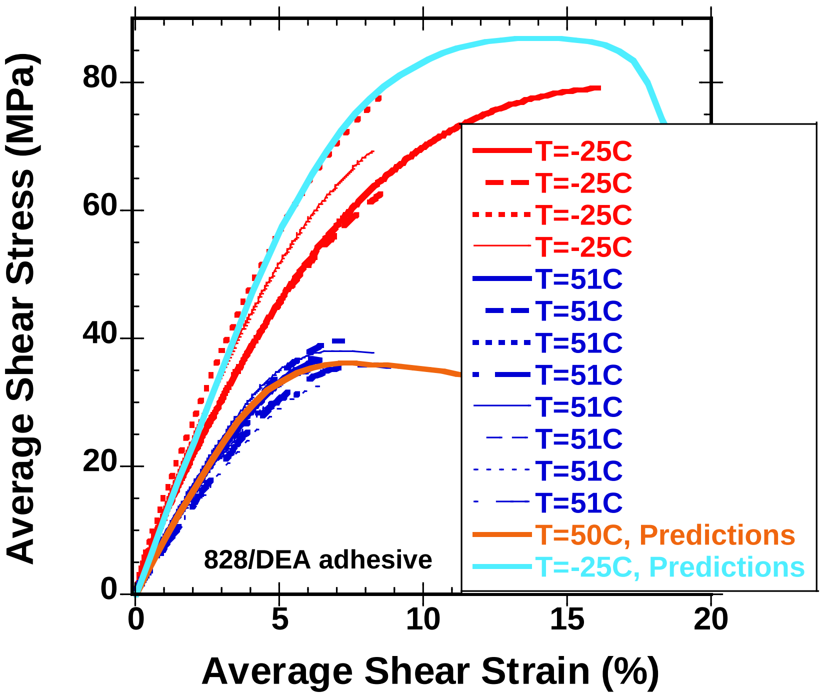


joint strength equilibrates in < 2 weeks

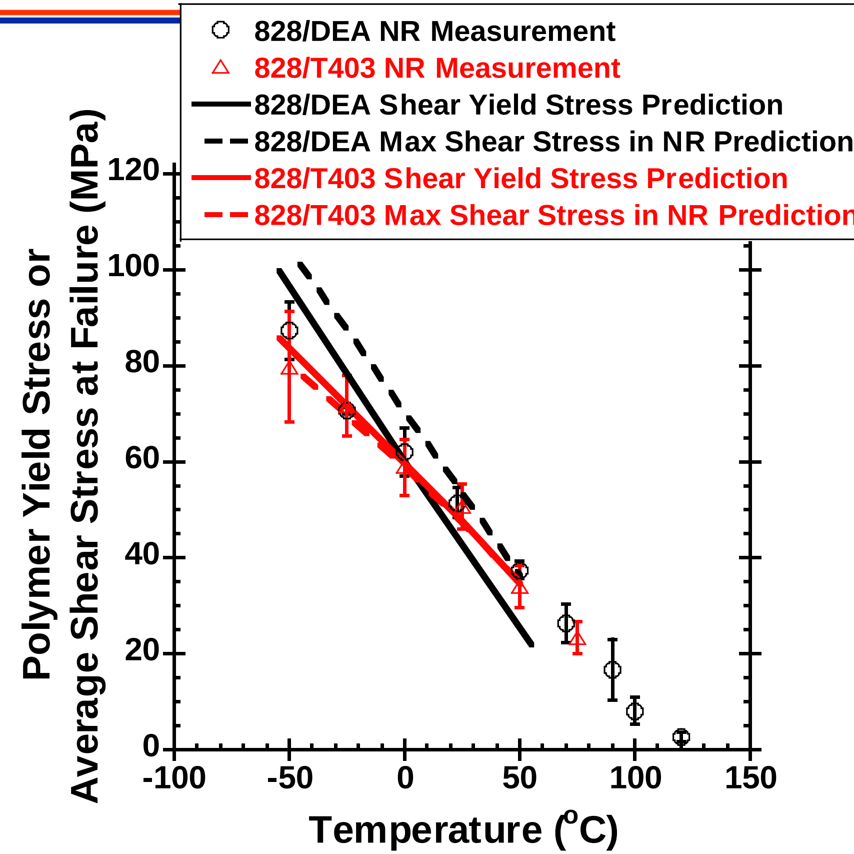
Eng Strain ~ 2(tensorial strain)
**control remained in a 60C, dry environment

Develop a Predictive Approach

Napkin-Ring Macroscopic Response



predictions track data to failure

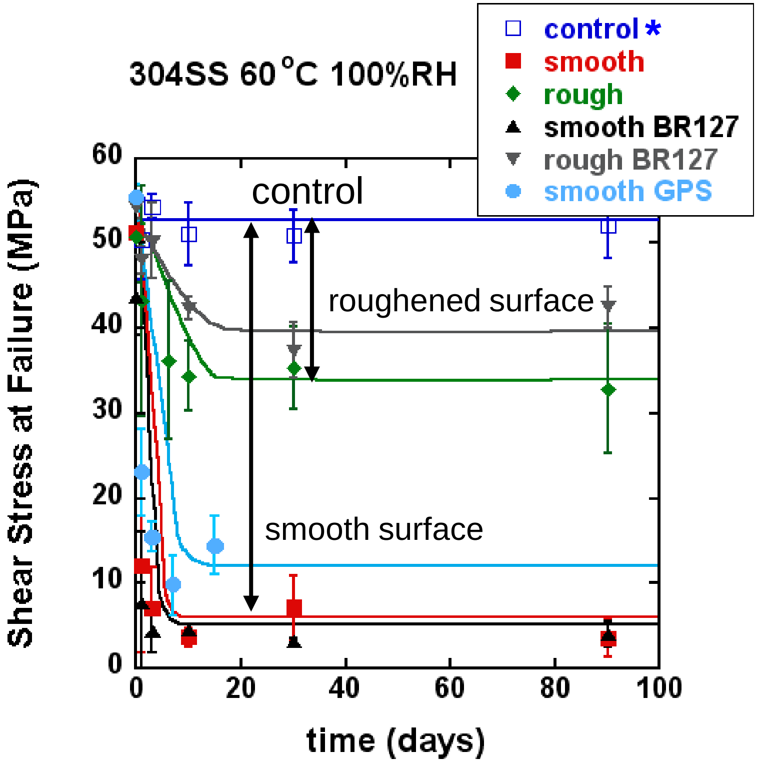


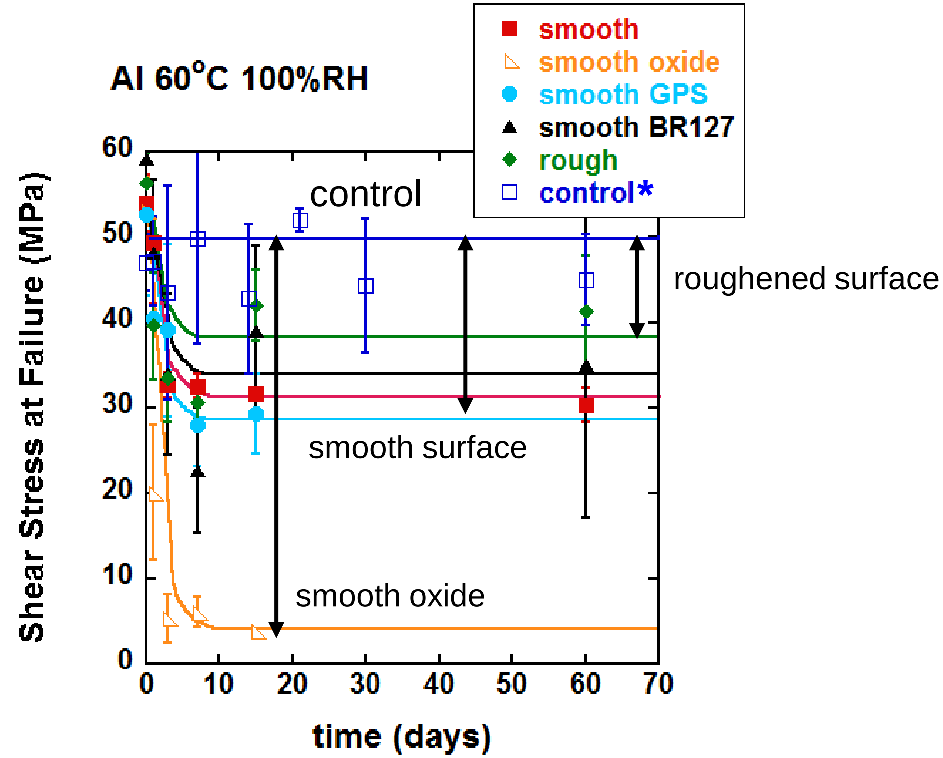
experimental failure stress tracks both the predicted "yield stress" of the polymer and the maximum predicted stress in the NR joint

NLVE model "yield" is a valuable metric for failure predictions

*polymer cross-sections all modeled with 90 degree corners

Joint Strength in Humid Environments: Polymer-Metal Joints





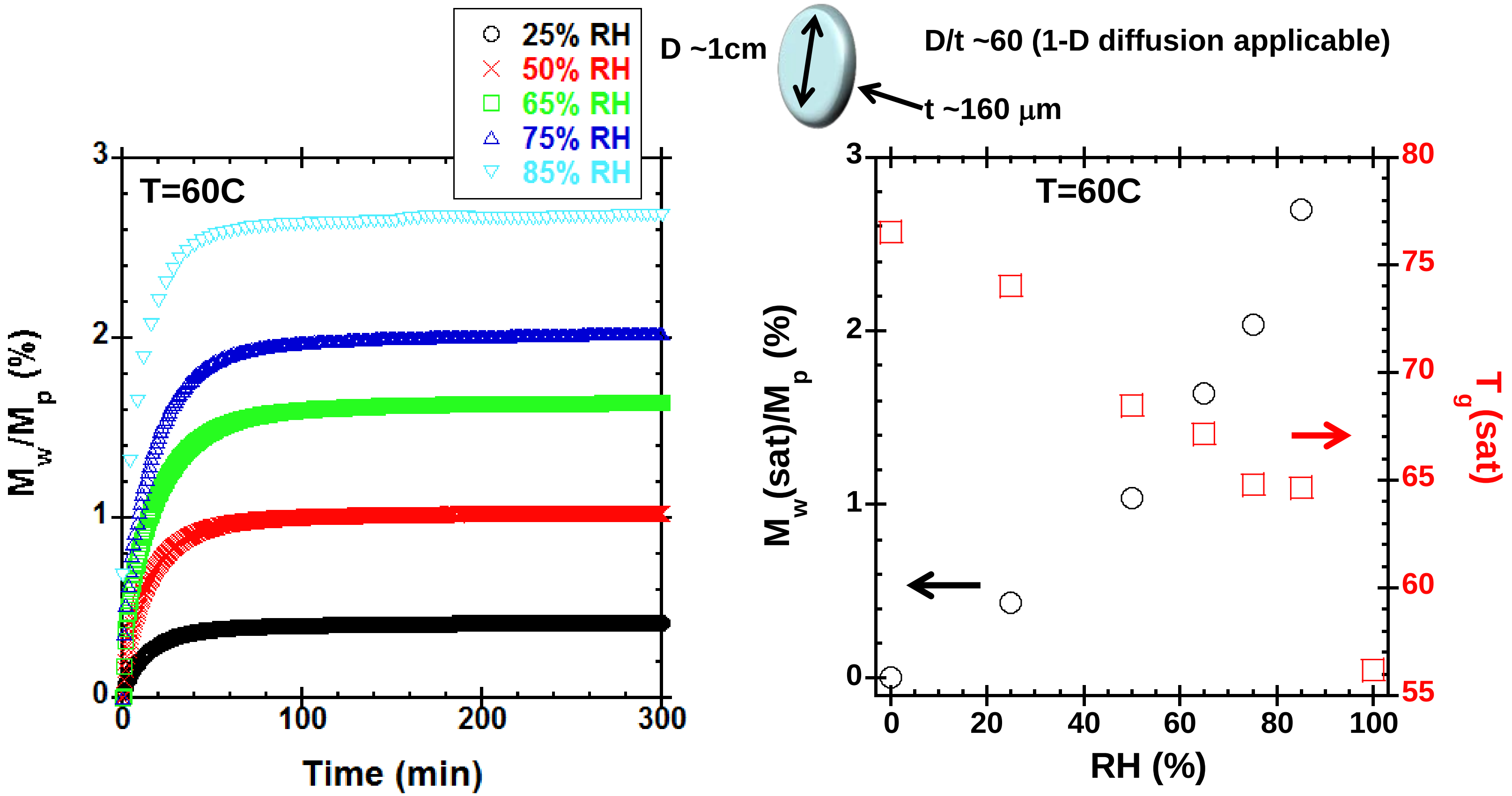
Findings**:

- Virgin joint strengths are independent of surface preparation
- Joint strengths equilibrate to a depressed level in 100% RH conditions at 60C
  - Effect is associated with the presence of moisture, not elevated temperature
- Both the interface materials and surface preparation are factors in determining the magnitude of the equilibrated strength

*control remained in a 60C, dry environment

**More detailed analyses available in J.M. Kropka et al. "Mechanisms of Degradation in Adhesive Joint Strength: Glassy Thermoset Polymer Bond in a Humid Environment", under review.

Understanding Joint Strength Depression: Bulk Water Absorption into Adhesive



Findings:**

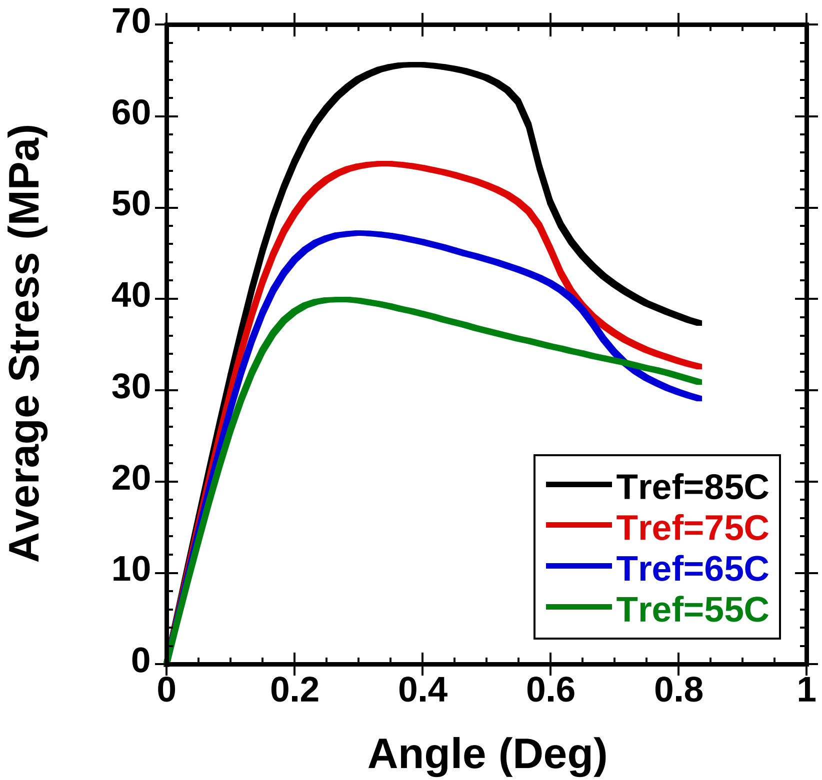
- Water reaches an equilibrium concentration in the polymer and subsequent absorption-desorption cycles were equivalent (not shown), suggesting no irreversible processes occur during water sorption
- Absorbed water acts as a plasticizer and depresses the glass transition temperature (T_g) of the bulk polymer. The magnitude of the T_g depression increases with the amount of water absorbed in the polymer.

Note: bulk sorption rates suggest that napkin-ring joints would be water saturated in ~3 days, which is about the time that it took joint strength to equilibrate

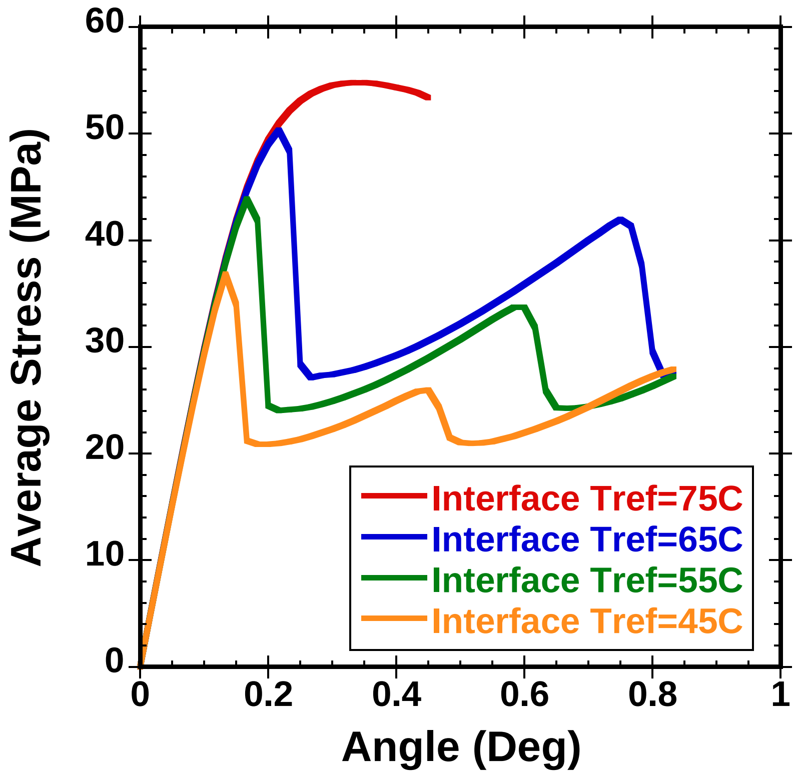
**More detailed analyses available in J.M. Kropka et al. "Mechanisms of Degradation in Adhesive Joint Strength: Glassy Thermoset Polymer Bond in a Humid Environment", under review.

Adhesive Tg Depression and Joint Strength: Predictions[1]

Bulk Tg Change[2]



Interfacial Tg Change[3]



Predictions suggest that either a bulk or interfacial T_g change of the magnitude associated with water absorption into 828/DEA will significantly impact the maximum stress sustained by a napkin-ring joint

[1]Predictions were made using the SPEC model (D.B. Adolf, et al., *Polymer*, 2009, 50, pp 4257-4269) to represent the 828/DEA adhesive in finite element calculations. An idealized napkin-ring joint. as depicted in slides 6-7, was taken as the geometry

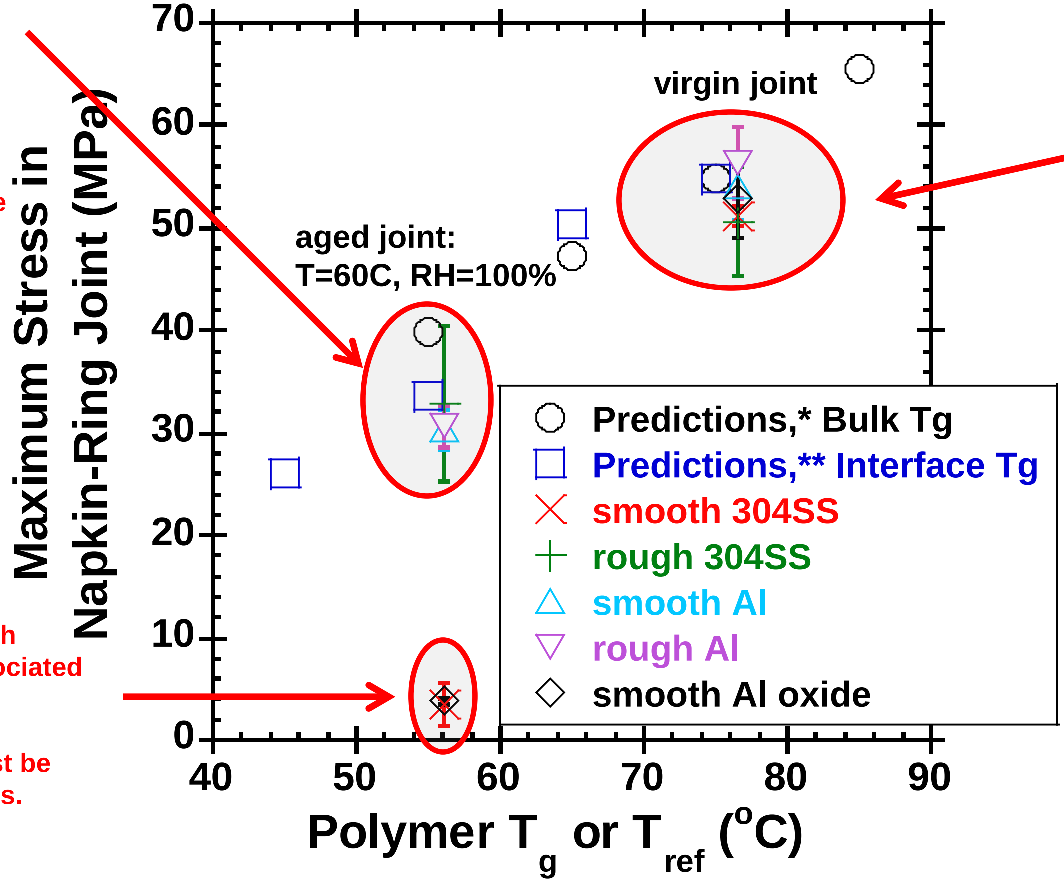
[2]Predictions assume stress free temperature of 75C. The joints are cooled at 5 C/min to T=25C and then rotated at 5 deg/min. Tref in the bulk epoxy is varied.

[3]Predictions assume stress free temperature of 75C. The joints are cooled at 5 C/min to T=25C and then rotated at 5 deg/min. Tref in the bulk epoxy is maintained at T=75C. The Tref of a 10 μm thick interface layer (at both adherend interfaces) is varied.

Adhesive Tg Depression and Joint Strength

Predictions suggest change in joint strength can be accounted for by the reduction in adhesive Tg (associated with water absorption). In these cases, joint strength can be rejuvenated by drying.

In some cases, joint strength decreases beyond that associated with adhesive Tg reduction. Other factors (e.g., specific interfacial phenomena) must be accounted for in these cases. Joint strength cannot be rejuvenated by drying.



Predictions agree with data for virgin joint strength

- Adhesive Tg reduction with water sorption often accounts for the change in joint strength and physically-based nonlinear viscoelastic polymer models can predict joint strength under these conditions
- In some cases, additional factors must be accounted for

*Predictions assume stress free temperature of 75C. The joints are cooled at 5 C/min to T=25C and then rotated at 5 deg/min. Tref in the bulk epoxy is varied.
**Predictions assume stress free temperature of 75C. The joints are cooled at 5 C/min to T=25C and then rotated at 5 deg/min. Tref in the bulk epoxy is maintained at T=75C. The Tref of a 10 μm thick interface layer (at both adherend interfaces) is varied.

Summary on Humid Environment Testing

- **"Yield" in Simplified Potential Energy Clock (SPEC) polymer NLVE model is a good metric for shear failure in Napkin-Ring joint**
- **Napkin-Ring geometry is an excellent tool to characterize wet adhesive failure: fast (days to equil.), directly yields stress-at-failure, simplicity allows mechanistic interpretation**
- **Bonding materials and surface preparation significantly affect the role of moisture on adhesive strength**
- **Predicting degradation in joint strength:**
  - **Bulk polymer Tg depression with water sorption can account for joint strength depression in humid environments when the equilibrated joint strength settles at ~30-40 MPa. In these cases, SPEC can predict changes in joint strength and strength can be rejuvenated by drying the joint.**
  - **In other cases, joint strength is depressed beyond that which can be accounted for by adhesive Tg depression. Additional factors, which may be associated with the details of specific surface phenomena, must be accounted for in these cases. For the "smooth" stainless steel joints, such specific surface phenomena may not occur until a critical humidity, or critical water concentration within the adhesive, is exceeded. Understanding what the surface specific phenomena are and how they affect interfacial failure are of interest and continue to be investigated. At this point, these failures mechanisms cannot be accounted for in a predictive technique.**

Path Forward on Humid Environment Testing

- **Continue to investigate differences between "rough" and "smooth" stainless steel performance in humid environments**
  - **What are differences associated with?**
    - **Surface topology?**
    - **Surface chemistry?**
    - **Other?**
- **Design and Testing of "Validation" Test Joint Geometry**

Role of Residual Stress on Strength of Adhesive Joints

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Altering residual stress state in napkin-ring joint

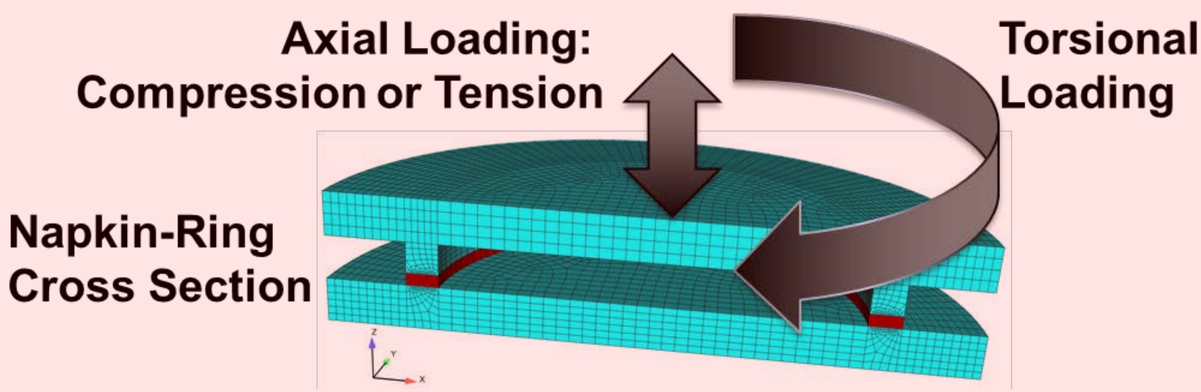
1. Alter width-to-thickness ratio of bond-line

No significant changes in joint strength resolved
-residual stress predictions were well below the initiation of "yield"

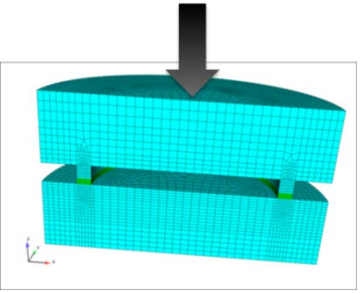
2. Axially fix adherends during processing

No significant changes in experimental joint strength resolved
-even predictions suggested < 20% change in maximum shear stress sustainable in NR joint over experimentally practical limits

3. Combined axial and torsion loading of the joint

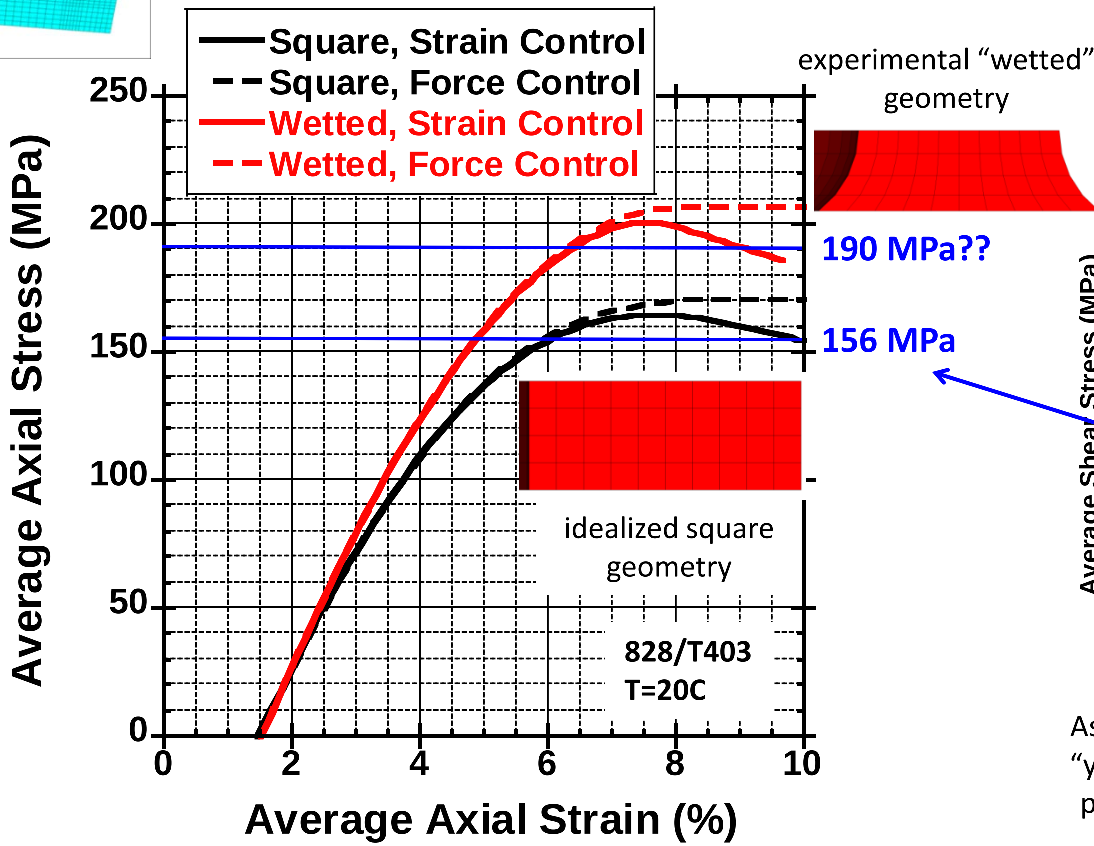


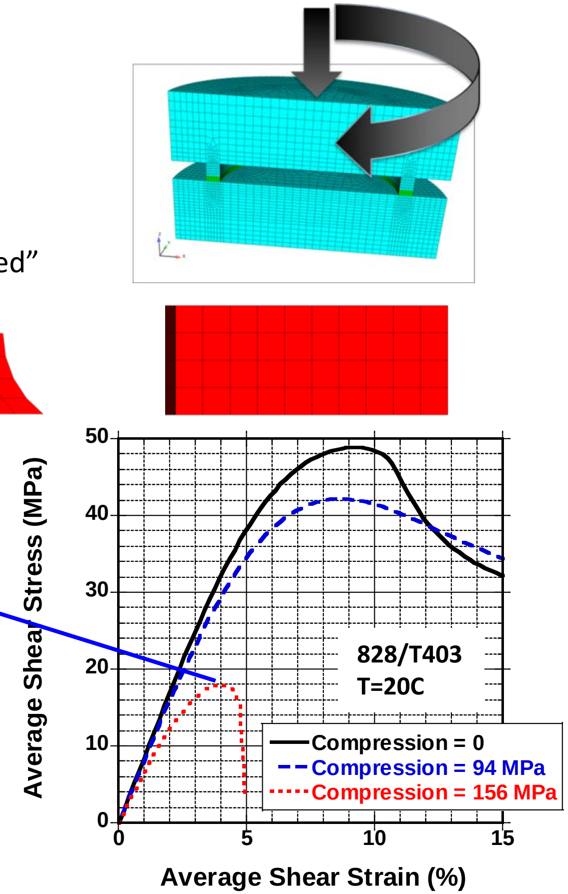
What do models predict for torsion response under compression?



Model history:

- Stress free at T=90C
- Cool to T=20C at 0.5C/min
- Apply compressive load to entire outer surface of adherend

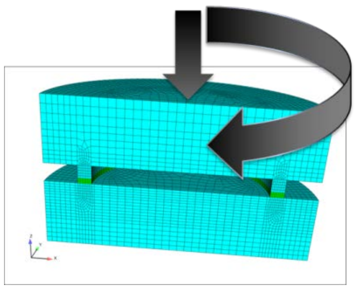


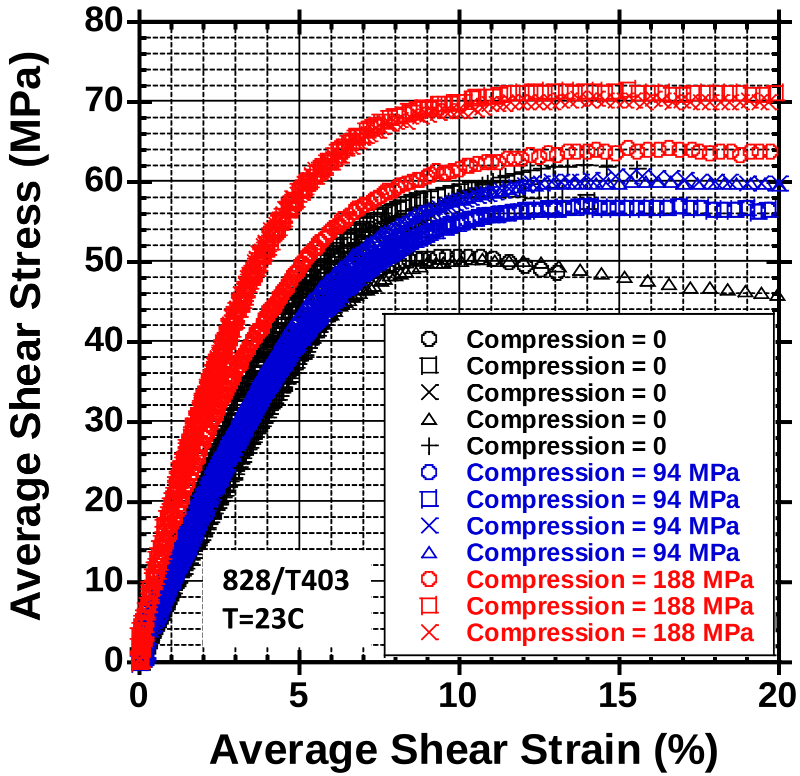


As compressive load approaches predicted "yield", further "yielding" under shear load predicted to occur at smaller shear stress

Will a compressive load of ~190 MPa alter the experimental shear stress-strain response of the joint?

Combined compression (stress control) and torsion load experiments*





Observations:

- No signature of early "yielding" or joint failure resolved under combined compression and torsion load
  - Compressive load will prevent torsion load from dropping to zero even upon joint failure due to friction
- If anything, compressive loading increases the maximum shear load the joint can sustain

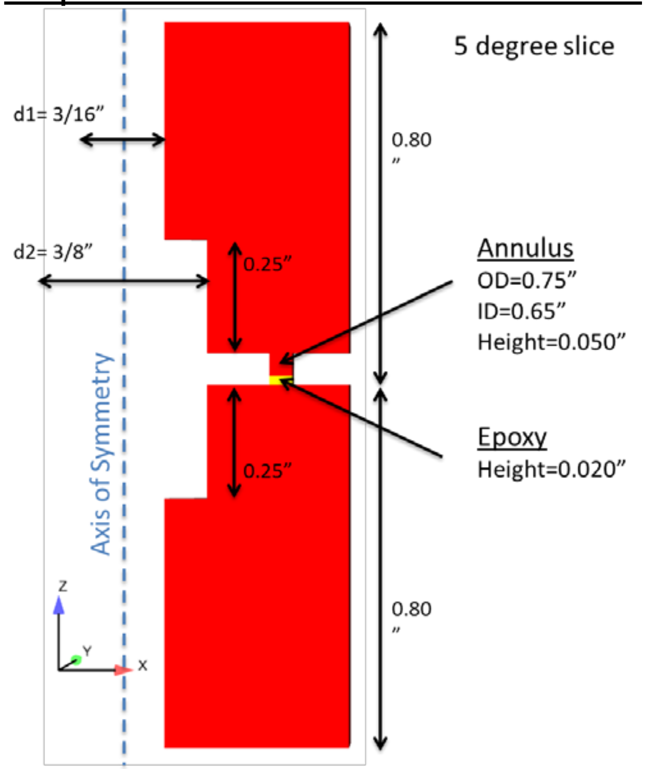
Simpler question: Does model get the joint compressive response right?

*stress controlled compressive load ramped over 10 seconds, followed by strain controlled rotational displacement

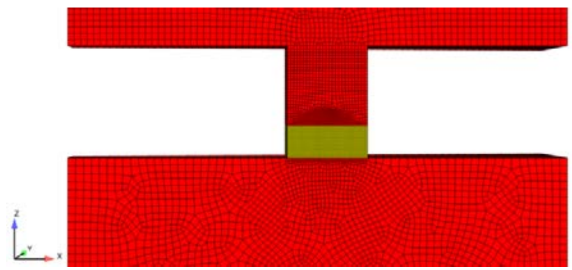
Simpler question: Does model get the joint compressive response right?

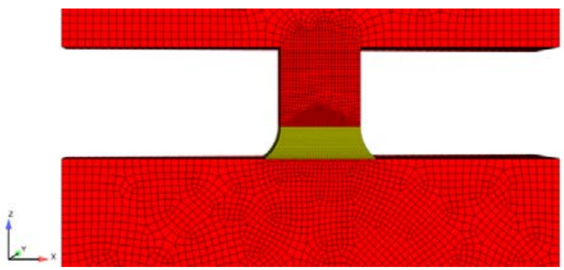
Step One: Model Napkin-Ring with More Fidelity

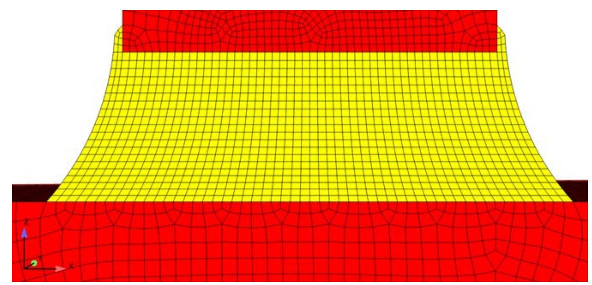
Capture all Axial Features of Joint



Increase Finite Element Mesh Resolution and
Examine Potential Adhesive Bondline Geometries



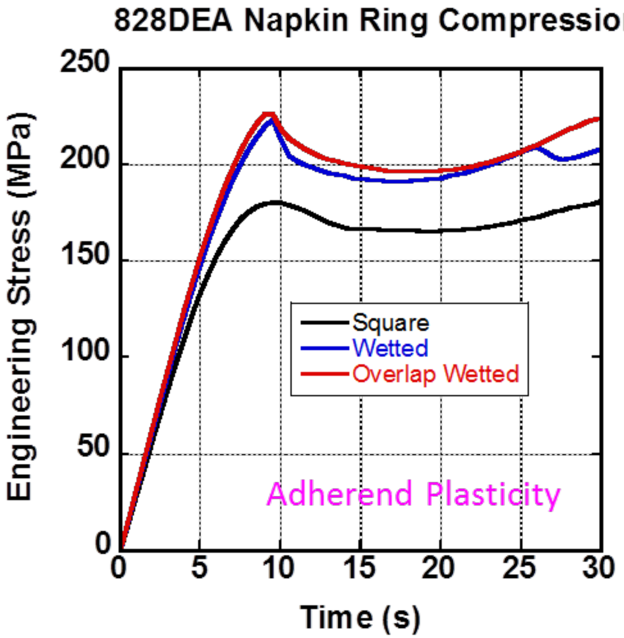




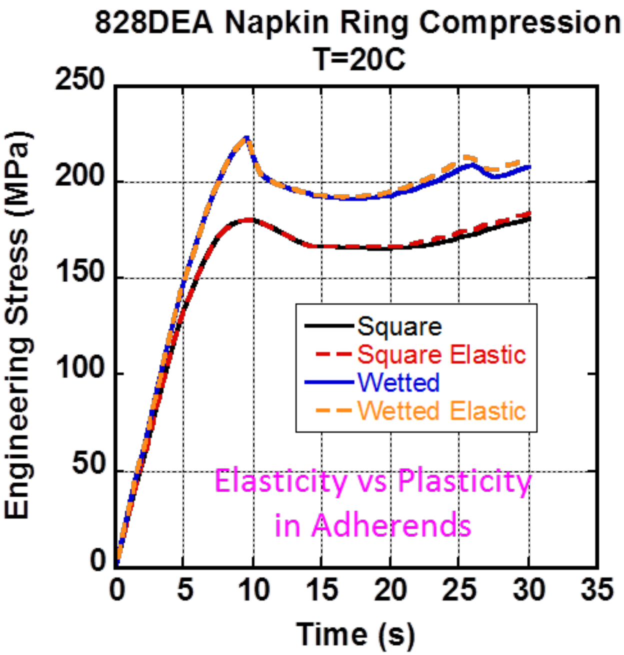
Simpler question: Does model get the joint compressive response right?

Step Two: Examine Model Sensitivities

Adhesive Bondline Geometric Sensitivity



Adherend Yield Behavior Sensitivity



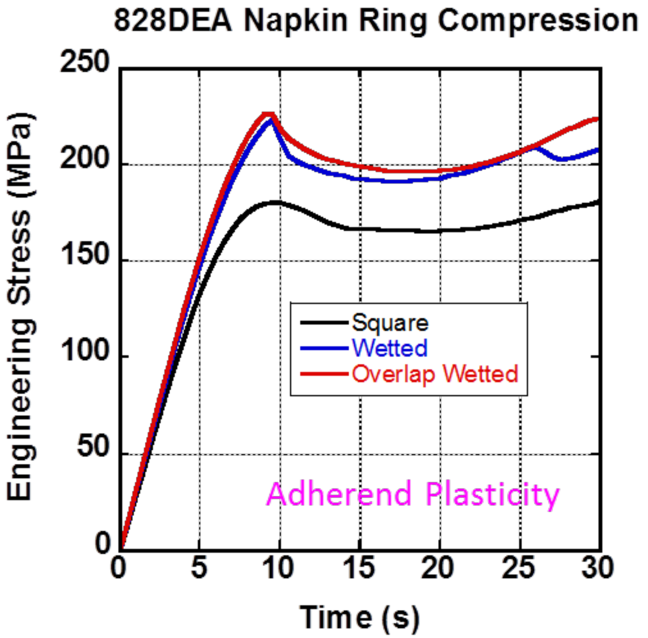
Findings:

- **Only square geometry is unique to adhesive "yield", hence focus on wetted (or overlapped wetted) going forward**
- **Adherend plasticity is not a factor until well beyond adhesive yield, doesn't matter what use**

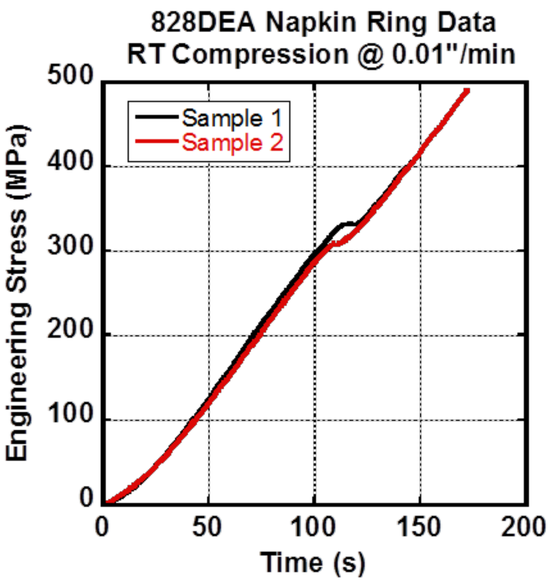
Simpler question: Does model get the joint compressive response right?

Step Three: Compare to Experiments

Model Predictions



Experimental Data



Findings:

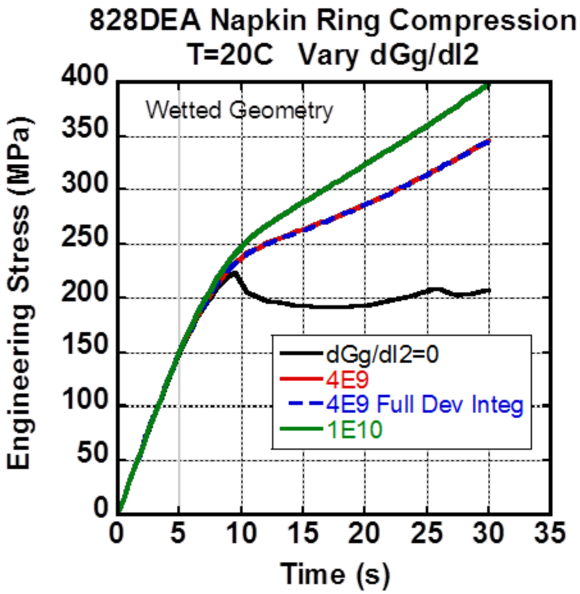
- Data shows signature of adhesive yield much later than predictions—potentially due to experimental compliance
- Post-"yield", data exhibits approximately same slope as pre-"yield" while model shows significant softening

Is this indicative of a heretofore unobserved strain hardening effect in material behavior?

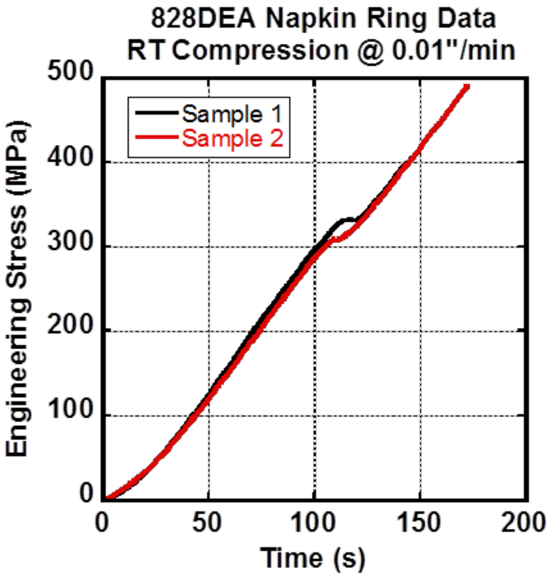
Simpler question: Does model get the joint compressive response right?

Step Four: Add Strain Hardening to More Closely Resemble Data

Sensitivity of Model Predictions to Adhesive Strain Hardening



Experimental Data



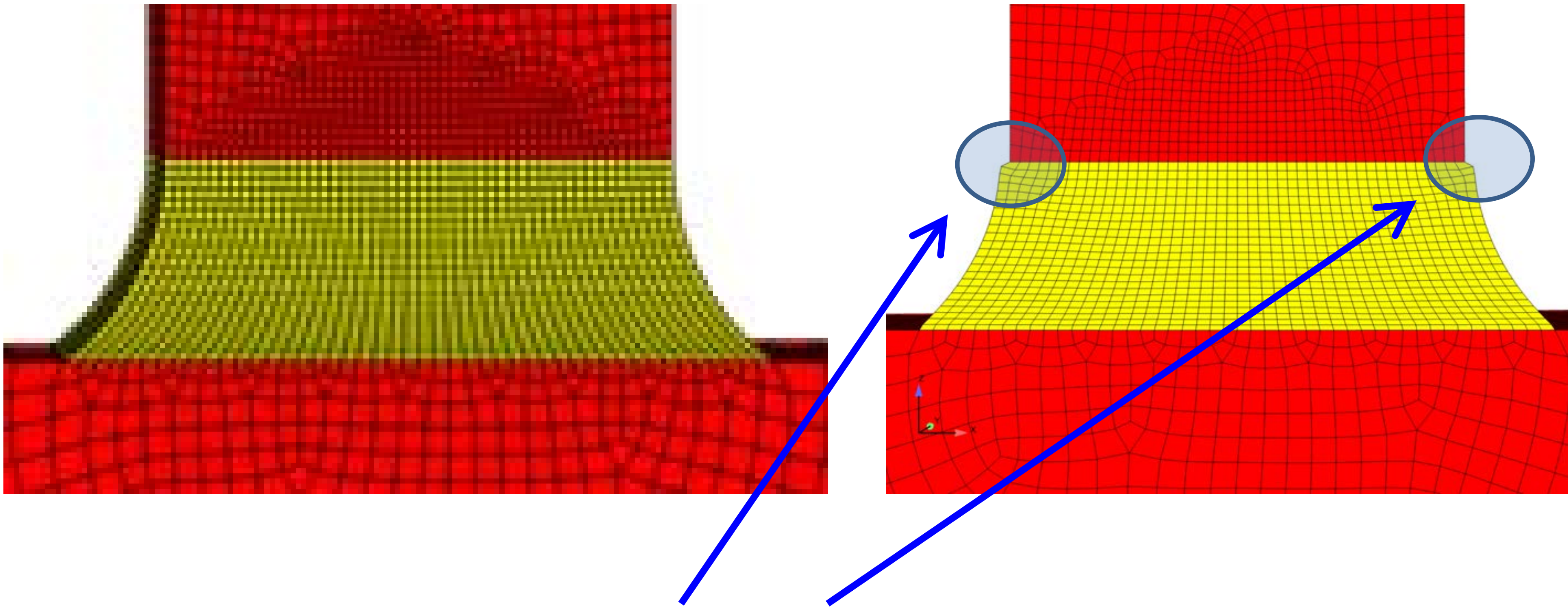
Finding:

Addition of adhesive strain hardening to model can more closely match pre- and post-"yield" slopes, as found in data (granted differences in slope between model and experiment still exist)

Additional Consideration: Likely Debonding Under Compression Alone

No Deformation

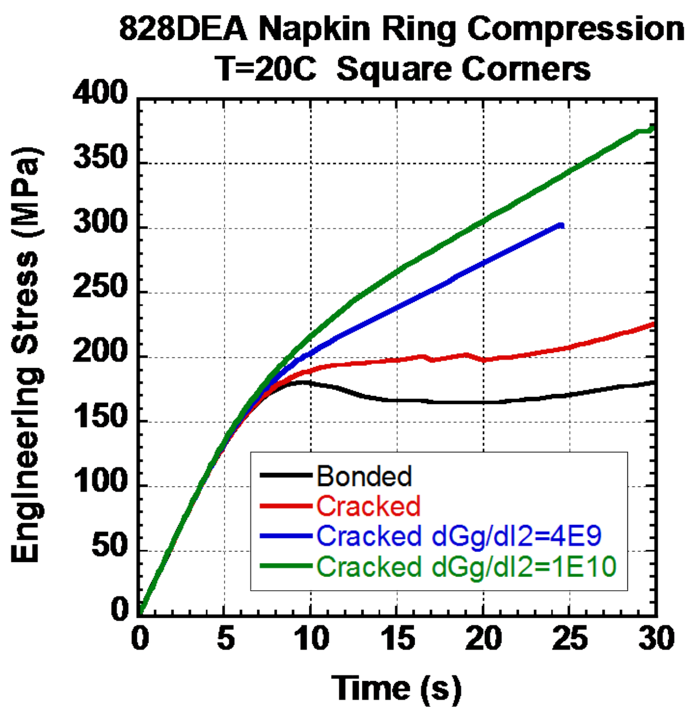
Deformation at t=10s (~"yield")



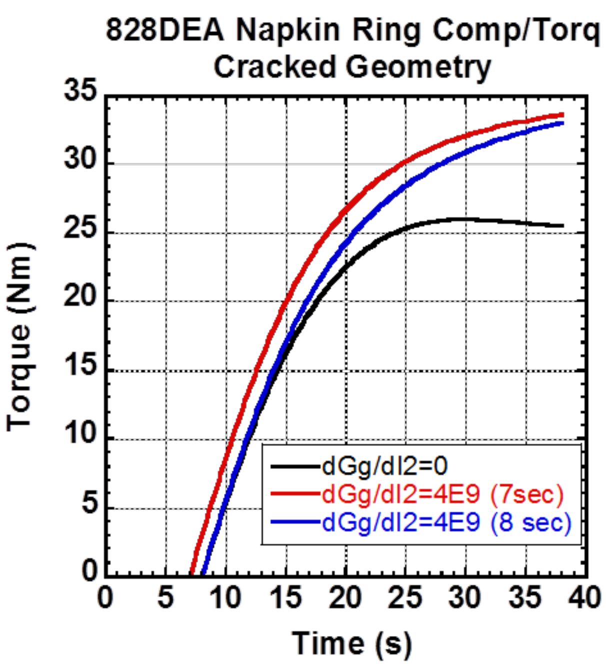
Severe shear strains at corners are likely to induce debonding and result in interface crack

Compression Plus Torsion Response with 0.003” Debond (Crack) at Corners and Adhesive Strain Hardening

Predicted Compression



Predicted Compression + Torsion



Findings:

- **Corner debond further stiffens compression response post-”yield”**
- **Strain hardening and corner debond counteract depression of maximum predicted torque with compressive load and may account for lack of experimental observation of napkin-ring joint failure at reduced shear stress under large compressive loads**

Details Matter!

Summary for Role of Residual Stress on Strength of Adhesive Joints

- Must capture the details of the test in model in order to predict experimentally observed behavior
- To predict experiments that explore behavior at or beyond "yield" of the adhesive, strain hardening of the adhesive may be important. Model parameterization for strain hardening should be completed using simple tests designed to resolve this effect rather than the complicated adhesive joint tests here, which are unable to resolve geometric deformation details during the test.

Back-Up Slides

Adhesion Task - Key Personnel

Name	Org	Role
Jamie Kropka, Doug Adolf (jmkropk@sandia.gov; 505-284-0866)	*SNL*	*Task Leader for Aging of Adhesive Joints and mechanical testing*
Mike Bucher (michael.bucher@navy.mil; 301-643-3772)	*NSWC-IH*	*Working Group Leader for Aging of Adhesive Joints*
Scott Spangler (sspangl@sandia.gov; 505-845-3069)	*SNL*	*Polymer properties and mechanical testing*
Bob Chambers (rschamb@sandia.gov; 505-844-0771)	*SNL*	*Finite element analyses*
Dave Dunaj (david.dunaj@navy.mil; 951-204-4933)	*China Lake*	*Navy working group representative*
Alexander Steel (alexander.steel@us.army.mil; 256-876-3867)	*RDECOM*	*Army working group representative*
Jim Mazza (james.mazza@wpafb.af.mil; 937-255-7778)	*AFRL*	*Air Force working group representative*
Aisha Haynes (aisha.s.haynes@us.army.mil, 973-724-9674)	*ARDEC*	*Army working group representative*

Adhesion Task Four-Question Chart

What are you trying to do in this task?

- Measure and predict the critical stresses for adhesive de-bonding
- Measure and predict the change in de-bonding stress when components age in dry and humid environments
- Relate the de-bonding stress to processing history

What makes you think you can do it?

- Leverages previous SNL-funded research on measuring and predicting adhesive strength
- Adhesion working group involves DOE and DoD members to direct goals and share knowledge/experience

What difference will it make?

- Component designs can be more robust if de-bonding stress margins are known
- Knowledge of aging mechanisms improve material selection for given environments
- Processes can be defined to improve adhesive strength

What / When / To Whom Will You Deliver?

- Deliverables are metrics and procedures to measure and predict de-bonding
- Delivery will be staged to provide capability on successively more difficult systems
- Adhesion working group will identify a DoD contact to share capabilities

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Adhesion Task GOTChA

Goal: --

Predict de-bonding of adhesively bonded components

Objective: --

Develop a straightforward experimental test, unravel the underlying mechanisms, develop a predictive approach, and implement it in a computational procedure

Challenges: ---

experimental | mechanism | theory | computational | validation

Approach: --

napkin ring test | NLVE polymer model | finite element stress prediction

Tasks: ---

develop experimental path | assess sensitivities | develop computational approach