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Experiments and modeling of post-yield deformation in Epon 828/DEA and 828/T403 epoxy

Scott Grutzik, E Dave Reedy

Component Science and Mechanics
Sandia National Laboratories
Albuquerque, NM

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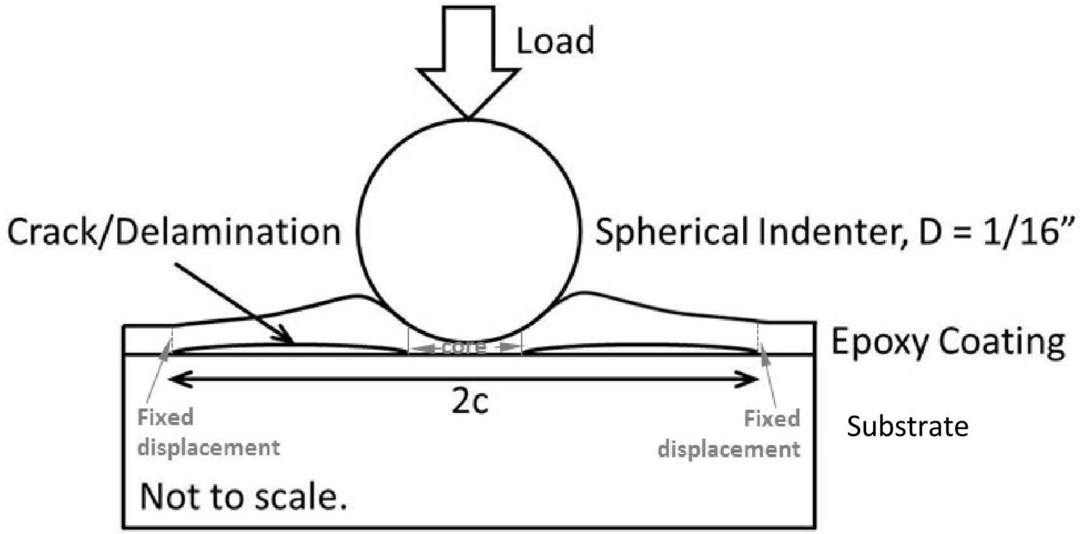
Motivation for investigation of viscoelastic softening and hardening

- A common test for interfacial strength is spherical indentation of a film (blister test)
- Quantitative results can be hard to obtain, results are often comparative or "pass/fail"
- Detailed understanding of the mixed mode delamination and mechanics of the epoxy under the indenter is required
- Sandia currently uses a potential energy clock based nonlinear viscoelastic model for epoxy materials (SPEC)
- SPEC predicts post-yield softening but ellipticity is lost
- SPEC does not predict hardening at large strains
- This work is an effort to experimentally characterize and model the post-yield behavior (both softening and hardening)
- Will investigate compression cylinder specimen and butt tensile joint specimen



Tested two epoxy materials

EPON® Resin 828 cured with DEA (diethanolamine)

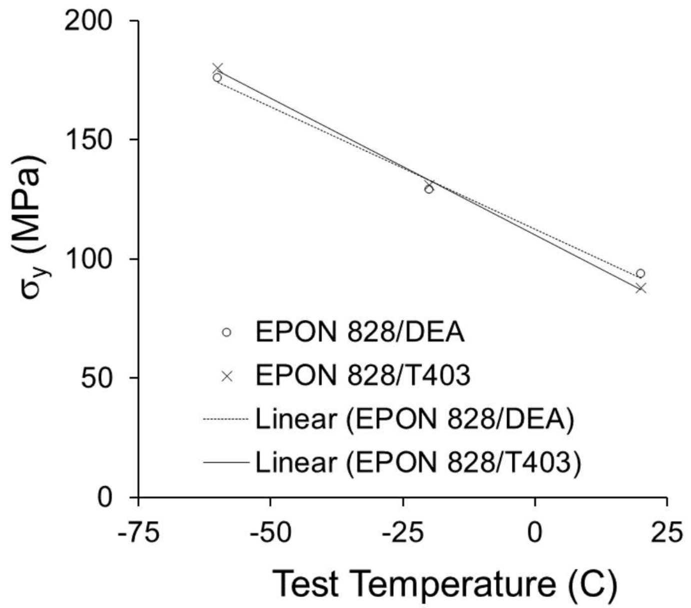
- 100:12 pbw mix ratio.
- Cure cycle: 24hr at 45C, ramp to 71C in $6\,{}^{1}/_{3}$ hr, hold at 71C for 5 hr, cool down to RT.

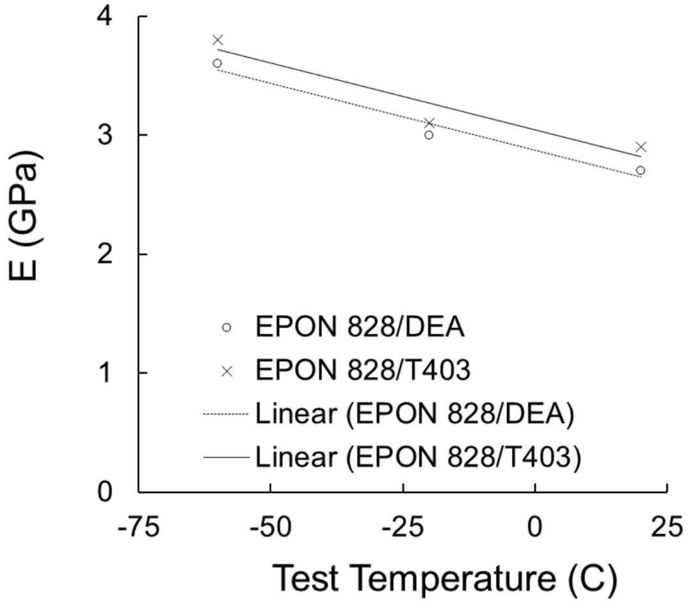
EPON® Resin 828 cured with Jeffamine® T-403 (polyetheramine)

- 100:43 pbw mix ratio.
- Cure cycle: 24hr at 23C, followed by 3hr at 50C, followed by 15hr at 80C, cool down to RT.

Compression plug stress-strain data
(nominal strain rate = 0.001/s)

	EPON 828/DEA Tg=70 °C		EPON 828/T403 Tg=85 °C	
T (C)	E (GPa)	σ_y (MPa)	E (GPa)	σ_y (MPa)
RT	2.7	94	2.9	88
-20	3.0	129	3.1	131
-60	3.6	176	3.8	180



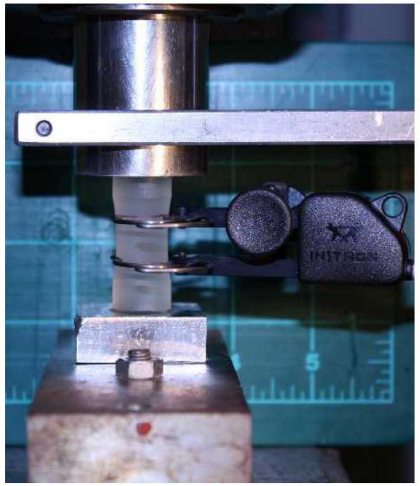


Performed compression tests of waisted epoxy cylinders

Goal of the following is to provide illustrative results that demonstrate the general features of the large strain response of this class of epoxy.

- Used waisted cylindrical specimen that are nominally 38 mm long, with a 90% reduced diameter at mid-plane to initiate yielding at a known position (minimum radius ~ 7.2 mm).
  - Specimen bonded to platens of a sub press to keep specimen from shifting (since sample is waisted, there is limited yielding at platens during test).
  - Changes in midplane cross-sectional area measured using a diametral extensometer.
  - Axial displacement about waist's center measured with a 12.7 mm extensometer.
  - Either axial or diametral displacement measured during a given test (not both at same time).
  - Must use waisted specimens with the same initial geometry to correlate axial and diametral data since results are geometry dependent.

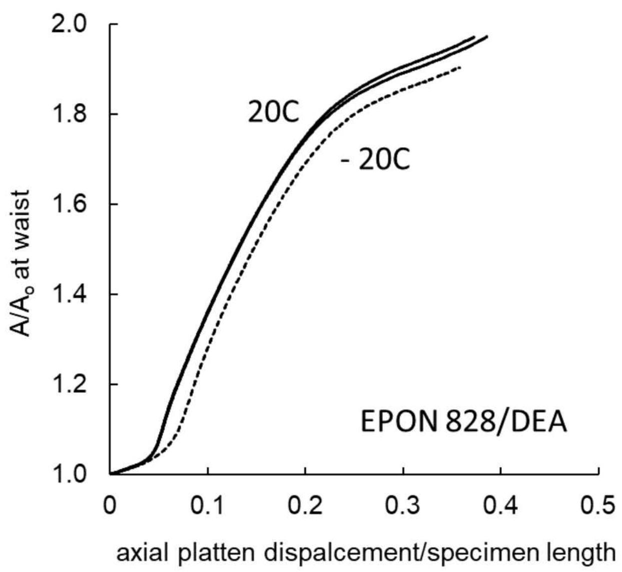
specimen

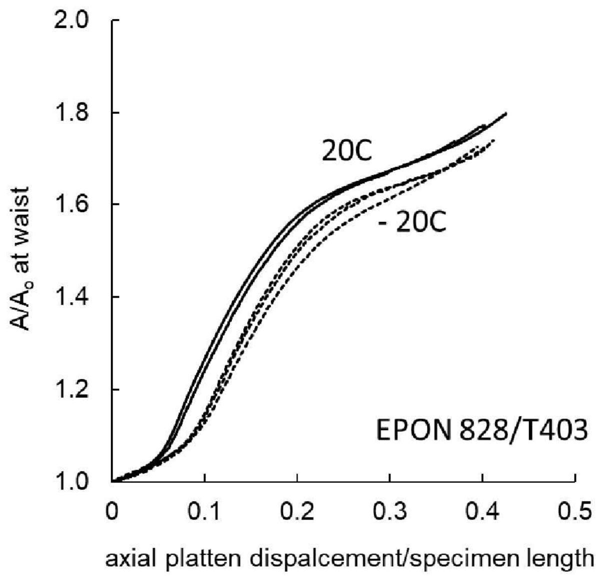


measure axial displacement

measure change in diameter

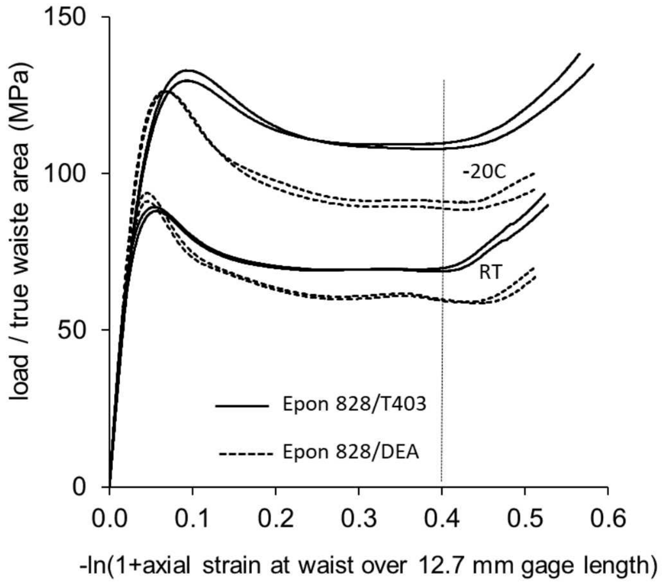
Large strain diametral compression data

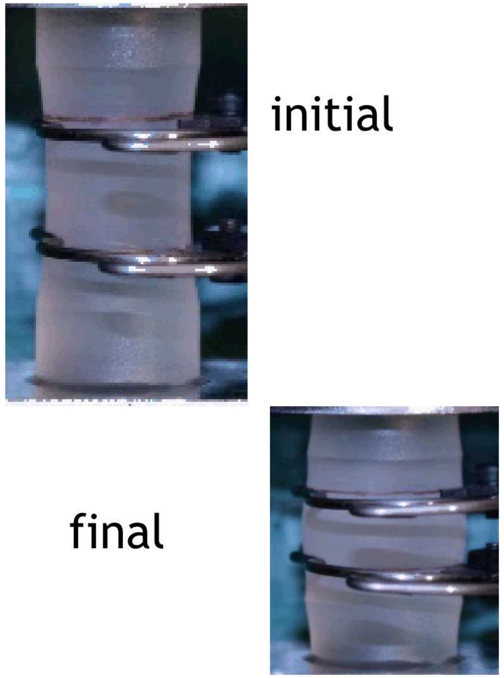




- Measured the diameter at the center of the waist to determine the ratio of current/initial waist cross-sectional area.
  - initial upturn in curve occurs sooner at higher temperature--- lower σ_y at higher T.
  - noticeable decrease in slope at higher strain --- waist area increasing more slowly.
- Determined average fit for a given temperature (i.e., polynomial fit of averaged data).
- Used fit of A/A_o to determine true stress in those tests where waist diameter was not measured (i.e., in tests where there was an axial extensometer across waist).

Large strain axial compression data





- Measured data for axial compression shows an initial upper yield point, strain-softening, a lower stress plateau, and finally hardening at large strain (tests terminated when strain ~0.5).
  - both epoxies show similar response, but differ in detail.
  - strong temperature dependence.
  - plastic deformation occurs at a roughly constant flow stress (σ_{py}) after initial post yield softening.
  - a substantial amount of energy dissipation can occur after initial yield and prior to hardening at high strain.

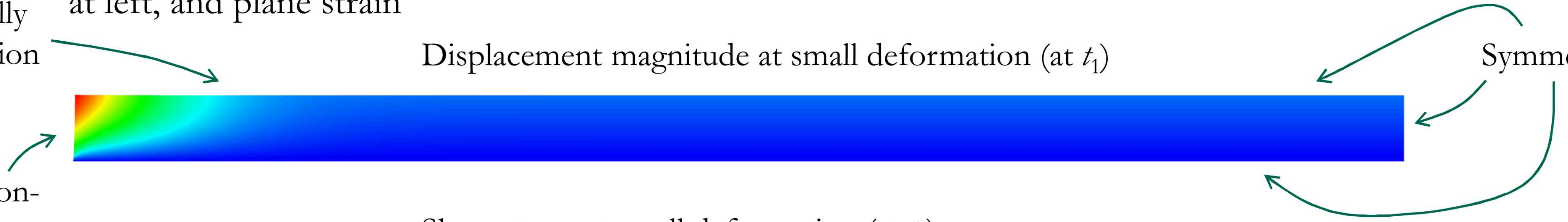
Butt joint tensile softening

- We model a thin adhesive layer bonding two rigid adherends which are pulled in tension
- In this specimen a residual stress field exists at room temperature prior to loading
- The stress concentration at the bond/adherend interface corner is of interest
- Boundary conditions are rigidly clamped along the bottom, symmetry at top and right, traction free at left, and plane strain

Pulled vertically in tension

Displacement magnitude at small deformation (at t_1)

Symmetry



Traction-free

Shear stress at small deformation (at t_1)

Time shift factor at small deformation (at t_1)

Butt joint tensile softening

- As tension is applied, the first row of elements adjacent to the interface softens in shear
- The stress singularity moves off the corner and propagates along the interface

Displacement magnitude after initial softening (at t_2)

Shear stress after initial softening (at t_2)

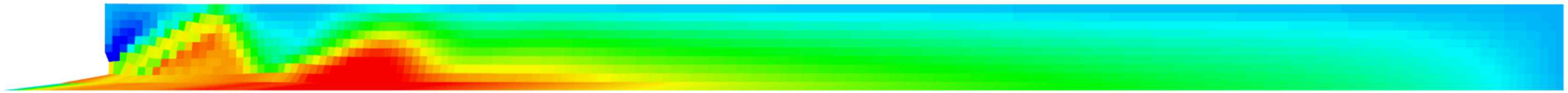
Time shift factor after initial softening (at t_2)

Butt joint tensile softening

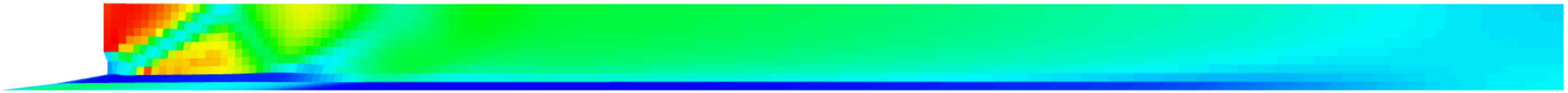
- Stress builds up at the second row of elements, which acts like a new interface corner
- Eventually the second row of elements softens as well

Displacement magnitude after second row softening (at t_3)

Shear stress after second row softening (at t_3)

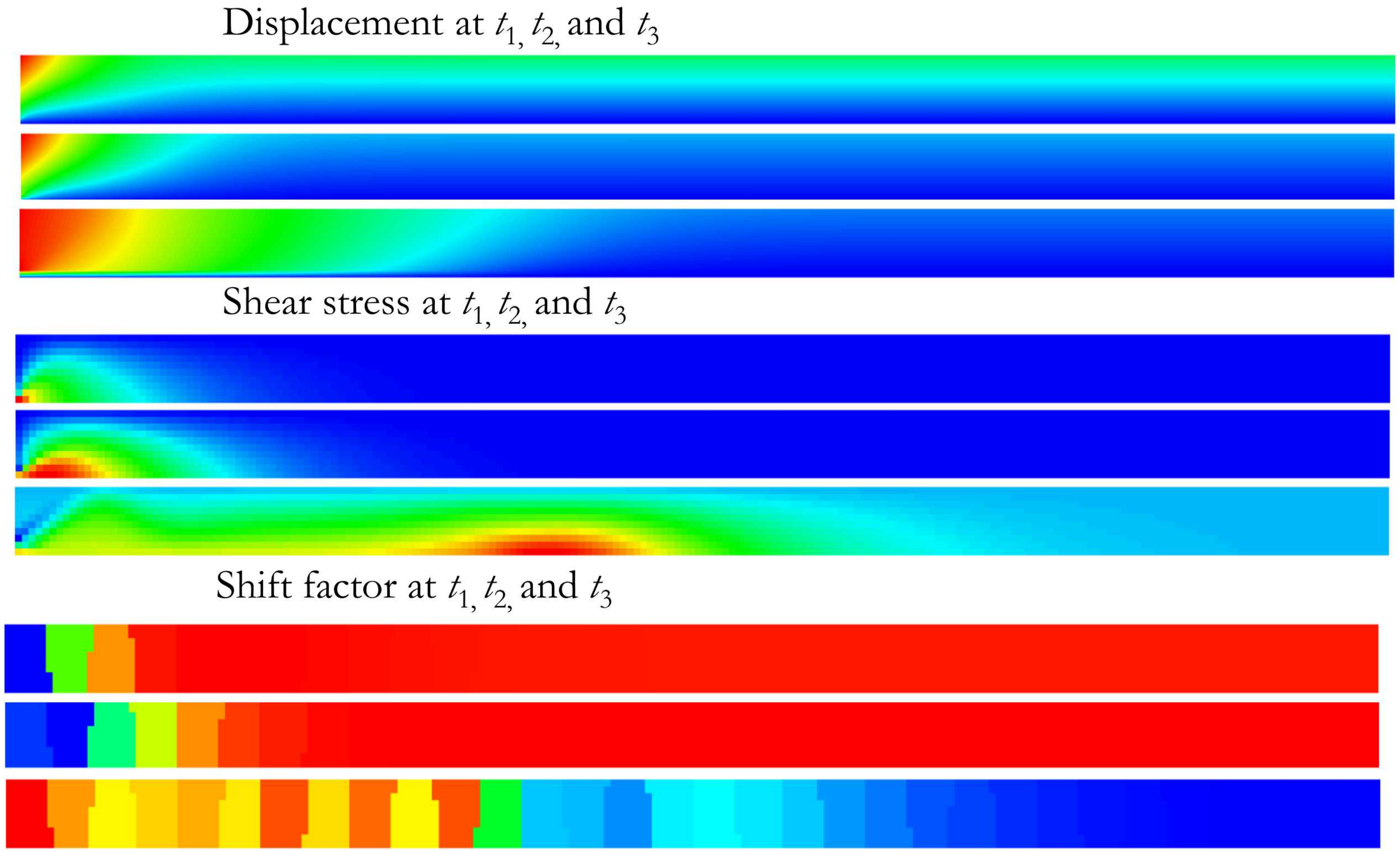


Time shift factor after second row softening (at t_3)



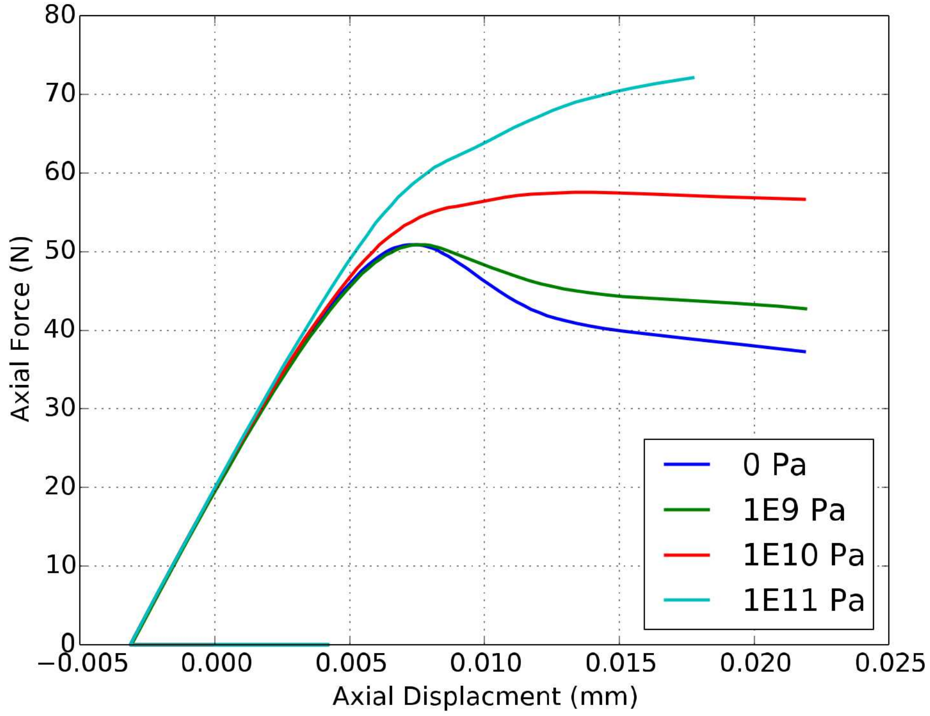
Regularization with non-local averaging

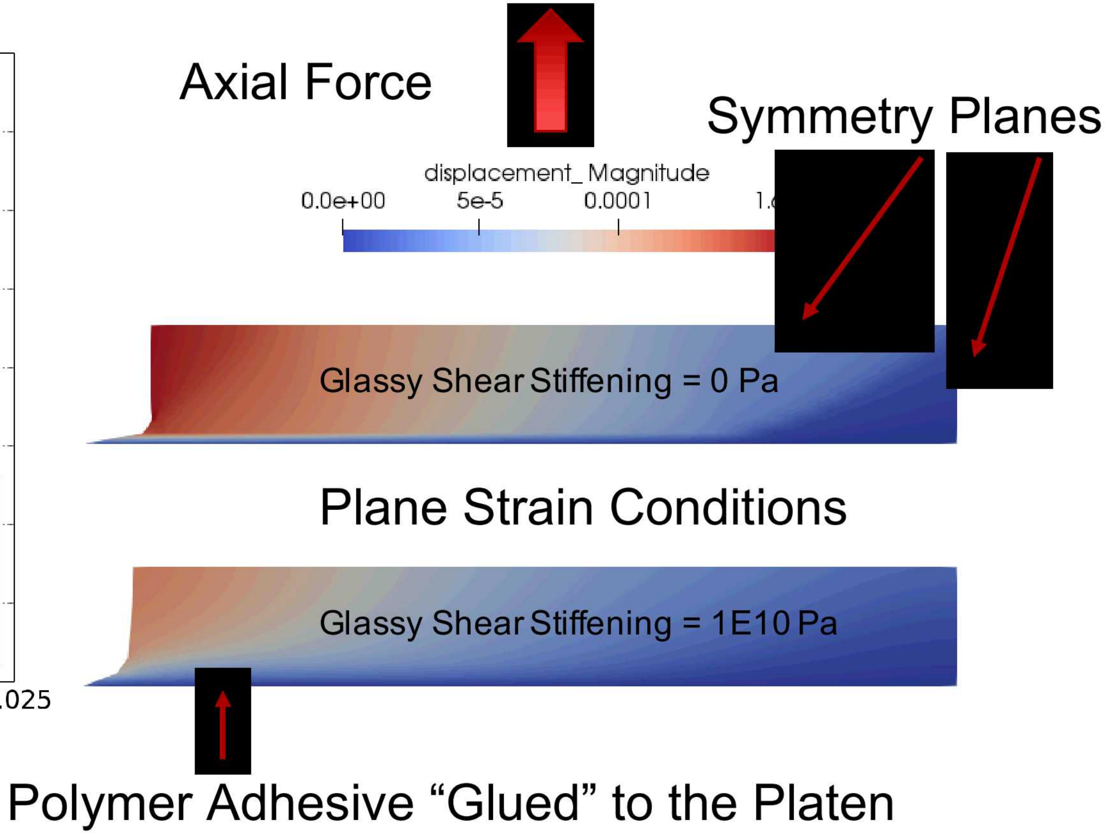
- Non-local averaging of the time shift factor was used in an attempt to regularize the mesh dependent, row-by-row softening behavior
- Onset of softening is delayed but qualitatively the same type of row-by-row response is seen



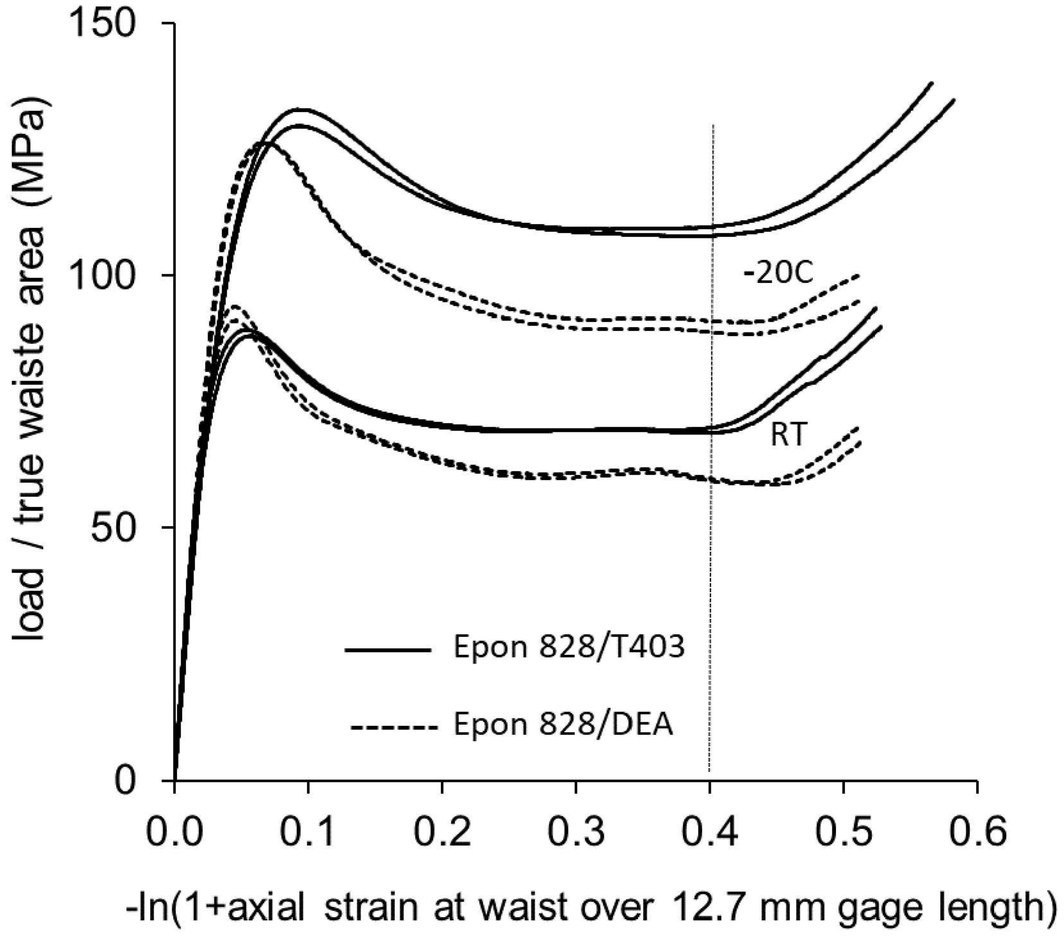
Butt joint with hardening terms

- In a separate effort, strain invariant dependence was added to the SPEC hereditary integral prefactors which can be used to model strain hardening behavior
- The prefactors are of the form $G_G - G_R$ and $K_G - K_R$ where $G_G = G_G^0 + G_G^1 I_1 + G_G^2 I_2 + G_G^3 I_3$ and $I_1 = \mathrm{tr}(\mathbf{H})$, $I_2 = \mathrm{tr}(\mathbf{H}^2)$, $I_3 = \mathrm{tr}(\mathbf{H}^3)$, and $\mathbf{H}$ is the Hencky strain (log strain)



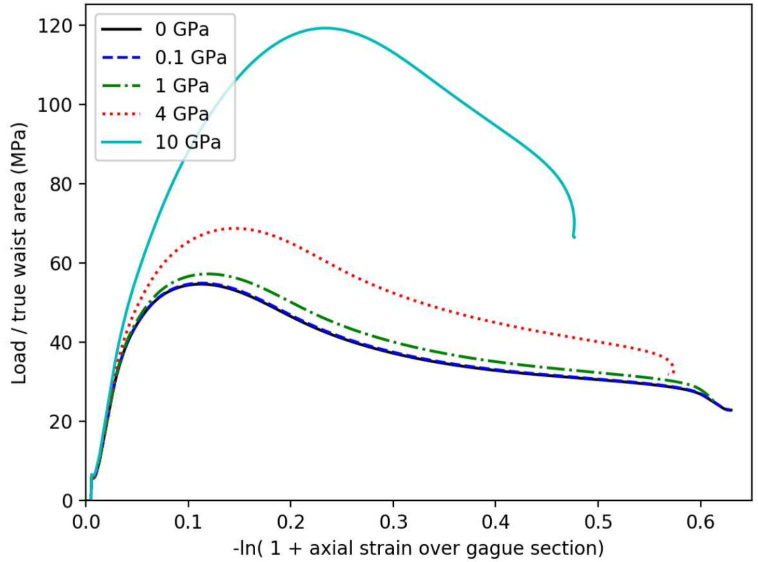


Waisted compression cylinder with hardening

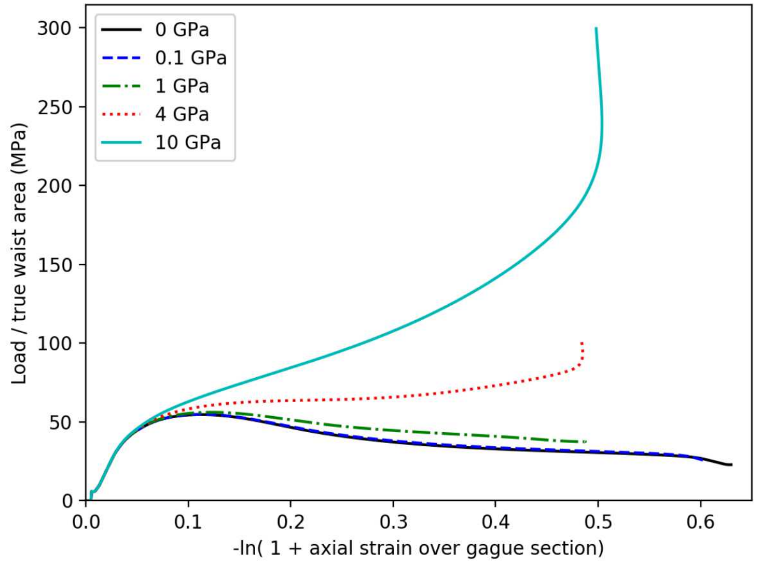


Experimental data

Effect of G_G^1



Effect of G_G^2



Simulation of 828/DEA at RT

Modeling conclusions and future work

- Regularization of mesh-dependent softening behavior with non-local averaging is ineffective in its current form. Adding strain hardening terms helps with regularization, but doesn't fix everything.
- With the addition of strain hardening to the SPEC model there are up to 12 new parameters (glassy and rubbery dependence on 3 strain invariants for bulk and shear). More experiments are needed to calibrate these parameters.
- Such experiments could investigate interesting questions:
  - Are relaxation/creep the same at large static strain/stress as at small static strain/stress?
  - Is deformation induced anisotropy observed and is it important? (This would require a major shift in modeling approach).
  - Does a large static strain change the effective glass transition temperature?

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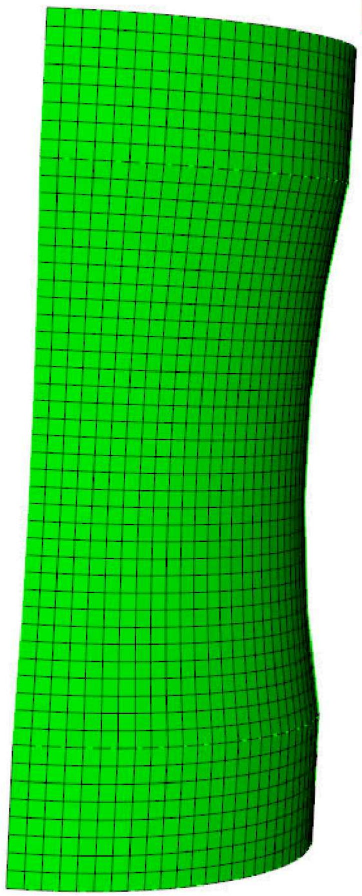
Consequence of post-yield softening

- Post-yield softening generates localized deformation within the waisted specimen (initiates a bulge at the waist that grows with increased compression).
- Similar to Luder's bands in metals.
- Consequently, post-yield deformation is dependent on specimen geometry and loading.
  - cannot simply define a specimen-independent stress-strain curve using the measured load-displacement data.
  - could attempt an iterative approach for identifying the parameters in a large strain polymer constitutive model so that those parameters generate large deformation FEA results that best match the deformed specimen shape (beyond the scope of the present study).
- Anticipate that the overall shape of the specimen-dependent stress-strain curve plotted above should reflect the polymer's yield strength, the post yield stress plateau, and the strain at which final strain hardening occurs.
  - Expect details, like the precise nature of the post-initial yield stress decrease, to be highly specimen dependent.

Model problems

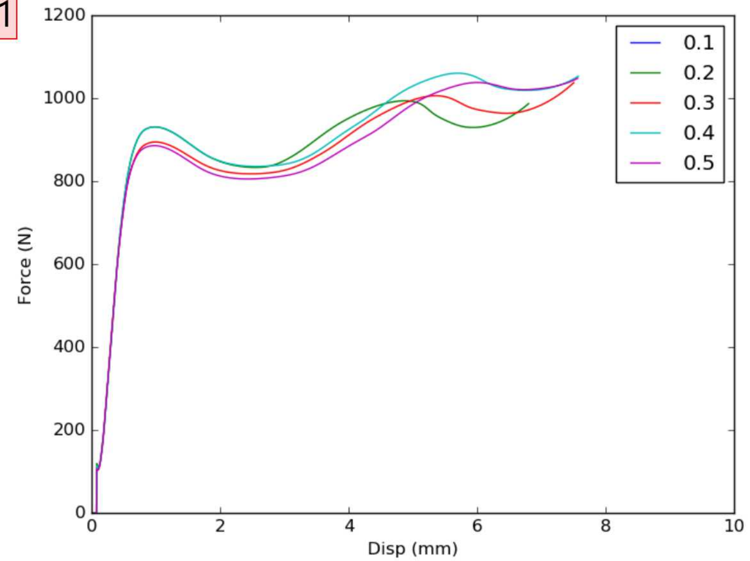
- 2 problems will be considered
  - Waisted compression cylinder to match experiments
  - Butt joint tension
- All analysis uses SPEC non-linear viscoelastic model
- Thermal history in model matches that in physical specimens
- Initial results with tapered cylinder geometry indicate mild mesh sensitivity post-yield
- Efforts to regularize the localization with non-local averaging and a meshless are under way



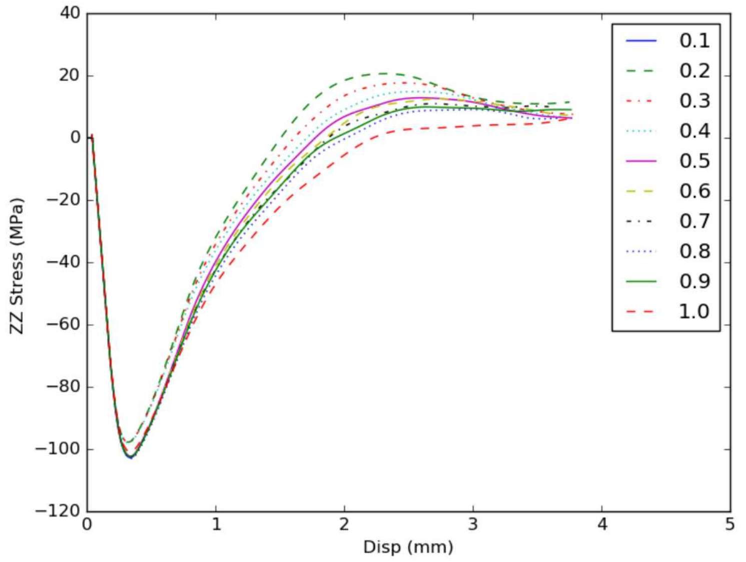
Tapered cylinder geometry

SJG [3]1

Global Force-displacement of tapered cylinder under compression



Stress at center of cylinder exterior under compression (location of onset of softening)



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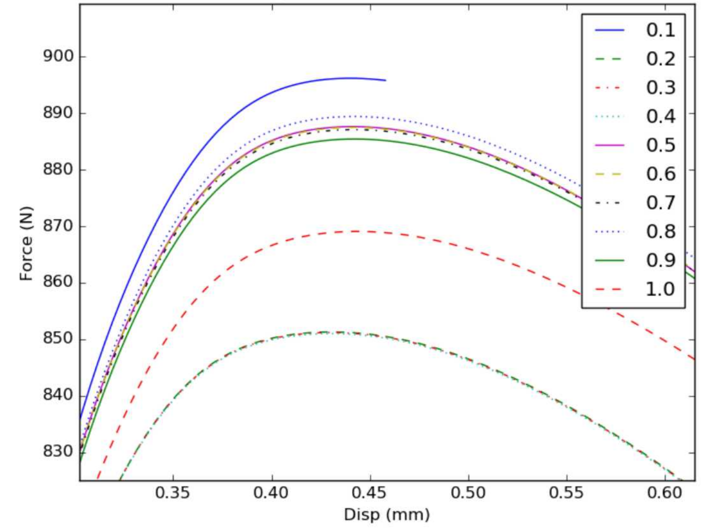
SJG [3]1 Show deformed geometry, maybe compare to physical specimen defromation

Scott J Grutzik, 1/24/2019

Tapered cylinder mesh sensitivity

- Legend indicates characteristic mesh size (0.1 is fine, 1.0 is coarse)
- In the case of the tapered cylinder, results are not extremely sensitive to the mesh
- Nonetheless, they do not converge with mesh refinement
- Onset of softening appears to switch between two slightly different modes. This may be due to solver behavior.

Global force-displacement at onset of softening



Stress at localization region at onset of softening