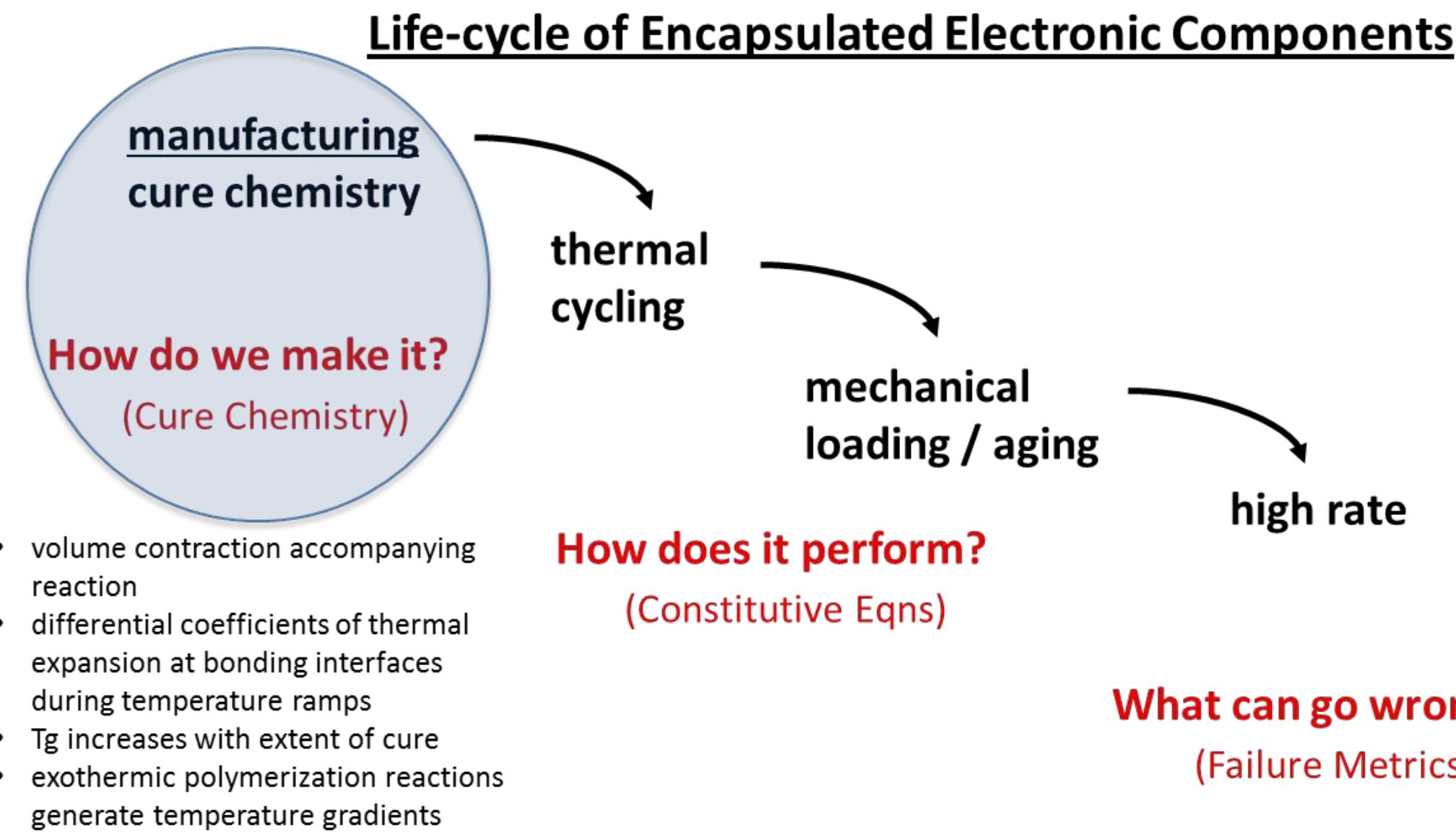


Residual Stress Developed During the Cure of Thermosetting Polymers: Optimizing Cure Schedule to Minimize Stress

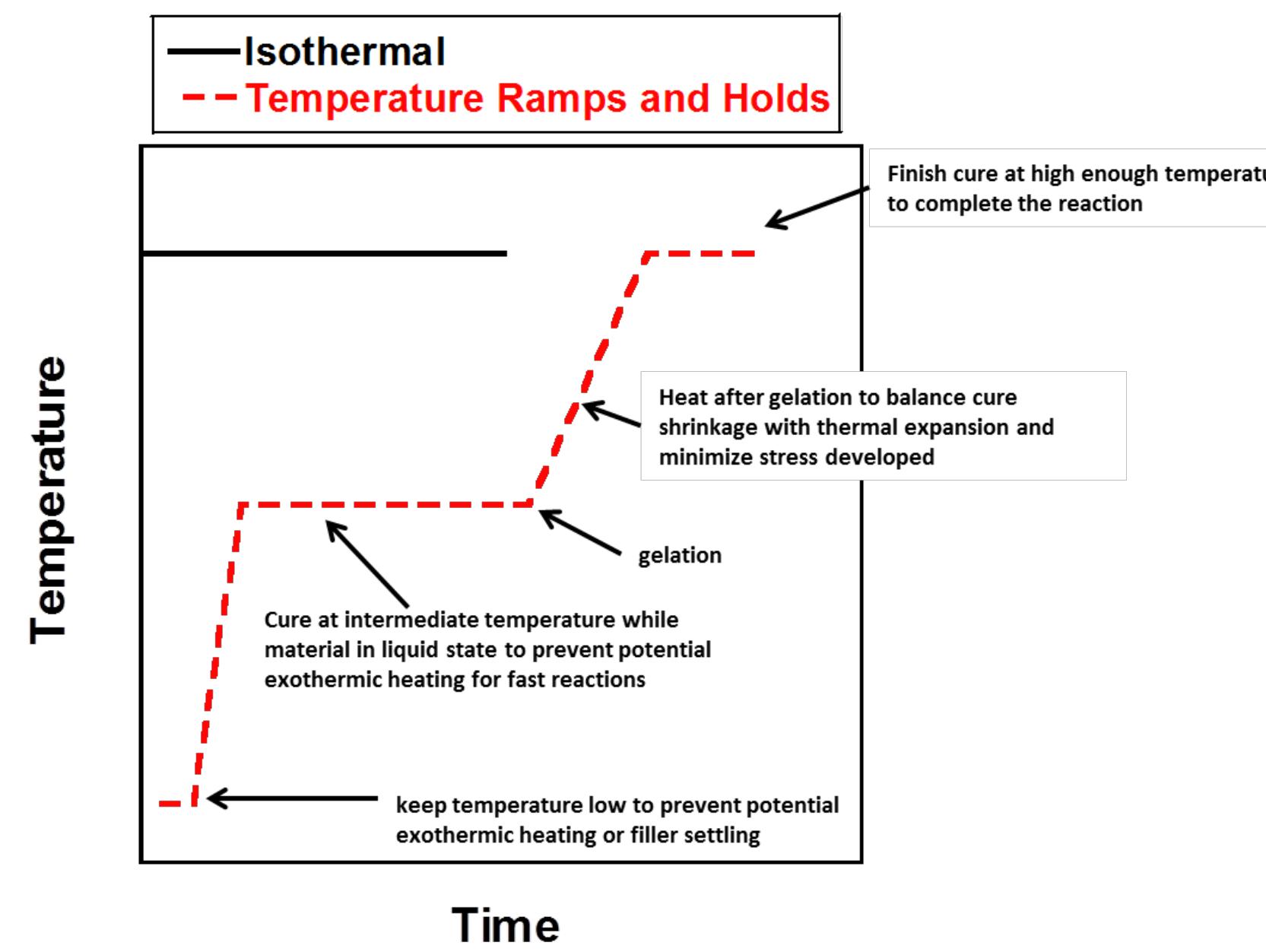
Jamie M. Kropka, Mark E. Stavig, and Rex Jaramillo
Sandia National Laboratories, Albuquerque, NM



Why is Polymer Cure Stress Important?

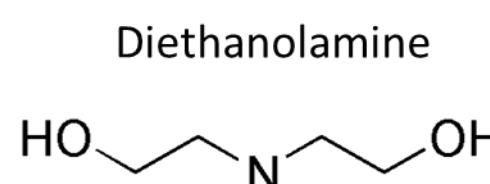
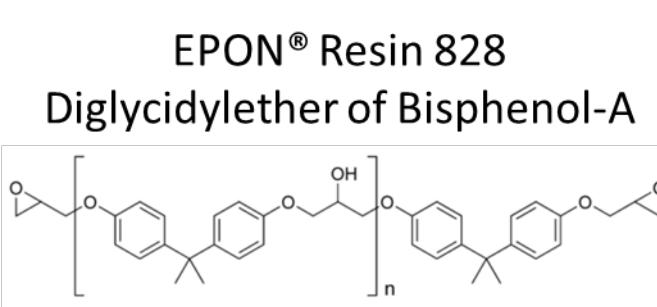


Designing an Optimum Cure Schedule



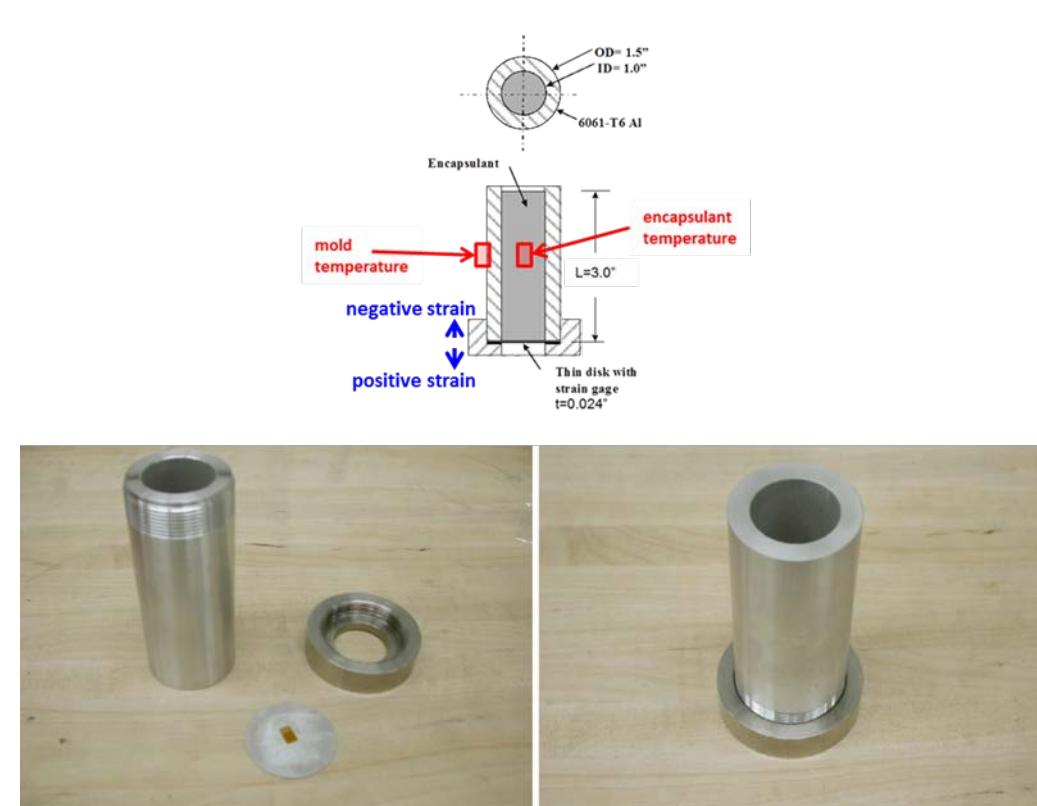
Experimental Materials, Apparatus and Procedure

828/GMB/DEA¹



3M D32 glass microballoons

Thin-Disk-On-Cylinder



Procedure

- Instrument cylinder with strain gauge and thermocouples
- Preheat constituent materials and cylinder to $T = 71^\circ\text{C}$
- Mix and degas constituents
- Pour mixture into cylinder
- Place cylinder into heating chamber and run programmed thermal profile
- Log strain and temperature measurements

¹Mix ratio, cure and typical properties can be found at: http://www.sandia.gov/polymer-properties/828_DEA_GMB.html

Conclusions

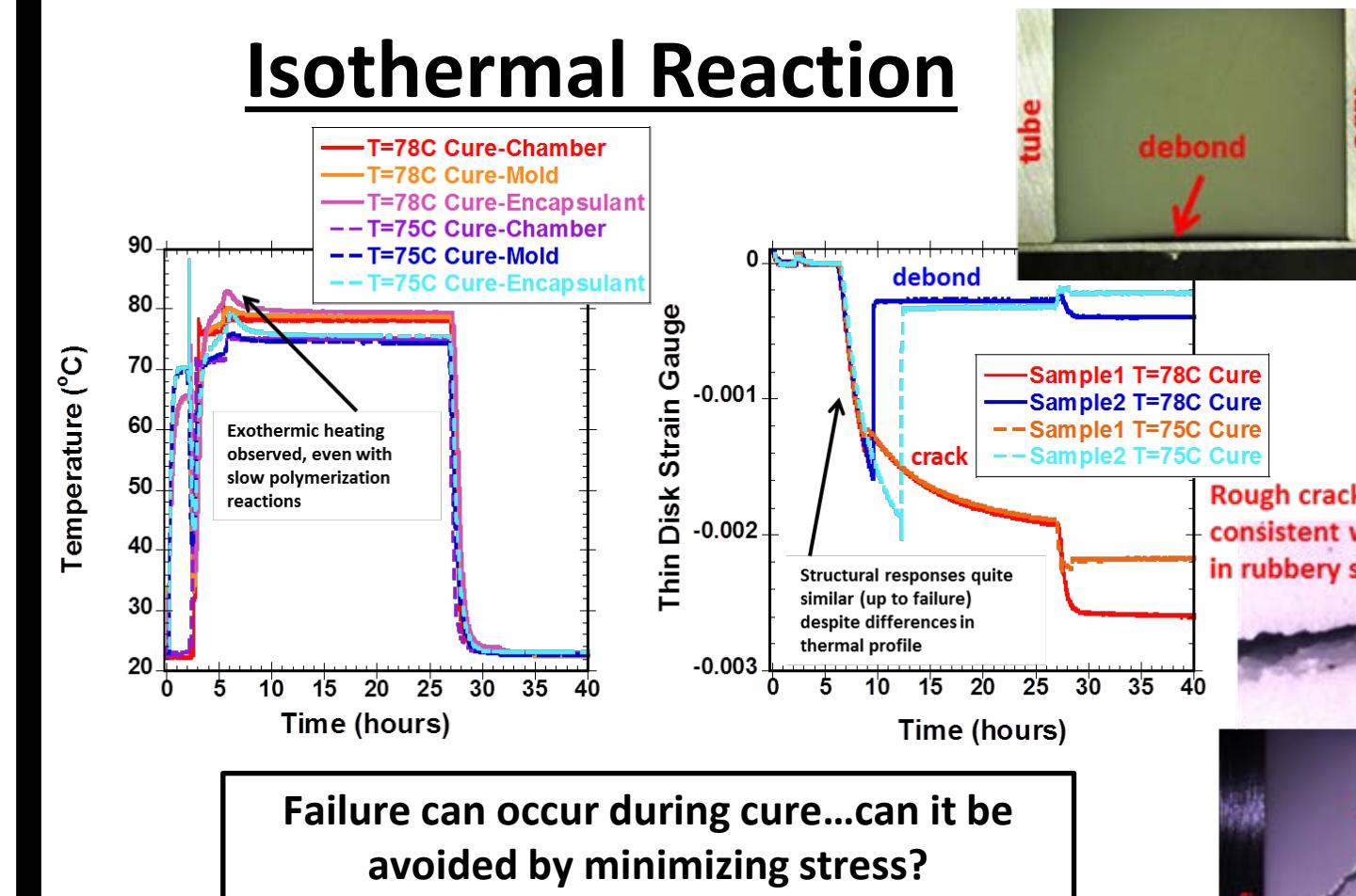
- The Thin-Disk-on-Cylinder structural response test is a powerful tool to design epoxy encapsulant cure schedules experimentally, even when all the details of the material evolution during cure are not explicitly known
- For the 828/GMB/DEA material in the Thin-Disk-on-Cylinder geometry, the stress associated with cure is significant and outweighs that associated with cool down from the final cure temperature to room temperature
- Increasing $T_f - T_{\text{gel}}$ leads to a reduction in cure stress that is described as being associated with balancing some of the 828/GMB/DEA cure shrinkage with thermal expansion
- The ability to tune residual stress associated with cure by controlling $T_f - T_{\text{gel}}$ is anticipated to translate to other thermosetting encapsulation materials (appropriate times and temperatures will vary with material)

Future Directions

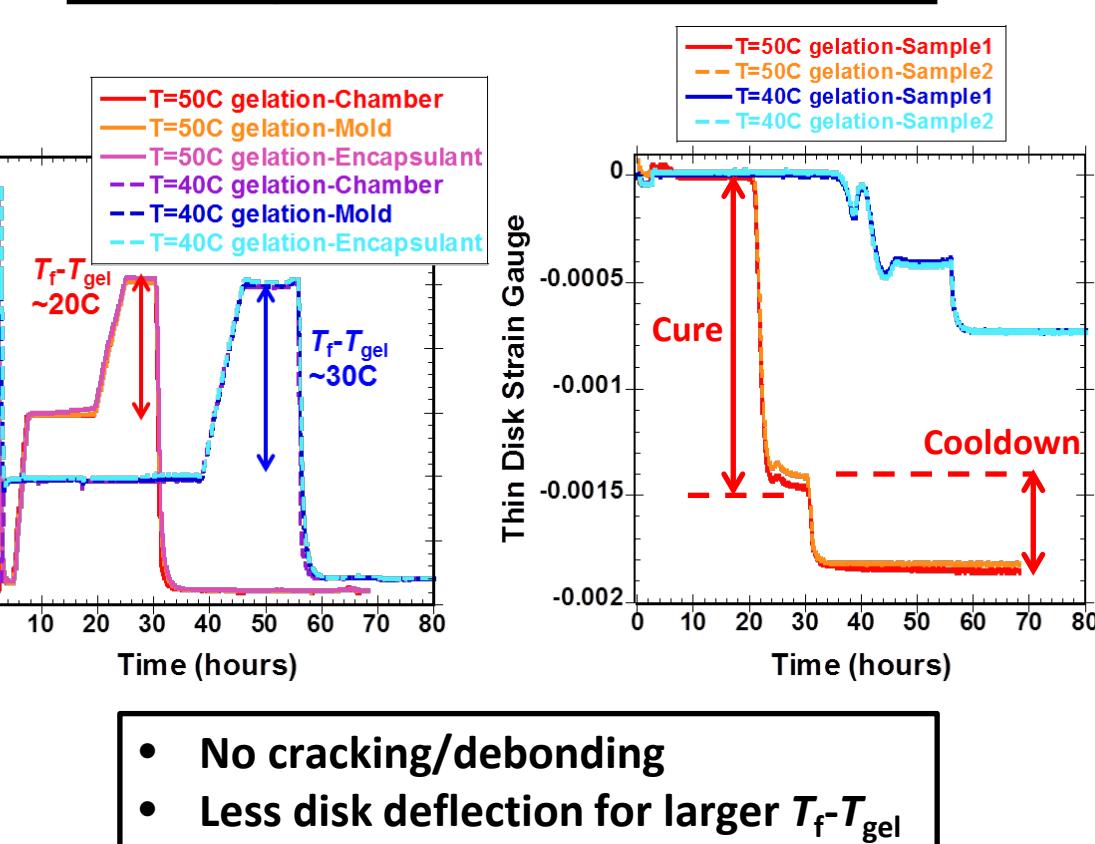
- Changing geometry (particularly the amount of confinement experienced by the thermosetting material) may have a significant effect on the ability to control residual stress associated with cure
- Evaluate layer-by-layer deposition of thermosetting materials versus single "shot" pour encapsulation
- Enable prediction of how the residual stress associated with cure evolves based on a knowledge of the underlying material chemistry and physics involved

Results

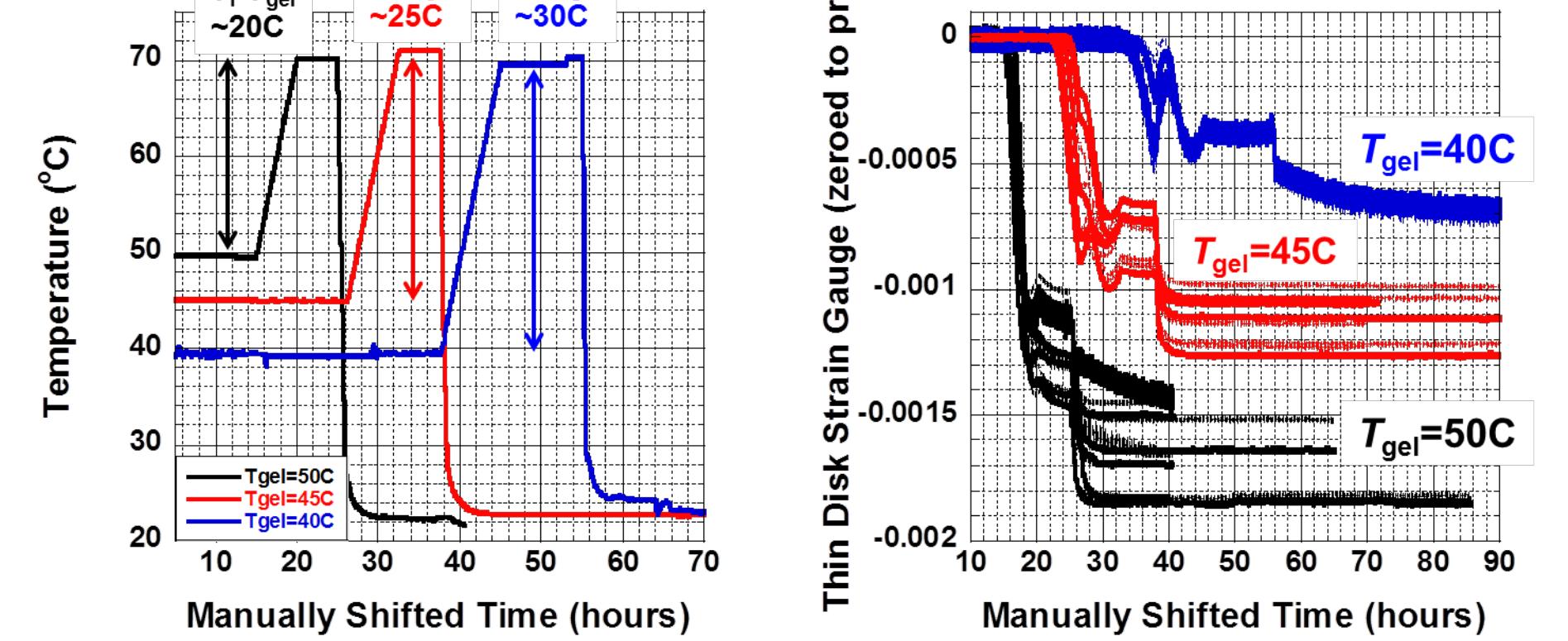
Isothermal Reaction



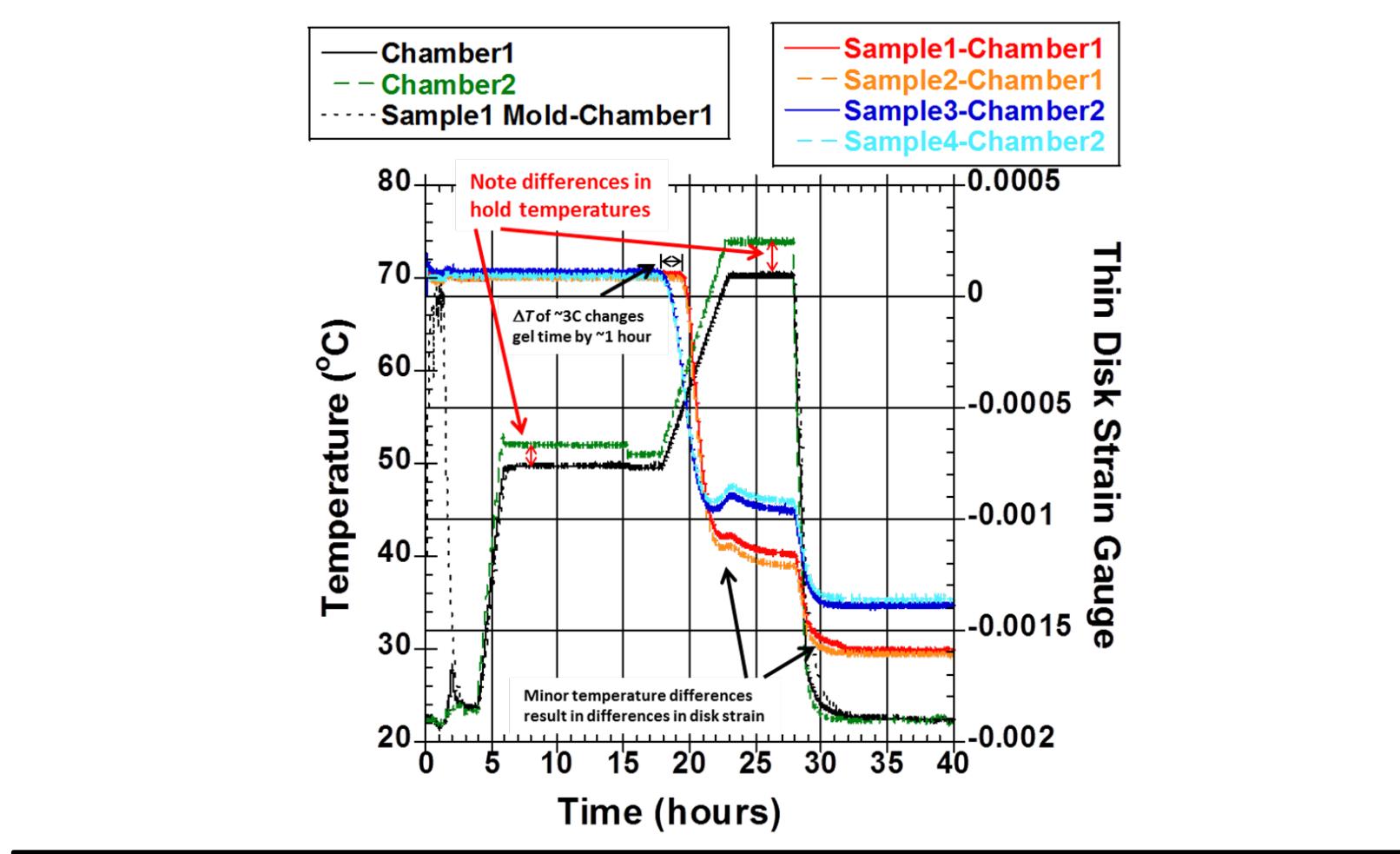
Post-gel Thermal Ramp



Role of $T_f - T_{\text{gel}}$

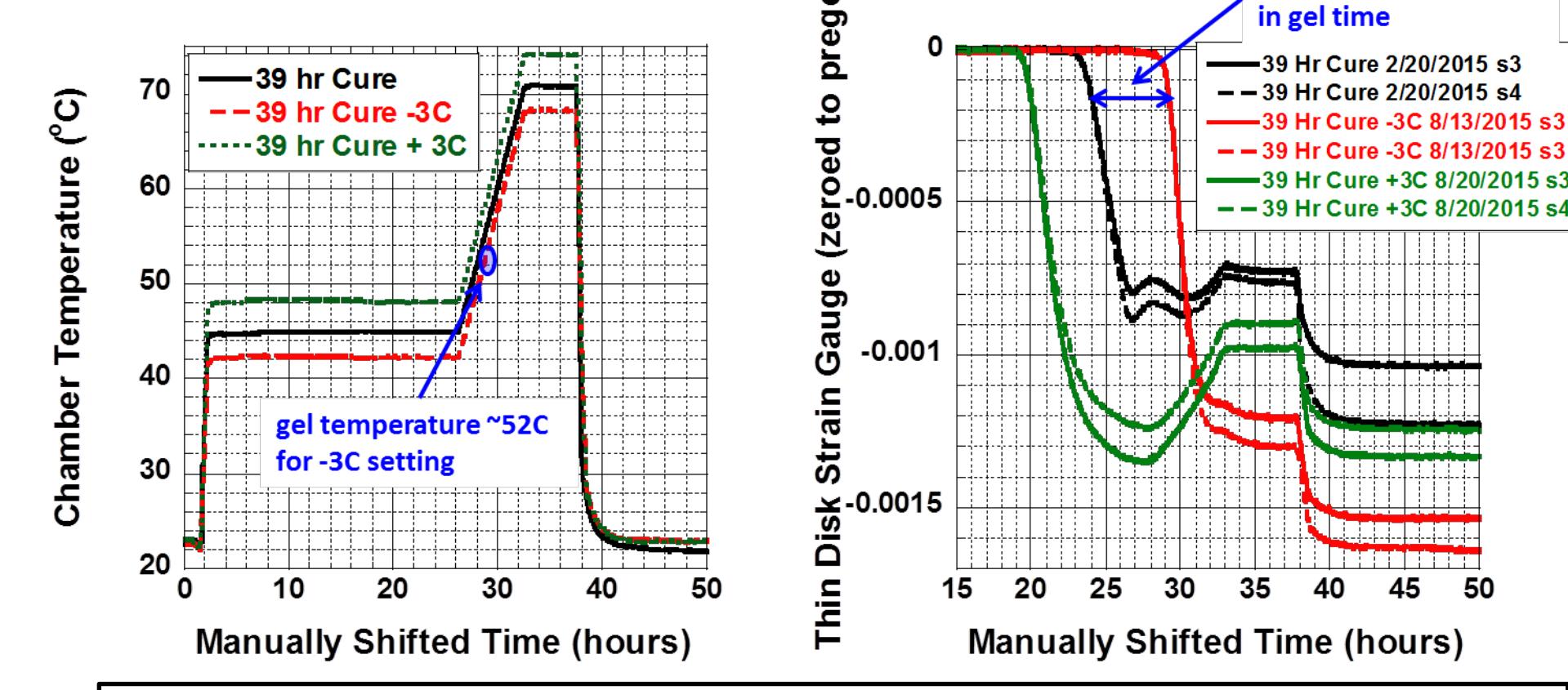


Temperature Sensitivity of Test



Relatively small changes in pre-gel hold temperature affect (1) gel time, (2) gel temperature, (3) initial lid deflection rate, and (4) final lid deflection

Implications of Temperature Tolerances



Both the +/- 3°C offsets result in an increase in the residual stress associated with cure

- 3°C results in a decrease in $T_f - T_{\text{gel}}$ (from baseline +/- 0°C case)
- +3°C offset results in equal $T_f - T_{\text{gel}}$ (to baseline +/- 0°C case)