

Proposal Name and Technical Area: Predictive Modeling of Polymer Mechanical Behavior Coupled to Chemical Change/ Technique development for measuring polymer physical aging

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Other contributors: Mark Stavig, Gabe Arechederra, John McCoy

Work Category: predictive aging models and capabilities, new material aging

Primary material/component aging risk addressed, benefitting components and systems: Chemical and Physical Changes in Epoxy Thermosets used as Encapsulants and Adhesives

Single year, multi-year, or continuation: Continuation

Budget for FY18: Unknown

Technical issue addressed:

Develop an understanding of the evolution of glassy polymer mechanical response during aging and the mechanisms associated with that evolution. That understanding will be used to develop constitutive models to assess the impact of stress evolution in encapsulants on NW designs.

Technical Approach and Results:

Assess the evolution of encapsulant volume and mechanical response during isothermal aging

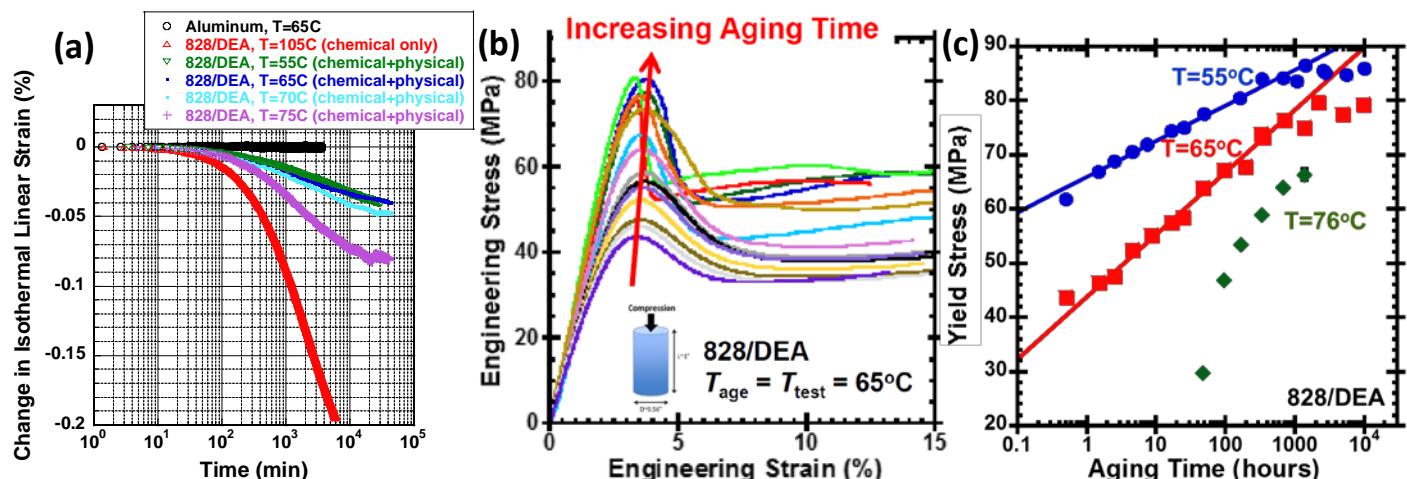


Figure 1. Evolution of encapsulant volume and mechanical response during isothermal aging. Linear strain as a function of aging time (a), compressive stress-strain response as a function of aging time (b), and compressive yield stress as a function of aging time (c).

Findings:

- The 50 nm resolution of the DIL806 optical dilatometer enables quantitative tracking of material length over time

- Chemical changes in the material (e.g., continued cure) can impact volume significantly more than physical aging when remaining reaction potential exists (e.g., for 828/DEA)
- There are four distinguishable changes in compressive stress-strain response with aging: (1) increase in “elastic” compressive modulus, (2) increase in “yield” stress, (3) narrowing of “yield” peak, and (4) increase in “flow” stress
- Changes in yield stress are substantial (for 828/DEA), as high as 80%
- The evolution of yield stress with time changes (and possibly stops) after \sim 30 days (for 828/DEA)

Impact:

- Quantitative knowledge of substantial changes in material mechanical response with aging at temperatures relevant to NW
- High fidelity data that enables testing of constitutive models

Separate chemical and physical contributions to the evolution of mechanical response

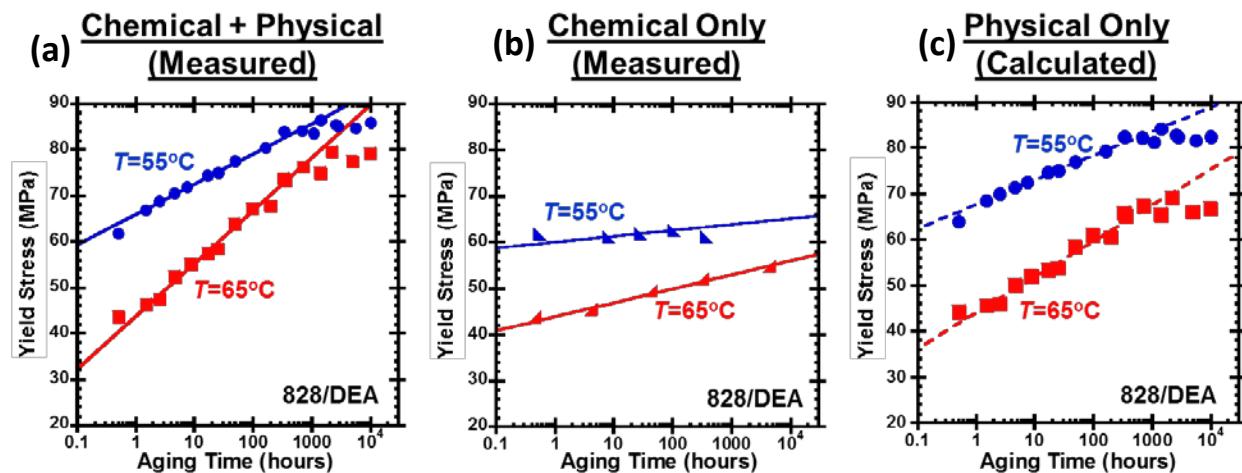


Figure 2. Compressive yield stress as a function of aging time for (a) material chemical and physical changes, (b) material chemical changes only, and (c) material physical changes only.

Findings:

- By thermally annealing the samples above the glass transition temperature (after aging), the physical history of the sample is erased and the chemical-only contributions to the evolution of the compressive stress-strain response are resolved. Physical-only contributions are calculated by subtracting the chemical-only contributions from the total (chemical plus physical) change.
- Physical aging contributes more to the change in yield stress than chemical aging (for 828/DEA)
- When physical-only contributions to the yield stress are calculated, there is little-to-no change in the yield stress (for 828/DEA) at long times ($> \sim$ 30 days). This is suggestive of the attainment of equilibrium.

Impact:

- Physical aging alone can have significant impact on material mechanical response

- At temperatures close to the glass transition (and above), chemical contributions to the change in yield stress are also significant (for 828/DEA)
- Knowledge of conditions under which chemical contributions may be able to be neglected

Simplify isothermal volume evolution measurements to enable parallel assessment of multiple materials

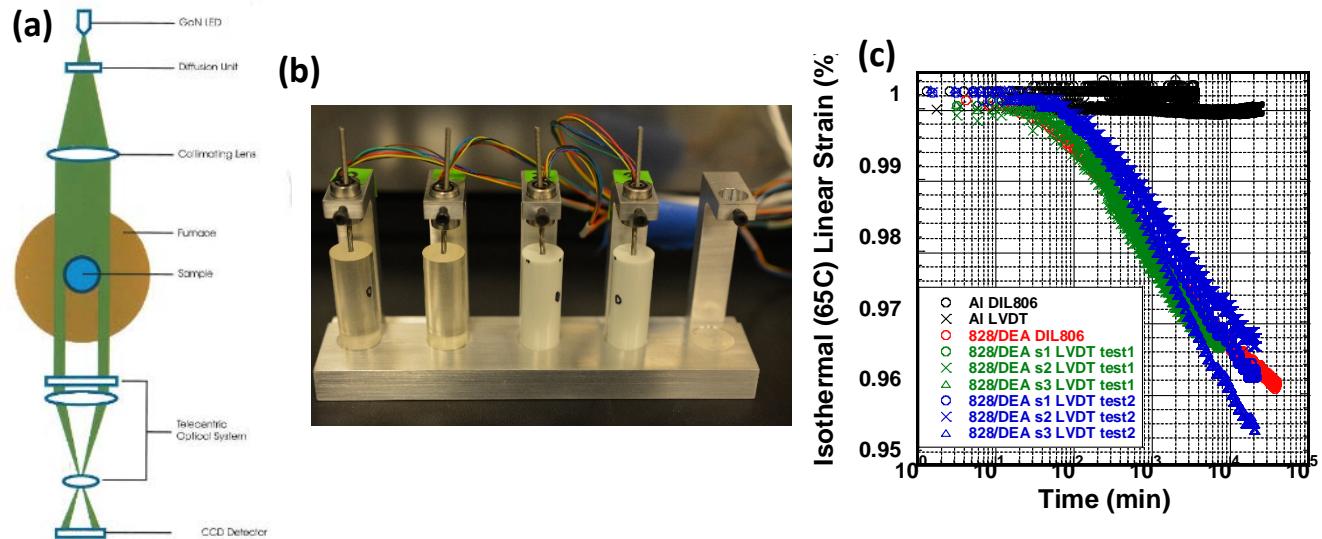


Figure 3. Schematic of the measurement system for the DIL806 optical dilatometer (a), picture of the simple LVDT apparatus (b), and comparison of changes in the isothermal linear strain measured by both apparatus (c).

Findings:

- LVDT across a sample measures approximately the same isothermal shrinkage of epoxy encapsulant (as it sits below its glass transition temperature) as the DIL806 non-contact optical dilatometer
- While LVDT readings in the test apparatus do not measure pure material dimensional changes during temperature ramps, initial indications suggest that material dimensional changes may be able to be extracted

Impact:

- The LVDT apparatus enables measurement of multiple samples/materials simultaneously, whereas the DIL 806 optical dilatometer can only measure one sample at a time. Thus, the LVDT apparatus provides a more economical and time efficient means to assess isothermal material volume changes in the hundreds of encapsulant/adhesive materials used in NW systems

Impacts to the NW program/Enhanced Surveillance mission

- **New understanding or capability developed**

WPA Deliverables/Date Completed

1. Report the effect of isothermal aging on 828/DEA volume and compressive stress-strain response/ September 2017 (JOWOG 28, 32nd Polymer Degradation Discussion Group Conference, Gabe Arechederra MS Thesis)
2. Define measurement apparatus and technique to assess volume relaxation process of multiple samples simultaneously/ September 2017

Other relevant information: