SAND2015-3052PE

Bonded Joint Analysis Annual Update – Experiments

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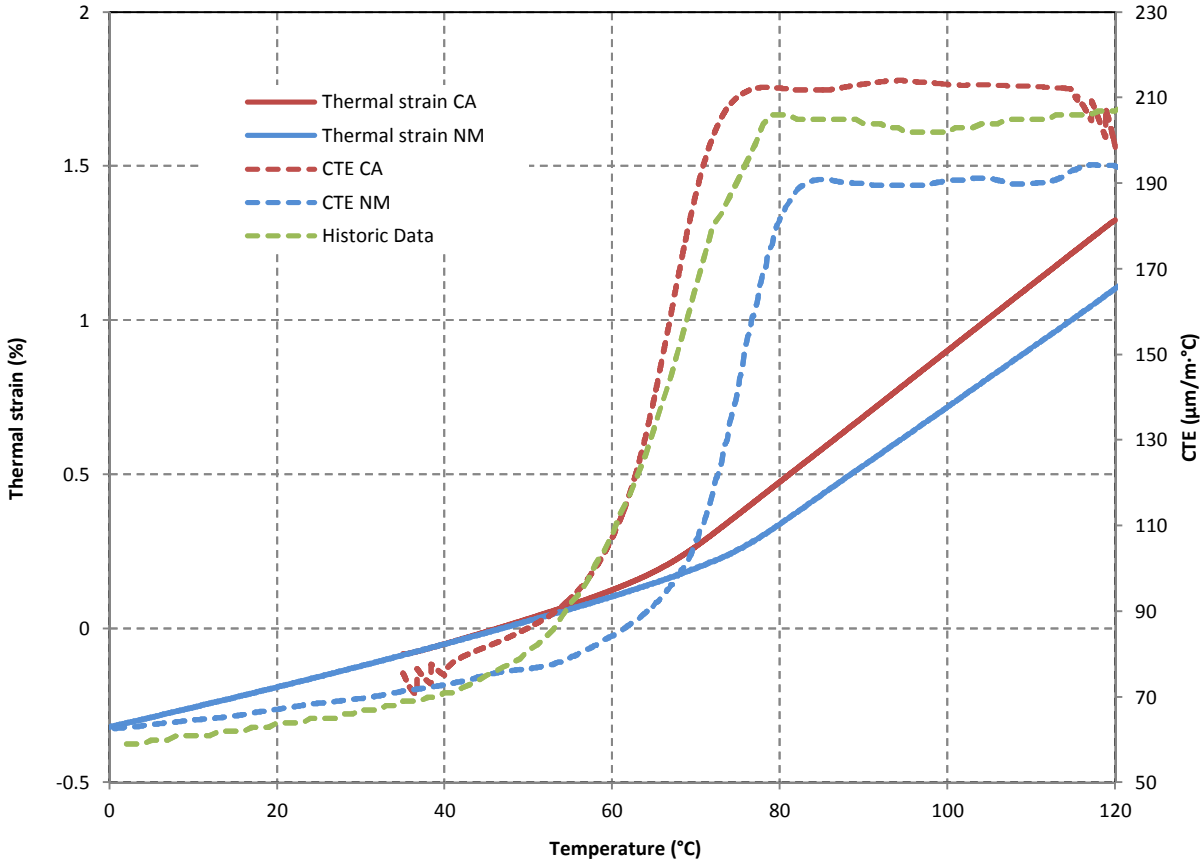
Material Characterization and Fracture Summary

- A set of four unique experiments are utilized for material characterization
  - Dynamic Mechanical Analysis (DMA) – used to determine the complex moduli of the material
  - Thermal Mechanical Analysis (TMA) – used to determine the temperature dependent thermal expansion and glass transition temperature (T_g)
  - Compression yield – used to fit parameters to predict glassy yield as a function of temperature
  - Tension – used to calibrate the temperature dependent bulk modulus
- Three Fracture Tests are to be completed
  - Double Cantilever Beam (DCB) – used to determine Mode I fracture toughness
  - End Notched Flexure (ENF) – used to determine Mode II fracture toughness
  - Notched Coating Adhesion (NCA) – a mixed mode adhesion test to determine moisture effects
- Conditions for the fracture tests include:
  - Temperature: both room and high (below T_g)
  - Bond thickness: glass bead controlled thin (0.005”) and thick (0.0234”)
  - Moisture: uncontrolled lab conditions and fully saturated

DGEBA/DEA (828/DEA) Verification



Thermal strains and coefficient of thermal expansion from TMA measurements

DGEBA/DEA (828/DEA) Moisture Expansion

- In the Notched Coating Adhesion, the expansion due to moisture is required in order calculated the mixed mode energy release rate; given as:

$$G = \frac{h}{E}\left\{[(\sigma_t + \sigma_m)^2 + (\sigma_t + \sigma_m)\varepsilon E](1-\nu) + \frac{1}{2}(\varepsilon E)^2\right\}$$

  where ε is the uniaxial strain in the specimen, σ_m is the biaxial stress due to moisture, σ_m is the biaxial stress due to curing and E and ν are the instantaneous elastic constants of the adhesive.
- The coefficient of moisture expansion is defined as

$$CME = \frac{\Delta\varepsilon}{\Delta M}$$

  where:

$$\Delta M = \frac{m_1 - m_o}{m_o}$$

- Specimens where placed in a 90C 100% humidity environment for a number of weeks to ensure full saturation. Then the specimens where brought back to room temperature and remained at 100% humidity for additional weeks.
- The mass of a specimen was measured then immediately placed in a thermal mechanical analyzer (TMA) at 24 C to dry. The change in strain and final mass was recorded.
- The CME of the 828/DEA was found to be 0.194, which matches well to similar material's data found in literature

DGEBA/DEA (828/DEA) Notched Coating Adhesion

- A series of proof of concept experiments were conducted
- These experiments assessed various sensitivities such as:
  - Initial flaws
  - Grip configuration
  - Test rate
  - Initial pre-crack size and uniformity
- The preliminary results for the average stress in the specimen at the moment of debonding are: 316.9, 318.1, 318.3 and 317.3 MPa (very repeatable)

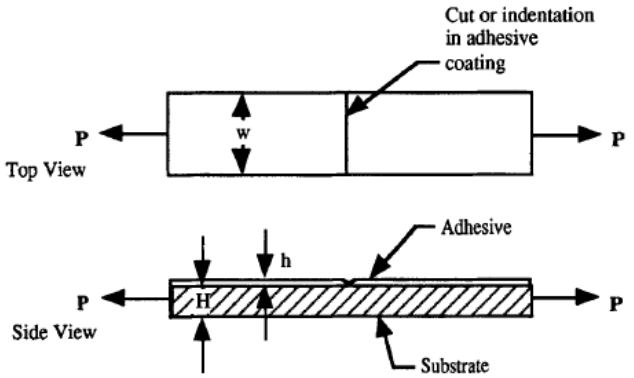
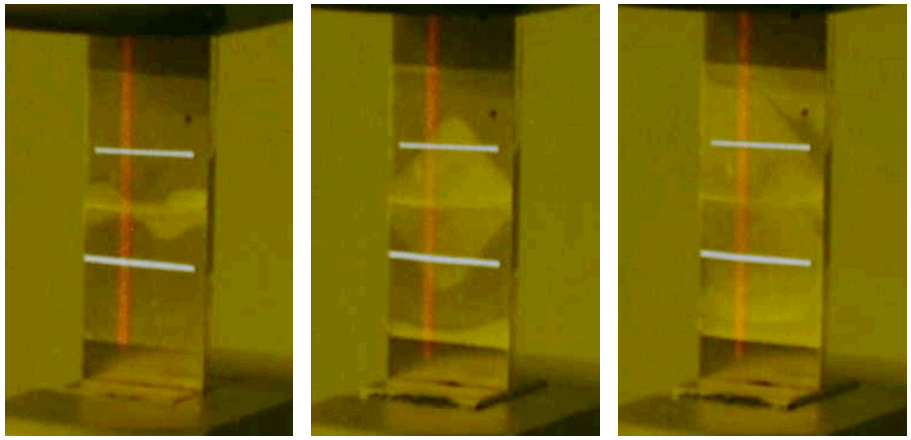


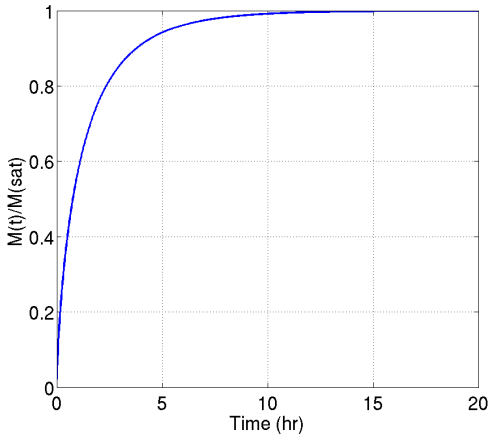
FIGURE 1 Notched coating adhesion specimen.

Pre-crack Propagation Full Debond



Increasing Load →

Screen shots of preliminary tests

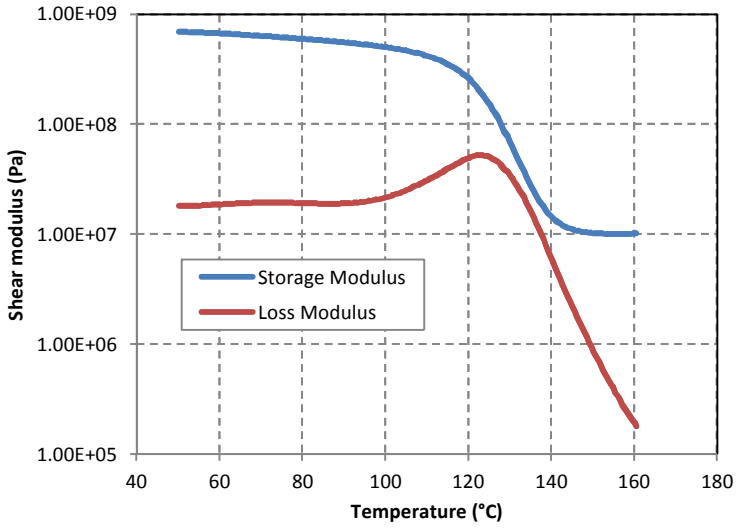


1D Moisture infusion into coating:

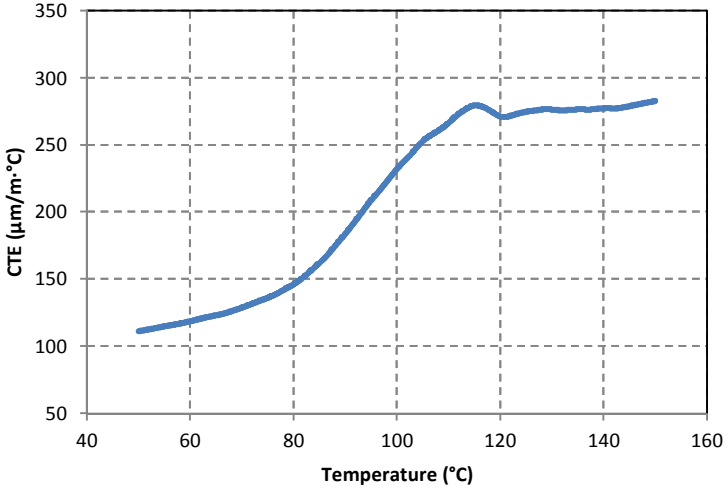
- 0.999 saturation occurs at 16.4 hr
- Moisture infusion data is measured in Sandia New Mexico's Materials Sciences and Engineering Center and is included in an unpublished draft by Jamie M. Kropka, Douglas B. Adolf, Scott Spangler, Kevin Austin and Robert S. Chambers

Loctite EA 9360 Characterization Experiments

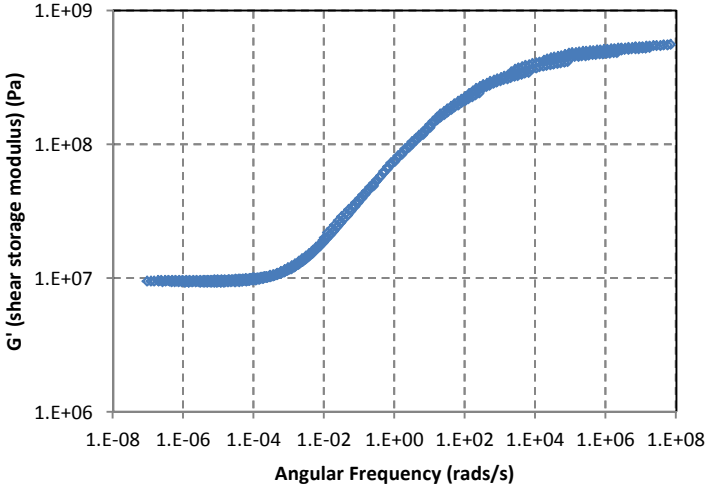
Test Name	Temperature	Moisture Content
DMA	Various	Dry
TMA	Various	Dry
Compression Yield	RT	Dry
Compression Yield	45 C	Dry
Compression Yield	65 C	Dry
Tensile Modulus	RT	Dry
Tensile Modulus	45 C	Dry
Tensile Modulus	65 C	Dry
Tensile Modulus	115 C	Dry
Tensile Modulus	125C	Dry
Tensile Modulus	135 C	Dry



Dynamic shear moduli versus temperature



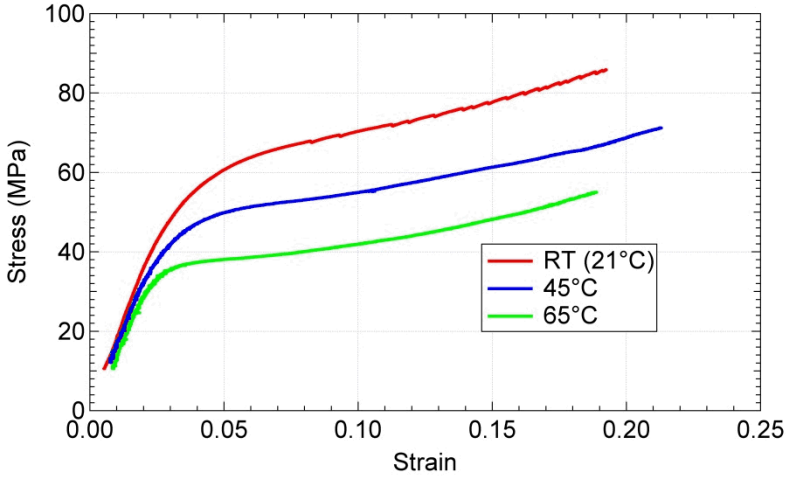
Coefficient of thermal expansion from TMA measurements



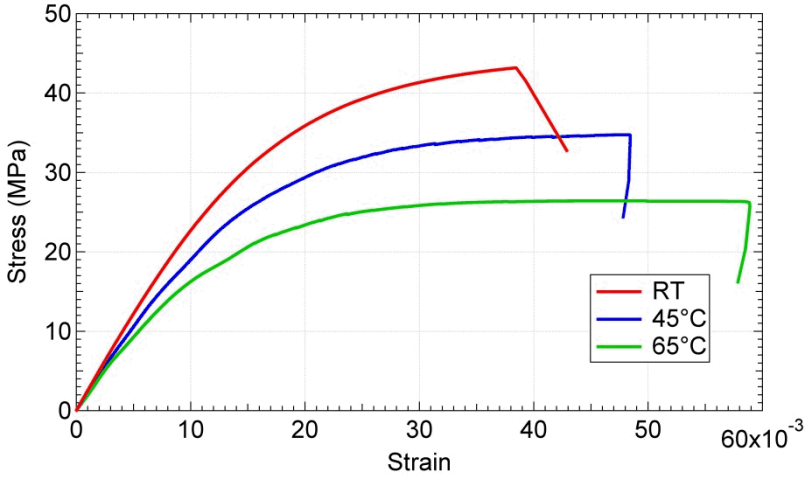
Master Curve at 75C from DMA measurements

Loctite EA 9360 Characterization Experiments

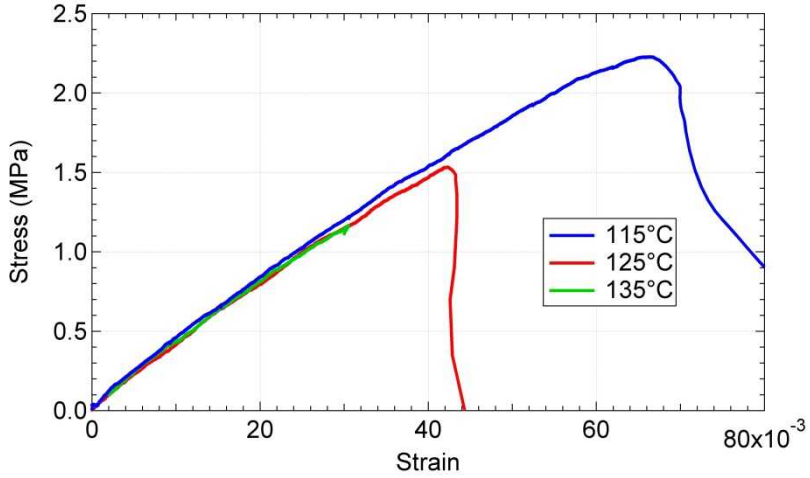
- Tension
  - Used to calibrate the temperature dependent bulk modulus
  - D638 − 14: Standard Test Method for Tensile Properties of Plastics
- Compression
  - Used to calibrate relaxation acceleration rate parameter



Compression yield at temperatures below T_g



Tensile yield at temperatures below T_g



Tensile yield at temperatures above T_g

Fracture Characterization

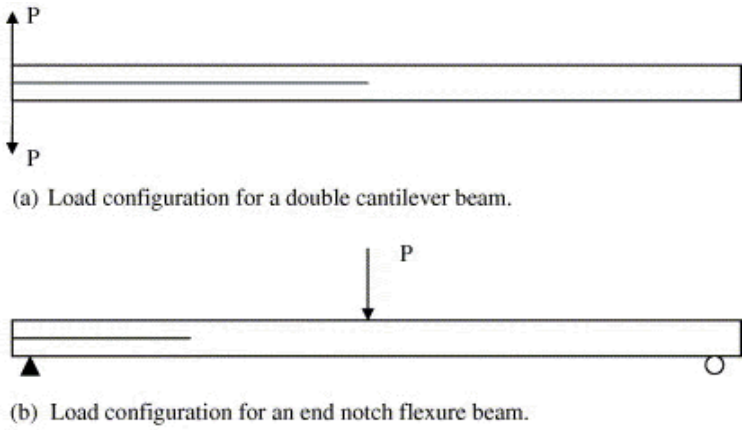
DGEBA/DEA (828/DEA) Fracture Tests

Test Name	Bond Thickness	Temperature
DCB	Thin	RT
DCB	Thin	59°C
DCB	Thick	RT
DCB	Thick	59°C
ENF	Thin	RT
ENF	Thin	59°C
ENF	Thick	RT
ENF	Thick	59°C

- 6061 Aluminum Substrates
- The fracture test are planned at three replicates per test condition
- All bonded surfaces are BR-127 primed as per manufacturer's recommendations
- Pre-cracks are embedded with Teflon tape
- Hinges are bonded to the SCB specimens with a Hysol adhesive of equal or greater strength than the test adhesive

Loctite EA 9360 Fracture Tests

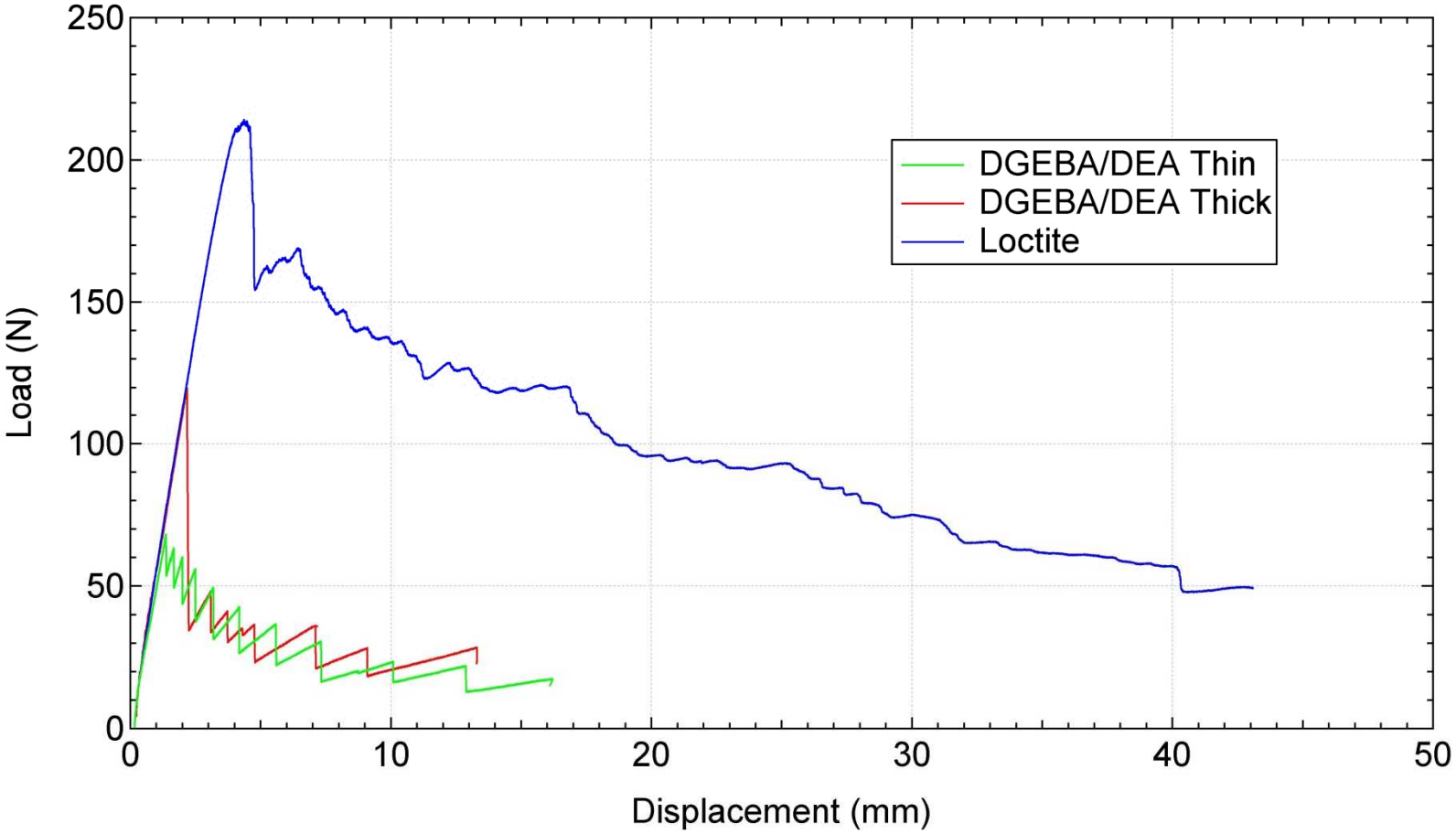
Test Name	Bond Thickness	Temperature
DCB	Thin	RT
DCB	Thin	80°C
ENF	Thin	RT
ENF	Thin	80°C



DCB (a) and ENF (b) load configurations

Fracture Characterization

Preliminary results



DCB Load displacement plot for Hysol 9360 and 828/DEA thick and thin specimens at room temperature

Summary of Experiments to Date

- All specimen manufacturing has been completed
- Material Characterization experiments have been completed
  - DMA
  - TMA
  - Tension and Compression Yield
  - Moisture expansion and absorption rate
- Fracture specimens are made and some have been tested
  - Room Temperature DCB for each material and thickness have been completed
  - High temperature and ENF experiments are to be completed shortly
- While much time has been spent on research and exploration of experimental methods, the total time to execute the reduced experimental procedure is expected to be reasonable