TEMPERATURE EFFECTS ON ADHESIVE BOND STRENGTHS AND MODULUS FOR COMMONLY USED SPACECRAFT STRUCTURAL ADHESIVES

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Agenda

• Purpose and Scope
  – Establish property guidelines to help select adhesives.

• Lapshear Testing (ASTM D1002)
  – Sample preparation
  – Test methods
  – Results (Explanation of statistical analysis/ reason for using B-basis allowables)

• Dynamic Mechanical Analysis (DMA)
  – Sample preparation
  – Test Methods
  – Results

• Future Work
  – Different Adherends
  – Tensile testing adhesive dog bones (ASTM D638)

• Questions?
The purpose of this effort was to study how changes in temperature affected:
- Average bond strength
- Loss and storage modulus as a function of temperature.

Seven commonly used adhesives were tested:
- Hysol EA9394, EA9309.3, EA9360, EA9361
- STYCAST 2850FT Black with Catalyst 9 and 24 LV
- Scotch-Weld EC 2216

Two Test types:
- ASTM D1002
- Dynamic Mechanical Analysis

Using these two methods, data was collected, providing details about the bondline properties at various temperatures:
- ASTM D1002 Data used for B-basis reference database and graphs over temp range
- DMA provided graph of Storage and Loss modulus over selected temp range
ASTM D1002-
Sample Fabrication and Surface Preparation

- Sample Fabrication for sets of five single lap shear joints
  - Adherends made from Aluminum 6061-T6 (same lot of material for all)
  - Cut using water jet method to minimize oils deposited on the surface
  - After machining, panels were then deburred and cleaned

- Surface Cleaning
  - Cleaning using JPL specification for bonding
  - Elevated temperature alkaline cleaning
  - Elevated temperature Sodium Dichromate bath
  - Primed surface using BR-127

- Adhesive mixed and accelerated cure per manufacturer’s datasheet

- Bonding
  - 5 mil stainless steel bond wire used to maintain uniform bondline
  - Alignment maintained using lapshear bonding tool
Deviations from ASTM D1002 were for adherend construction

- Thicker than specified
  - The panels were 3.2 mm (0.125”) thick
  - Minimizes issues related to adherend bending and twisting during the pull test
- Through hole through tops of adherend
  - 9.5mm holes were drilled into the ends of the lap shear specimens
  - Eliminated grips “freezing up” at low temperatures

Figure 1. Modified Test Panel
Testing Procedure

- Lap shear coupons pulled using an Instron testing machine with a 22,000 lb load cell

- Sample loaded into the machine testing grips and pin and clevis at low temperatures

- Pulled at rate of 2.03 mm/min (0.08 in/min)

- Load vs. displacement results were recorded with Labview

- Thermal couple attached to every specimen to ensure testing done at correct temperature
  - Allowed to equilibrate at temperature for a minimum of 5 minutes

- All equipment was calibrated at time of test

- 10 coupons tested at each temperature from -150 °C to 175 °C
• Data complied using Stat 17 for B-basis allowables

<table>
<thead>
<tr>
<th>Temperature</th>
<th>9394 (MPa)</th>
<th>2216 (MPa)</th>
<th>2850-9 (MPa)</th>
<th>2850-24LV (MPa)</th>
<th>9309.3 (MPa)</th>
<th>9360 (MPa)</th>
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Results - Table B-Basis Allowables (Cont)

- Data complied using Stat 17 for B-basis allowables in English units

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<th>Temperature</th>
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Results - Graph of B-Basis Allowables

- Data compiled using Stat 17 for B-basis allowables in English units

B-Basis Test Results

- Ultimate Strength (psi) vs. Temperature (°C)

- Data points for different materials and temperatures.
Results - Graph of B-Basis Allowables for Stycast 2850FT Black Catalyst 9 and 24LV

- Data complied using Stat 17 for B-basis allowables in English units

B-Basis Test Results for Stycast 2850FT Catalyst 9 and 24LV

Ultimate Strength (psi) vs. Temperature (°C)
Results - Graph of B-Basis Allowables for EA9309.3, EA9361, and EC 2216

- Data complied using Stat 17 for B-basis allowables in English units

B-Basis Test Results for EA9309.3, EA9361, and EC2216
Results- Graph of B-Basis Allowables for EA9360 and EA9394

- Data complied using Stat 17 for B-basis allowables in English units.

B-Basis Test Results for EA9360 and EA9394
Dynamic Mechanical Analysis – Sample Preparation and Testing

• Sample Preparation:
  – Samples mixed and cured per manufacturer’s datasheet
  – Cured as a flat panel (6mm thick) and cut into 25mm x 75mm x 6mm bars
  – Further cut down in Analytical Chemistry Lab using fine hacksaw
    ▪ Approx. 17.5mm x 13mm x 3 mm

• Testing
  – Performed on a TA Q800 DMA instrument
  – Configured in a single cantilever clamp mode
  – Temperature range from -130ºC to +150ºC; ramp rate 5ºC per minute
    ▪ Rate set to maintain thermal equilibrium in sample
  – Done at constant frequency (1 Hz) and constant amplitude (50 µm)
  – Storage and Loss modulus were graphed from results
Results - Example of DMA Graph (9309.3)

Sample: Ojeda 9309-3 purple color
Size: 17.5000 x 12.7000 x 3.0400 mm
Method: Temperature Ramp
Comment: Ojeda sample 9309-3, purple color, adhesive

File: C:...\OJEDA\9309-3 purple adhesive.001
Operator: WH
Run Date: 20-Jan-2011 08:56
Instrument: DMA Q800 V7.5 Build 127
DMA Results

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<th>2&lt;sup&gt;nd&lt;/sup&gt; Tg (ºC)</th>
<th>3&lt;sup&gt;rd&lt;/sup&gt; Tg (ºC)</th>
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Table 1. Glass Transition and Loss Modulus Temperatures

- Several Tan Delta and Loss Modulus peaks for each sample is indicative of morphological inhomogeneities
  - Attributed to a polymer blend, polymer “alloy” or a polymer that has additives (such as chain extenders or cross-linking agents)

- In general, the modulus results correlated well with the variations in adhesive bond strength as a function of temperature
Future Work

• Perform lapshears (ASTM D1002) using different adherend materials bonded with EA9309.3 and EA9394
  – Titanium
  – Invar

• Determine the tensile modulus as a function of temperature using ASTM D638 - Test Method for Tensile Properties of Plastic
Questions?