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?

Purpose and Scope of Testing

- 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

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

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

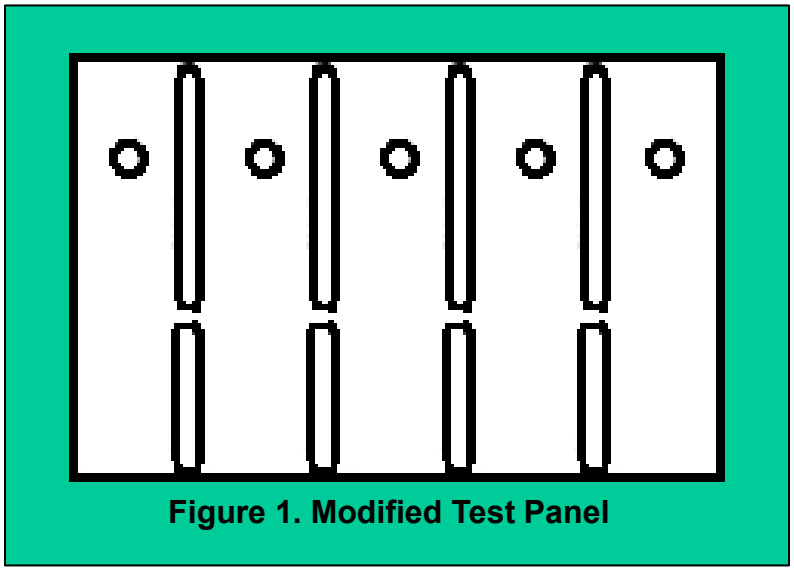


Figure 1. Modified Test Panel

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

Results- Table B-Basis Allowables

- Data complied using Stat 17 for B-basis allowables

Temperature	9394 (MPa)	2216 (MPa)	2850-9 (MPa)	2850-24LV (MPa)	9309.3 (MPa)	9360 (MPa)	9361 (MPa)
-150	15.2	15.8	7.8	15.7	15.2	18.3	24.9
-100	17.7	17.0	12.3	16.3	22.5	17.9	24.5
-70	15.4	14.5	12.5	24.2	39.1	20.8	25.9
-40	18.6	23.2	18.5	24.8	45.7	26.6	34.4
-10	21.0	29.3	16.0	22.7	44.0	31.7	35.7
25	27.2	25.3	15.0	23.3	37.6	35.3	27.4
50	26.4	11.7	17.5	9.1	26.6	29.1	17.5
75	18.8	6.1	16.2	4.3	19.0	23.8	8.0
100	15.9	3.8	11.4	3.2	5.0	16.6	4.8
125	12.9		8.5	2.7			
150	8.2		6.0	2.9			
175	4.7						

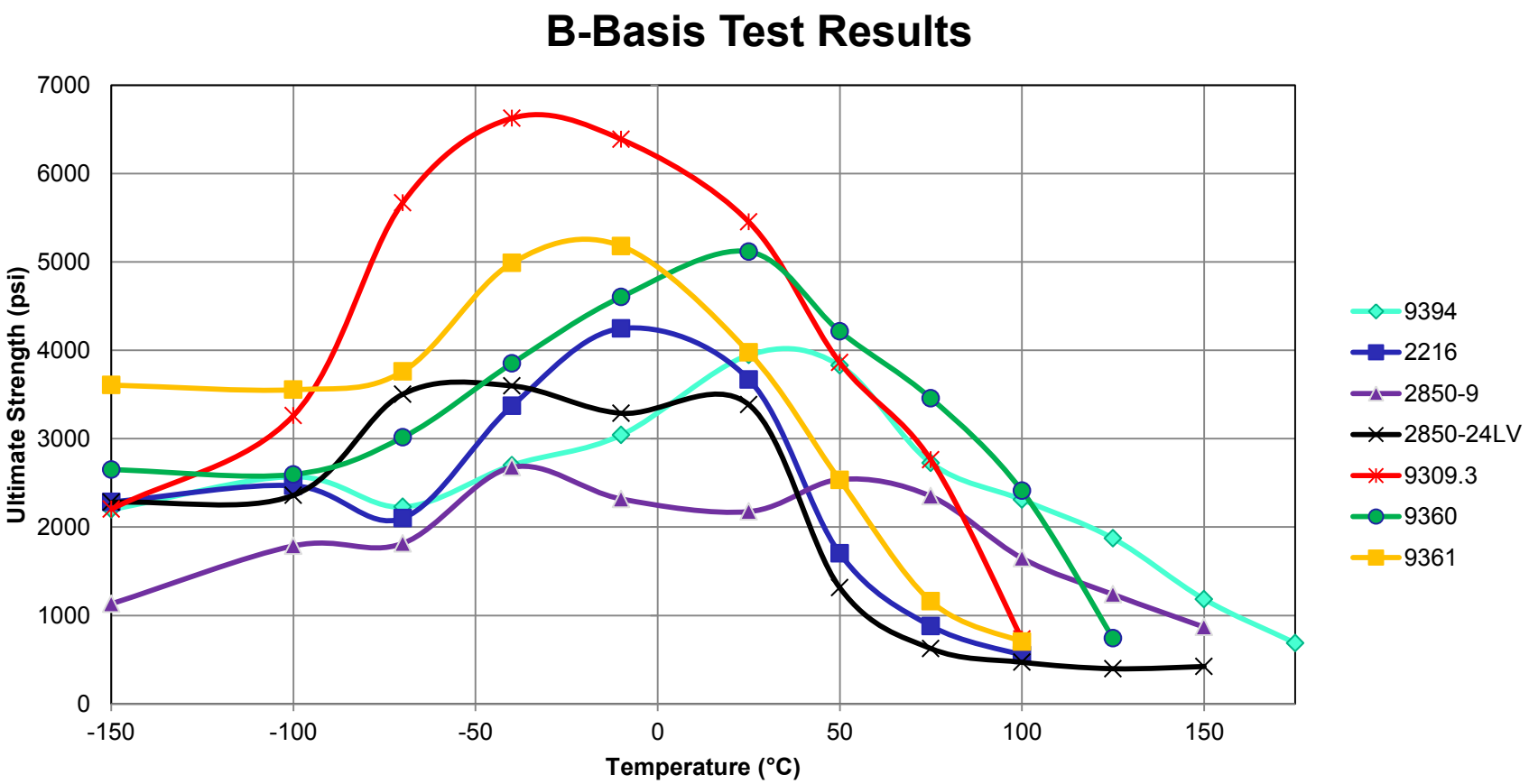
Results- Table B-Basis Allowables (Cont)

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

Temperature	9394 (psi)	2216 (psi)	2850-9 (psi)	2850-24LV (psi)	9309.3 (psi)	9360 (psi)	9361 (psi)
-150	2199	2285	1131	2280	2201	2650	3607
-100	2570	2468	1787	2359	3261	2596	3554
-70	2227	2100	1815	3503	5670	3015	3760
-40	2700	3371	2676	3598	6627	3852	4988
-10	3042	4247	2318	3288	6388	4604	5180
25	3946	3671	2175	3386	5454	5117	3978
50	3829	1701	2539	1313	3862	4214	2531
75	2726	880	2348	625	2761	3458	1160
100	2309	552	1648	470	732	2410	703
125	1872		1238	397		742	
150	1184		868	422			
175	688						

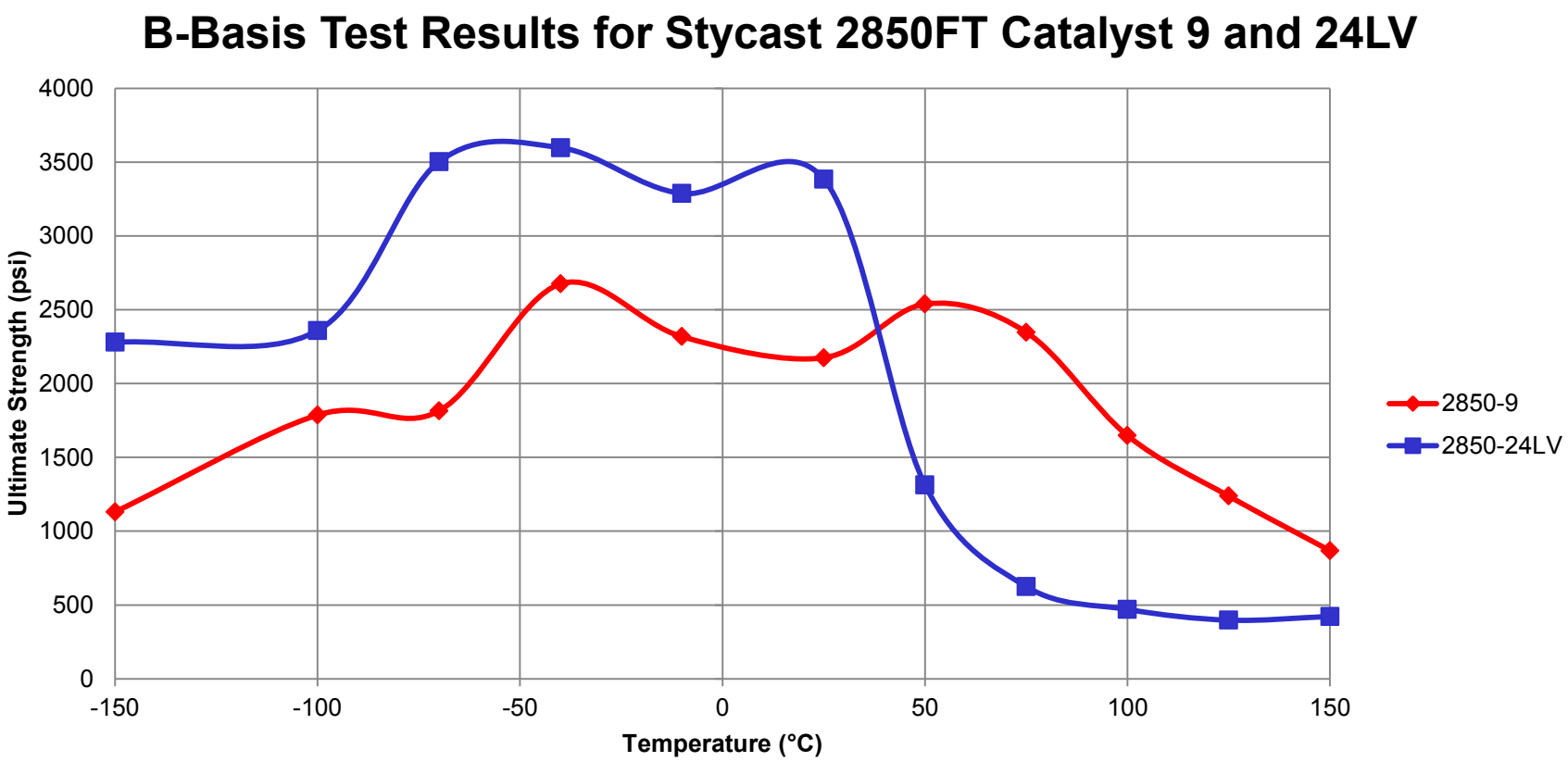
Results- Graph of B-Basis Allowables

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



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



Results- Graph of B-Basis Allowables for EA9309.3, EA9361, and EC 2216

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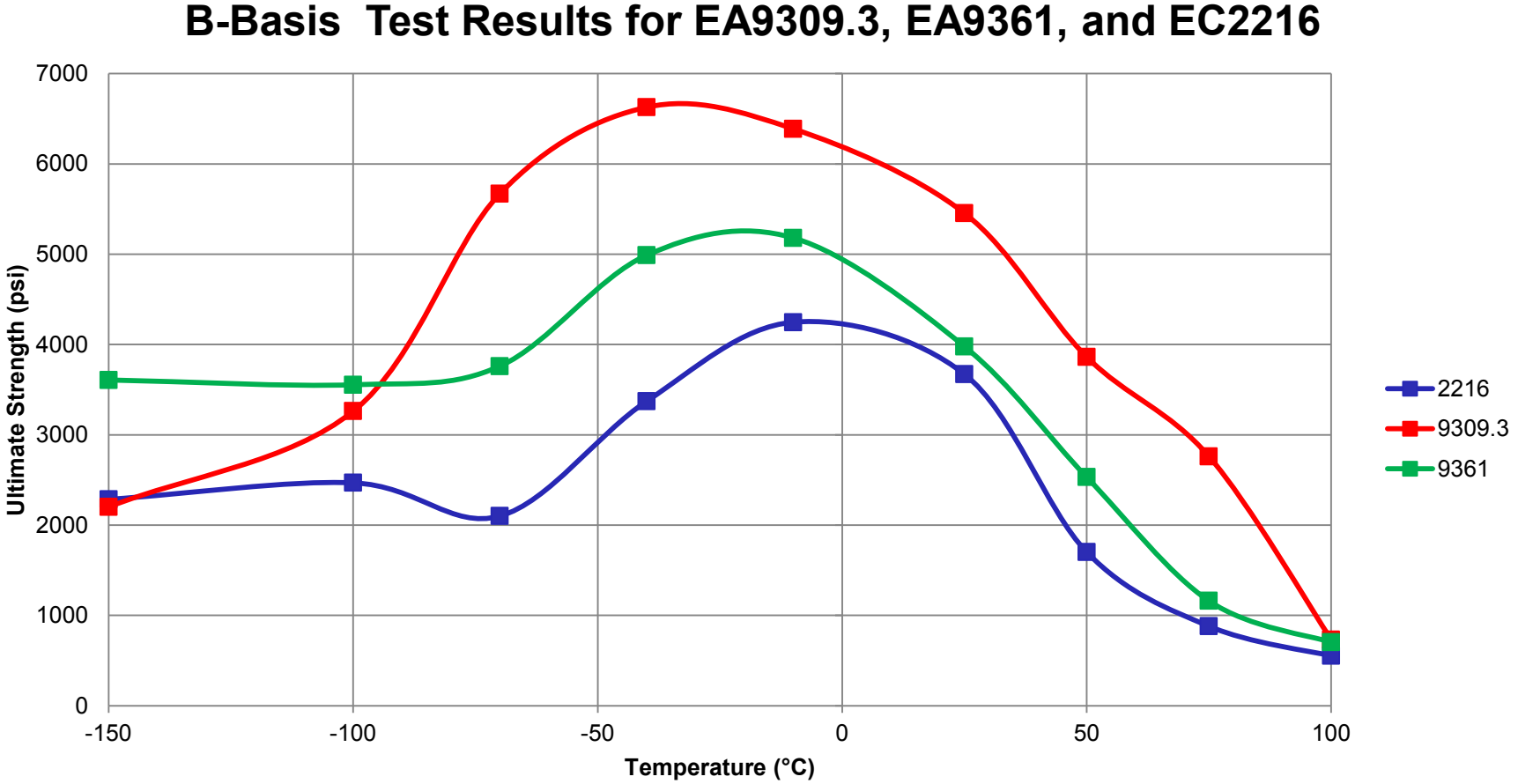
- Data complied using Stat 17 for B-basis allowables in English units



Results- Graph of B-Basis Allowables for EA9360 and EA9394

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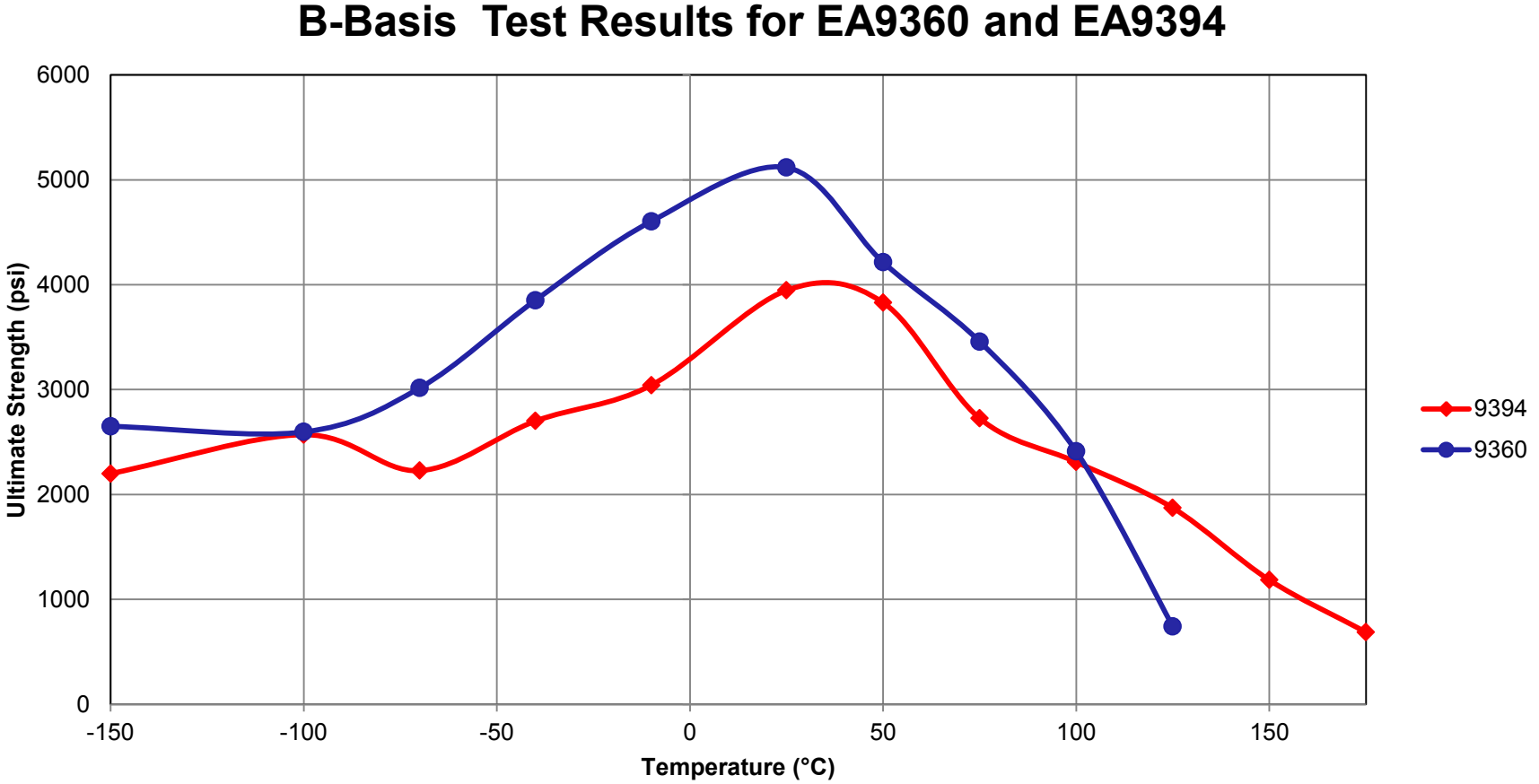
- Data complied using Stat 17 for B-basis allowables in English units.



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)

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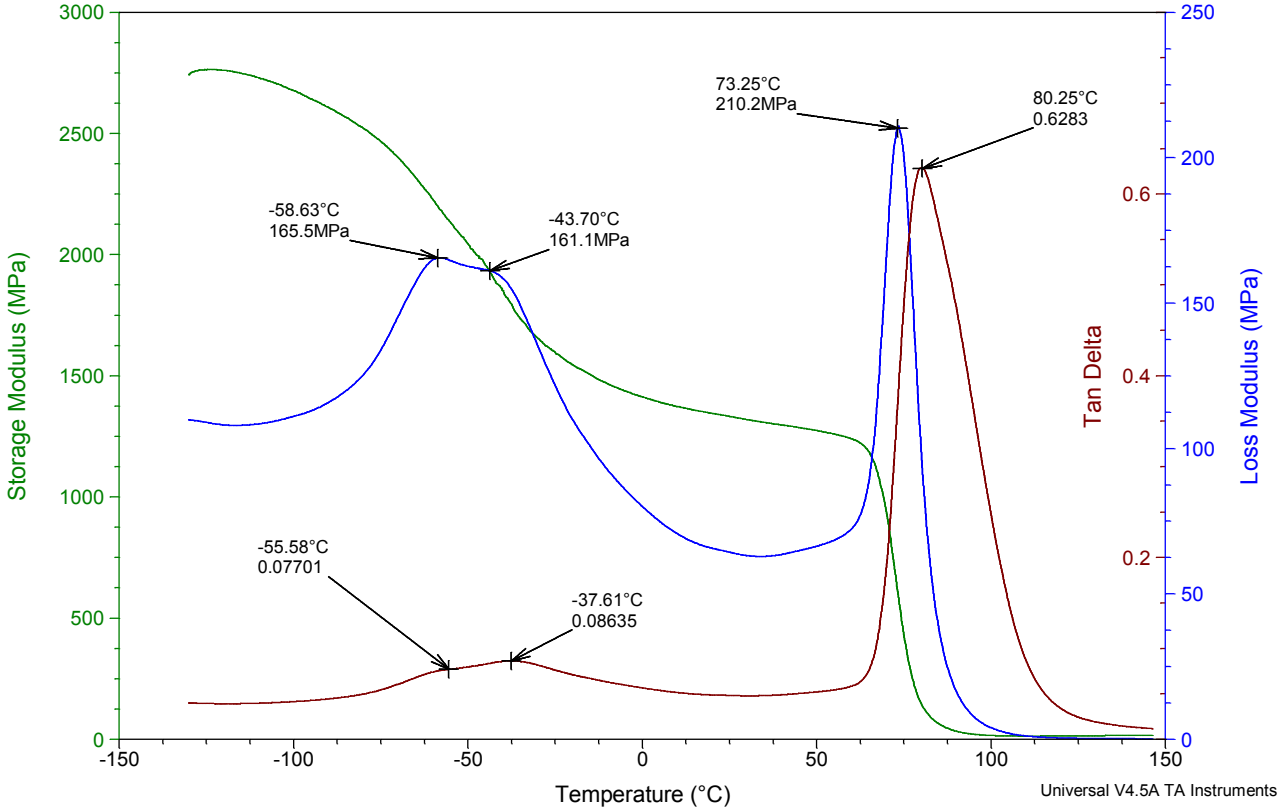
Sample: Ojeda 9309-3 purple color
Size: 17.5000 x 12.7000 x 3.0400 mm
Method: Temperature Ramp
Comment: Ojeda sample 9309-3, purple color, adhesive

DMA

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

Sample	1st Tg (ºC)	2nd Tg (ºC)	3rd Tg (ºC)	1st Loss Peak (ºC)	2nd Loss Peak (ºC)	3rd Loss peak (ºC)
9394	-33.6	106.1	NA*	-41.6	101.3	NA*
2216	-57.1	55.3	NA*	-60.2	39.1	NA*
2850 Cat 24LV	-41	74.	NA*	-50.7	66.9	NA*
2850 Cat 9	-39.4	97	NA*	-46.8	87.6	NA*
9309-3	-55.6	-37.0	80.2	-58.6	-43.7	73.2
9360	-62.3	-27.3	101.9	-63.8	-34.0	89.7
9360	-56.2	58.0	NA*	-59.5	40.7	NA*

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?

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