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Development of Dynamic Mechanical Analyzer (DMA) Calibration and Testing Procedures

2012 Technical Review

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Development of Dynamic Mechanical Analyzer (DMA) Calibration and Testing Procedures

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Development of Dynamic Mechanical Analyzer (DMA) Calibration and Testing Procedures

- Motivation and Key Issues
  - Tg by DMA is critical for characterizing polymeric materials
    - Material service temperature determination
    - Quality control tool to validate curing process
  - Lack of well defined calibration and testing procedures
    - ASTM D7028 and SACMA SRM 18R refer to the equipment manufacturer's recommendations in many cases.
      - Thermocouple location
      - Temperature calibration standards
    - Leads to poor lab-to-lab and equipment-to-equipment reproducibility
      - Round robin testing of ASTM D7028 (report number D30-1004 dated December 15th, 2007) provides a poor reproducibility coefficient of variation of 4.72%.
      - Significant reproducibility issues with several material qualification programs for FAA aircraft certification.
  - Accurate and repeatable Tg determination is critical to safety wherever polymer matrix composites are utilized.

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Development of Dynamic Mechanical Analyzer (DMA) Calibration and Testing Procedures

- Objective
  - Reduce the lab-to-lab and equipment-to-equipment variability of Tg measurements by developing universal guidelines for temperature calibration and testing procedures for DMA equipment to enhance pre-existing testing standards.
  - Improve industry applications and in-flight safety
    - Material service temperature definition
    - Quality control

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Development of Dynamic Mechanical Analyzer (DMA) Calibration and Testing Procedures

- Approach
  - Research areas that potentially influence Tg determination and repeatability.
    - Thermocouple location
    - Specimen dimensions
    - Temperature calibration
      - Calibration materials
      - Application of calibration materials
  - Develop universal guidelines for temperature calibration and testing procedures for DMA equipment.
  - Evaluate developed guidelines for lab-to-lab and equipment-to-equipment reproducibility through round robin testing.
  - Further evaluate developed guidelines by comparing to DSC and TMA test results.

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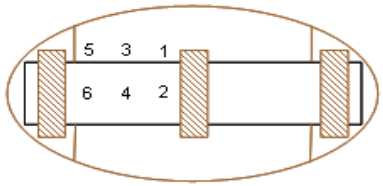
Thermocouple Location

- Thermocouple (TC) Location Evaluation
  - ASTM D7028 section 11.9 remarks, "*Follow the manufacturer's manual or recommendations to position the thermocouple. Typically the thermocouple should be as close to the sample as possible*."
  - Manufacturers recommend that the thermocouple proximity should be close to the sample, as well, but do not provide any specifics.
  - The fixture acts has a heat sink, affecting heat transfer to the sample.
    - Evaluate TC location, respective of location to fixture
    - Evaluate TC location, respective of location to sample

Thermocouple Location – Test Configurations

TC Location	Test Method	Specimens per Material (Number of Materials)		Material ID
		50mm 3-pt Bend Fixture	35mm Dual Cantilever Fixture	
1	ASTM D7028 & Figure 1	1 x (4)	1 x (4)	180F-Tg-EPXY 350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI
2		1 x (4)	1 x (4)	180F-Tg-EPXY 350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI
3		1 x (4)	1 x (4)	180F-Tg-EPXY 350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI
4		1 x (4)	1 x (4)	180F-Tg-EPXY 350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI
5		1 x (4)	1 x (4)	180F-Tg-EPXY 350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI
6		1 x (4)	1 x (4)	180F-Tg-EPXY 350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI

Top



Side

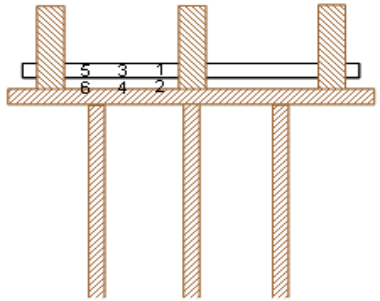


Figure 1

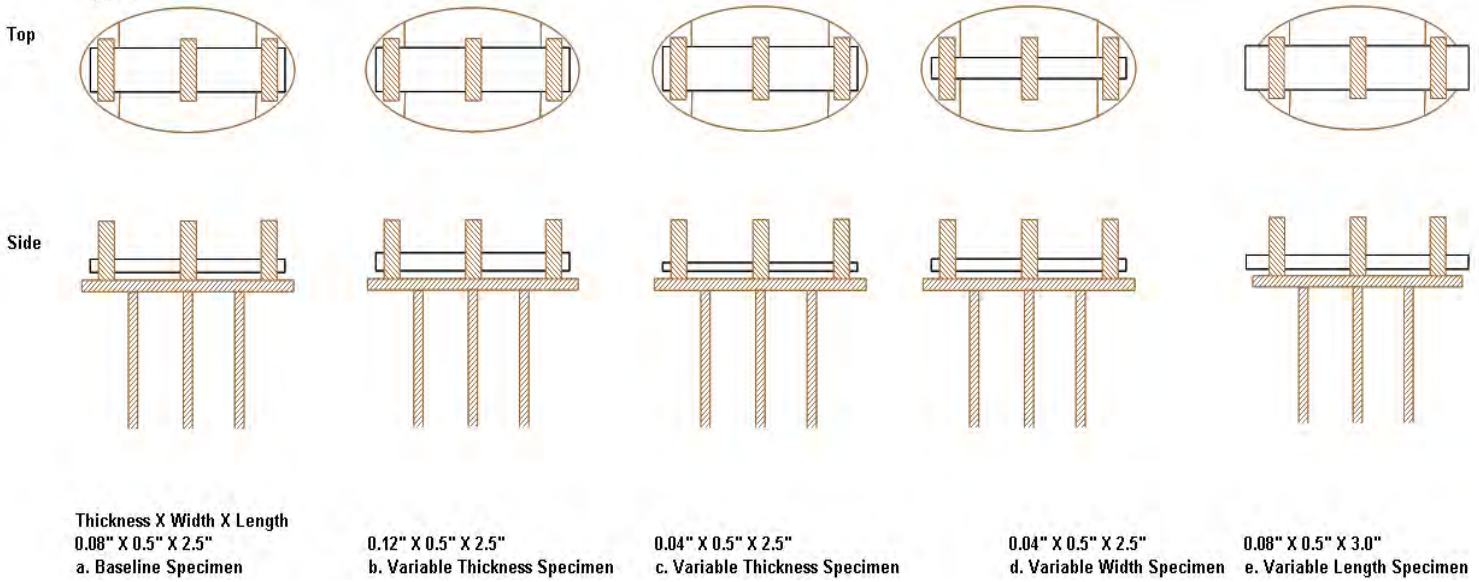
Specimen Dimensions

- Specimen Dimensions Evaluation
  - Determine if specimen dimensions affect Tg
    - Evaluate changes in thickness
    - Evaluate changes in width
    - Evaluate changes in length
  - Possible effects include
    - Thermal lag
    - Specimen stiffness

Specimen Dimensions – Test Configurations

Property	Test Method	Support Dimensions			Specimens per Material (Number of Materials)		Material ID
		Thickness (in.)	Width (in.)	Length (in.)	50mm 3-pt Bend Fixture	35mm Dual Cantilever Fixture	
Glass Transition Temperature (Tg), Dry, by DMA	ASTM D7028 & Figure 2	0.08	0.5	2.5	1 x (1)	1 x (1)	350F-Tg-EPXY
		0.12	0.5	2.5	1 x (1)	1 x (1)	350F-Tg-EPXY
		0.04	0.5	2.5	1 x (1)	1 x (1)	350F-Tg-EPXY
		0.08	0.25	2.5	1 x (1)	1 x (1)	350F-Tg-EPXY
		0.08	0.5	2.25	1 x (1)	1 x (1)	350F-Tg-EPXY

Figure 2



Thickness X Width X Length

0.08" X 0.5" X 2.5"
a. Baseline Specimen

0.12" X 0.5" X 2.5"
b. Variable Thickness Specimen

0.04" X 0.5" X 2.5"
c. Variable Thickness Specimen

0.04" X 0.5" X 2.5"
d. Variable Width Specimen

0.08" X 0.5" X 3.0"
e. Variable Length Specimen

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Temperature Calibration

- Temperature Calibration Evaluation
  - ASTM D7028, section 9.1 states, "*The DMA equipment shall be calibrated in accordance with Test Method E 1867 for temperature signals and in accordance with the equipment manufacturer's recommendation for the storage modulus*."
  - ASTM E1867 lists NIST traceable temperature calibration standards
    - Indium, Tm = 156.6 °C
    - Tin, Tm = 231.9 °C
    - Lead, Tm = 327.5 °C
    - Zinc, Tm = 419.5 °C
  - ASTM E1867 does not address the need for a rigid span support to hold the calibration standard.
    - Evaluate span support dimensions/configuration
    - Evaluate span support material

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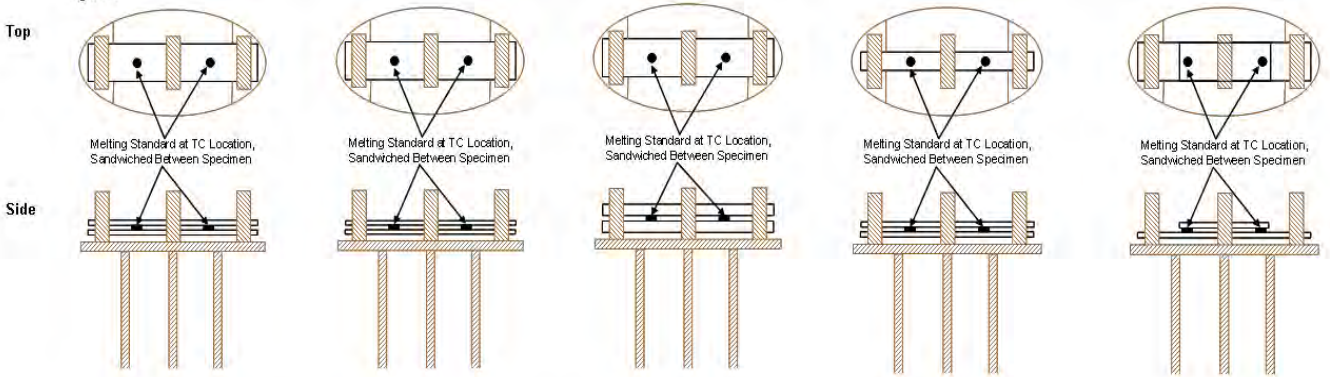
Temperature Calibration – Support Dimensions & Configurations

Property	Test Method	Support Dimensions			Specimens per Material (Number of Materials)		Material ID
		Thickness (in.)	Width (in.)	Length (in.)	50mm 3-pt Bend Fixture	35mm Dual Cantilever Fixture	
Indium Melting Temperature	ASTM E1867 & Figure 3	0.04	0.5	Bottom, 2.5 Top, 2.5	1 x (1)	1 x (1)	500F-Tg-BMI
		0.02	0.5	Bottom, 2.5 Top, 2.5	1 x (1)	1 x (1)	500F-Tg-BMI
		0.06	0.5	Bottom, 2.5 Top, 2.5	1 x (1)	1 x (1)	500F-Tg-BMI
		0.04	0.25	Bottom, 2.5 Top, 2.5	1 x (1)	1 x (1)	500F-Tg-BMI
		0.04	0.5	Bottom, 2.5 Top, 1.5	1 x (1)	1 x (1)	500F-Tg-BMI
Tin Melting Temperature		0.04	0.5	Bottom, 2.5 Top, 2.5	1 x (1)	1 x (1)	500F-Tg-BMI
		0.02	0.5	Bottom, 2.5 Top, 2.5	1 x (1)	1 x (1)	500F-Tg-BMI
		0.06	0.5	Bottom, 2.5 Top, 2.5	1 x (1)	1 x (1)	500F-Tg-BMI
		0.04	0.25	Bottom, 2.5 Top, 2.5	1 x (1)	1 x (1)	500F-Tg-BMI
		0.04	0.5	Bottom, 2.5 Top, 1.5	1 x (1)	1 x (1)	500F-Tg-BMI

Figure 3



Bottom Thickness,Top Thickness X Bottom Width,Top Width X Bottom Length,Top Length

0.04",0.04" X 0.5",0.5" X 2.5",2.5" a. Baseline Specimen

0.02",0.02" X 0.5",0.5" X 2.5",2.5" b. Variable Thickness Specimen

0.06",0.06" X 0.5",0.5" X 2.5",2.5" c. Variable Thickness Specimen

0.04",0.04" X 0.25",0.25" X 2.5",2.5" d. Variable Width Specimen

0.04",0.04" X 0.5",0.5" X 2.5",1.5" e. Variable Length Specimen

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Temperature Calibration – Support Material

Polymer Matrix Material for Comparison to Evaluation of Support Materials

Property	Test Method	Specimens per Material (Number of Materials)		Material ID
		50mm 3-pt Bend Fixture	35mm Dual Cantilever Fixture	
Indium Melting Temperature	ASTM E1867	3 x (3)	3 x (3)	350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI
Tin Melting Temperature		3 x (2)	3 x (2)	500F-Tg-BMI 600F-Tg-PI
Specific Heat Capacity, Thermal Conductivity, and Thermal Diffusivity	ASTM E1461	1 x (4)		180F-Tg-EPXY 350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI

Support Material Evaluation

Property	Test Method	Specimens per Material (Number of Materials)		Material ID
		50mm 3-pt Bend Fixture	35mm Dual Cantilever Fixture	
Indium Melting Temperature	ASTM E1867	3 x (4)	3 x (4)	Macor Alumina Silicate Silica Glass Steel
Tin Melting Temperature		3 x (4)	3 x (4)	Macor Alumina Silicate Silica Glass Steel
Specific Heat Capacity, Thermal Conductivity, and Thermal Diffusivity	ASTM E1461	1 x (4)		Macor Alumina Silicate Silica Glass Steel

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Development of Dynamic Mechanical Analyzer (DMA) Calibration and Testing Procedures

- Development of Calibration and Testing Procedures
  - Utilize data from the DMA evaluations
    - TC location
    - Specimen dimensions
    - Span support dimensions/configuration
    - Span support material
  - Distribute new calibration and testing procedures and materials to labs for round robin testing for evaluation of procedures.
      - Low-to-high Tg materials will be evaluated
      - Specimens will be tested after dry and wet environmental conditioning

Round Robin Testing

Property	Test Method	Specimens per Material (Number of Materials)										Material ID
		Laboratory, 50mm 3-pt Bend Fixture					Laboratory, 35mm Dual Cantilever Fixture					
		Lab 1	Lab 2	Lab 3	Lab 4	NIAR	Lab 1	Lab 2	Lab 3	Lab 4	NIAR	
DMA Temperature Calibration	ASTM E1867	1 x (2)	1 x (2)	1 x (2)	1 x (2)	1 x (2)	1 x (2)	1 x (2)	1 x (2)	1 x (2)	1 x (2)	Indium Zinc
DMA Temperature Verification		1 x (2)	1 x (2)	1 x (2)	1 x (2)	1 x (2)	1 x (2)	1 x (2)	1 x (2)	1 x (2)	1 x (2)	Indium Zinc
Glass Transition Temperature (Tg), Dry, by DMA	ASTM D7028	3 x (5)	3 x (5)	3 x (5)	3 x (5)	3 x (5)	3 x (5)	3 x (5)	3 x (5)	3 x (5)	3 x (5)	180F-Tg-EPXY 350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI Polycarbonate
Glass Transition Temperature (Tg), Wet, by DMA		3 x (4)	3 x (4)	3 x (4)	3 x (4)	3 x (4)	3 x (4)	3 x (4)	3 x (4)	3 x (4)	3 x (4)	180F-Tg-EPXY 350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI

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Development of Dynamic Mechanical Analyzer (DMA) Calibration and Testing Procedures

- Viability of New Calibration and Testing Procedures
  - Data from the round robin testing will be analyzed to determine adequacy of the new procedures.
  - Additional thermal analysis techniques will be used on a small scale to further characterize the capabilities of these procedures.
    - Differential Scanning Calorimetry (DSC) by ASTM D3418
    - Thermo-Mechanical Analysis (TMA) by expansion method ASTM E1545 (procedure A)
    - Thermo-Mechanical Analysis (TMA) by flexure method ASTM E1545 (modified procedure B)

Comparison to DSC & TMA Techniques

Property	Test Method	Specimens per Material (Number of Materials)	Material ID
Glass Transition Temperature (Tg), Dry, by DSC	ASTM D3418	3 x (5)	180F-Tg-EPXY 350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI Polycarbonate
Glass Transition Temperature (Tg), Dry, by TMA Expansion	ASTM E1545, Procedure A	3 x (5)	180F-Tg-EPXY 350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI Polycarbonate
Glass Transition Temperature (Tg), Dry, by TMA Flex	ASTM E1545, Modified Procedure B	3 x (5)	180F-Tg-EPXY 350F-Tg-EPXY 500F-Tg-BMI 600F-Tg-PI Polycarbonate

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Looking Forward

- Benefit to Aviation
  - Improvement in industry applications and in-flight safety
    - Material service temperature definition
    - Quality control
- Future needs
  - Expand research to look at additional factors that influence Tg determination by DMA

End of Presentation.

Thank you.

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Backup Slide - 1

Material	Transition Temperature[A] °C	Transition Temperature[A] K	Reference
Cyclopentane (solid-solid)	-151.16	121.99	X1.1
Cyclopentane (solid-solid)	-135.06	138.09	X1.1
n-Heptane	-90.56	182.65	X1.2
Cyclohexane	-87.06	186.09	X1.3
n-Octane	-56.76	216.39	X1.1
n-Decane	-26.66	246.49	X1.1
n-Dodecane	-9.65	263.5	X1.1
Water	0.01	273.16	X1.4
Cyclohexane	6.54	279.69	X1.3
Indium	156.5985	495.7485	X1.4
Tin	231.928	505.078	X1.4
Lead	327.462	600.612	X1.5
Zinc[B]	419.527	692.677	X1.4

[A] The values in this table were determined under special, highly accurate test conditions that are not attainable or applicable to this test method. The actual precision of this test method is given in Section 13.

[B] Amalgamates with aluminum. Do not heat above 430°C.

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Backup Slide - 2

		RT	RT	RT	RT
Material ID	Material Description	Thermal Conductivity (W/m*C)	Specific Heat Capacity (J/g*C)	*Density (g/cm^3)	Calculated Thermal Diffusivity (cm^2/s)
Carbon Fiber / BMI	CF-BMI-PW	0.65	0.87	1.5	**0.0050**
Carbon Fiber / Epoxy	CF-EPX-8HS	0.82	0.80	1.45	**0.0071**
Carbon Fiber / Epoxy	CF-EPX-PW	0.66	0.83	1.45	**0.0055**
Carbon Fiber / Epoxy	CF-EPY-PW		0.86		**#DIV/0!**
Glass Fiber / Epoxy	GF-EPX-7781	0.46	0.86	1.8	**0.0029**

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Backup Slide - 3

Material ID	Material Description	Thermal Conductivity (W/m*C)	Specific Heat Capacity (J/g*C)	Density (g/cm^3)	Calculated Thermal Diffusivity (cm^2/s)	Difference from Desired Diffusivity (cm^2/s)	
Z	Glass-Mica Ceramic, McMaster #8489K241 & #8489K241	1.46	0.79	2.52	0.0073	0.0022	
Z	Zerodur Glass Ceramic, CiDRA Precision Services	1.46	0.82	2.53	0.0070	0.0019	
Z	ULE Glass Ceramic, CiDRA Precision Services	1.31	0.77	2.21	0.0077	0.0026	
X	Rescor 902 Alumina Silicate Ceramic, Cotronics Corp.	1.08	0.84	1.92	0.0067	0.0016	Specific Heat was not found, but assumed to be similar to other alumina silicate ceramics
X	Alumina Silicate Ceramic L911A (Lava), Professional Plastics	1.98	0.84	2.3	0.0102	0.0051	
X	M-120F Alumina Silicate Ceramic, Morgan Technical Ceramics	1.30	0.84	2.3	0.0067	0.0016	Specific Heat was not found, but assumed to be similar to other alumina silicate ceramics
Y	Soda Lime Glass, CiDRA Precision Services	0.94	0.88	2.44	0.0044	0.0008	
Y	Fused Silica Glass, CiDRA Precision Services	1.38	0.75	2.2	0.0084	0.0032	
Y	Vycor Glass, CiDRA Precision Services	1.40	0.75	2.18	0.0086	0.0034	
Y	Fused Quartz, CiDRA Precision Services	1.40	0.67	2.21	0.0095	0.0043	
Steel	Stainless Steel (Type 304), McMaster #89815K33 & #89815K37	15.20	0.50	8.03	0.0379	0.0327	

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