High Temple Workshop XXIII
Jacksonville, FL
10-13 February, 2003

Mechanical Testing of PMCs under Simulated Rapid Heat-Up Propulsion Environments (II. In-Plane Compressive Behavior)

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ABSTRACT

Carbon fiber thermoset polymer matrix composites (PMC) with high temperature polyimide based in-situ polymerized monomer reactant (PMR) resin has been used for some time in applications which can see temperatures up to 550°F. Currently, graphite fiber PMR based composites are used in several aircraft engine components including the outer bypass duct for the GE F-404, exit flaps for the P&W F-100-229, and the core cowl for the GE/Snecma CF6-80A3. Newer formulations, including PMR-II-50 are being investigated as potential weight reduction replacements of various metallic components in next generation high performance propulsion rocket engines that can see temperatures which exceed 550°F. Extensive FEM thermal modeling indicates that these components are exposed to rapid heat-up rates (up to ~200°F/sec) and to a maximum temperature of around 600°F. Even though the predicted maximum part temperatures were within the capability of PMR-II-50, the rapid heat-up causes significant through-thickness thermal gradients in the composite part and even more unstable states when combined with moisture. Designing composite parts for such extreme service environments will require accurate measurement of intrinsic and transient mechanical properties and the hygrothermal performance of these materials under more realistic use conditions.

The mechanical properties of polymers degrade when exposed to elevated temperatures even in the absence of gaseous oxygen. Accurate mechanical characterization of the material is necessary in order to reduce system weight while providing sufficient factors of safety. Historically, the testing of PMCs at elevated temperatures has been plagued by the antagonism between two factors. First, moisture has been shown to profoundly affect the mechanical response of these materials at temperatures above their glass transition temperature while concurrently lowering the

material's Tg. Moisture phenomena is due to one or a combination of three effects, i.e., plastization of polymeric material by water, the internal pressure generated by the volatilization of water at elevated temperatures, and hydrolytic chemical decomposition. However, moisture is lost from the material at increasing rates as temperature increases. Second, because PMCs are good thermal insulators, when they are externally heated at even mild rates large thermal gradients can develop within the material. At temperatures where a material property changes rapidly with temperature the presence of a large thermal gradient is unacceptable for intrinsic property characterization purposes. Therefore, long hold times are required to establish isothermal conditions. However, in the service environments high-heating-rates, high temperatures, high-loading rates are simultaneous present along with residual moisture. In order to capture the effects of moisture on the material, holding at-temperature until isothermal conditions are reached is unacceptable particularly in materials with small physical dimensions. Thus, the effects due to moisture on the composite's mechanical characteristics, i.e., their so-called analog response, may be instructive.

One approach employed in this program was rapid heat-up (~200°F/sec.) and loading of both dry and wet in-plane compressive specimens to examine the effects of moisture on this resin dominated mechanical property of the material. Table 1 shows the matrix of test performed in this effort.

Table 1. Matrix of Tests for High Heating Rate Compression Studies

TEST	ORIENTATION	Number of Test Specimens (M40JB Cross-ply Laminate)				COMMENTS
		4HS Fabric	Uni-fabric	Stitched 4HS fabric	Stitched Uni-fabric	
COMPRESSION (4 Ply)	Warp	3	3			Wet, no-hold
		3	3			Dry, no-hold
COMPRESSION (12 Ply)	Warp	3		3	3	Wet, isothermal
						Dry, isothermal
		3		3	3	Wet, no-hold
		3				Dry, no-hold
MOISTURE CONTENT		3	3	3	3	Wet
		6	3			Dry
Total Specimens		24	12	9	9	54

Mechanical Testing of Polymer Matrix Composites under Simulated Rapid Heat-Up Propulsion Environments (II. In-Plane Compression)

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Introduction

- Rational for high heating rates
  - Air breathing hypersonic propulsion
  - Rapid heating rates increases volatilization rates
  - Volatiles generate internal pore pressure . . . . **cause damage**
  - Possible adverse oxidative reactions . . . . **destructive**
  - Plasticiztion affects of moisture at high temperature . . **loss of stiffness**

Introduction

- Rational for in-plane compression
  - Resin dominated effects
  - AP Tension, Interlaminar Shear, and In-plane CM . . . .
    Resin dominated tests
  - Across Ply Tension and Interlaminar Shear tests . . . . . .
    require thick materials
  - In-plane compression . . . .
    Proven test exists
  - Capabilities . . . .
    >200°F/sec, 20 mil materials

Introduction

- Property vs. Characteristic
  - Property . . . . Density, Strength, Permeability
  - Characteristic . . Weight, Length, Load, Mass flow rate
    - High heating rates and short hold times . . . .
      thermal gradients.
    - Mechanical properties . . . .
      change rapidly with temperature.
    - With longer hold times . . . .
      thermal gradient is reduced, moisture is lost.
    - To capture the effects of moisture . . . .
      short hold required.
    - Characteristic may be instructive.

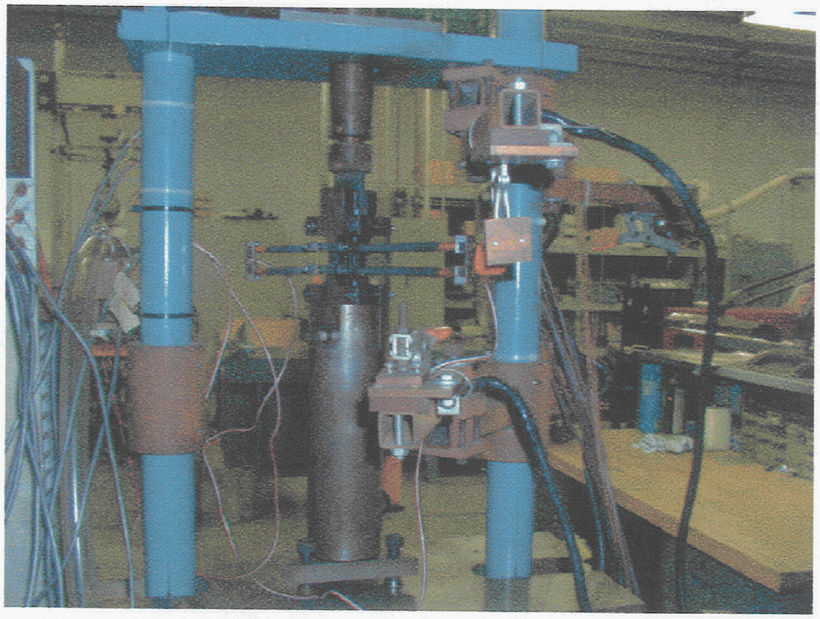
Materials and Methods

- High temperature graphite fiber polyimide composites
  - All M40JB based, balanced, and symmetrical composites
    - **4HS fabric, PMR-II-50, 12-ply, [0f,90f,90f,0f,0f,90f]1S**
      Panel dimensions: 12"x12"x0.095"
    - **Uni-fabric, PMR-II-50, 8-ply (equiv. to 4-ply woven), [0,90,90,0]1S**
      Panel dimensions: 12"x12"x0.036"
    - **Stitched - Uni-fabric, HFPE; 24-ply (eqv. to 12-ply woven), [0,90]6S**
      Panel dimensions: 10"x11"x0.11"
    - **Stitched - 4HS fabric, PMR-II-50, 12-ply, [0f,90f,90f,0f,0f,90f]1S**
      Panel dimensions: 5"x15"x0.11"
    - **4HS fabric, PMR-II-50; 4-ply, [0f,90f]1S**
      Panel dimensions: 12"x12"x0.033"

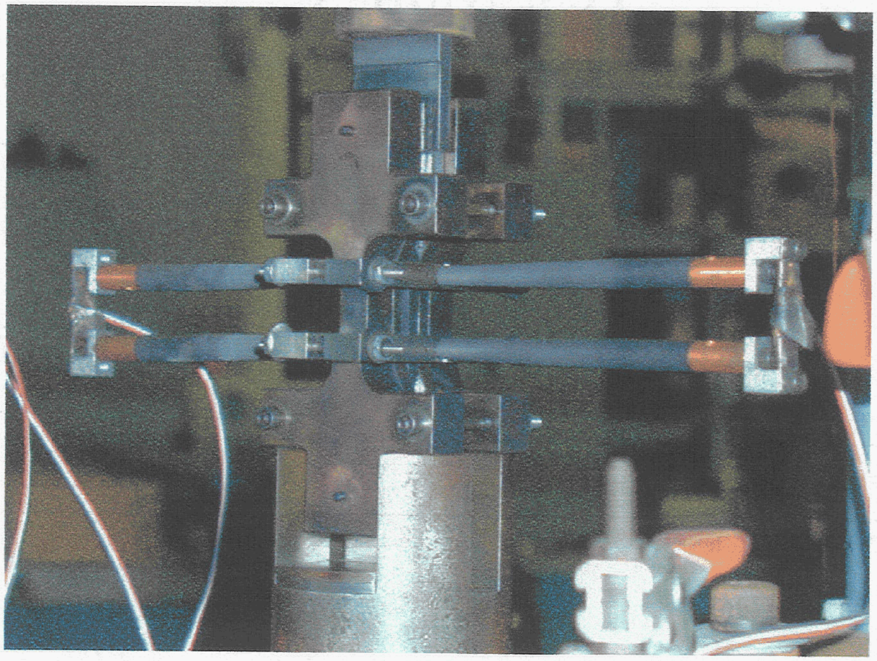
Test Matrix

TEST	TEST DIRECTION	Number of Test Specimens (M40JB Cross-ply Laminate)					COMMENTS
		4HS Fabric PMR-II-50 (12 Ply)	4HS Fabric PMR-II-50 (4 Ply)	Uni-fabric PMR-II-50 (8 Ply)	Stitched 4HS-fabric PMR-II-50 (12 Ply)	Stitched Uni-fabric HFPE (24 Ply)	
COMPRESSION	Warp	3			3	3	Wet, isothermal
							Dry, isothermal
		3	3	3	3	3	Wet, no-hold
		3	3	3			Dry, no-hold
MOISTURE CONTENT		3	3	3	3	3	Wet
		3	3	3			Dry
Total Tests		15	12	12	9	9	57

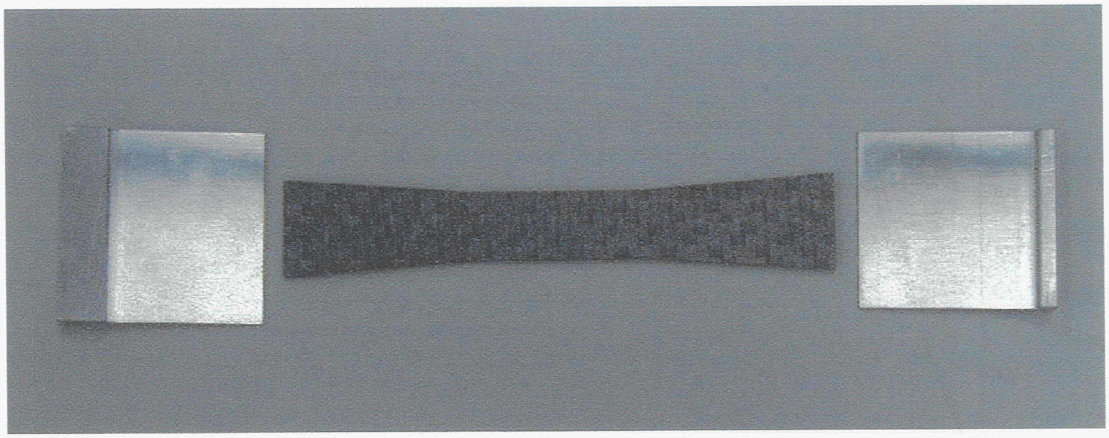
In-Plane Compression Facility



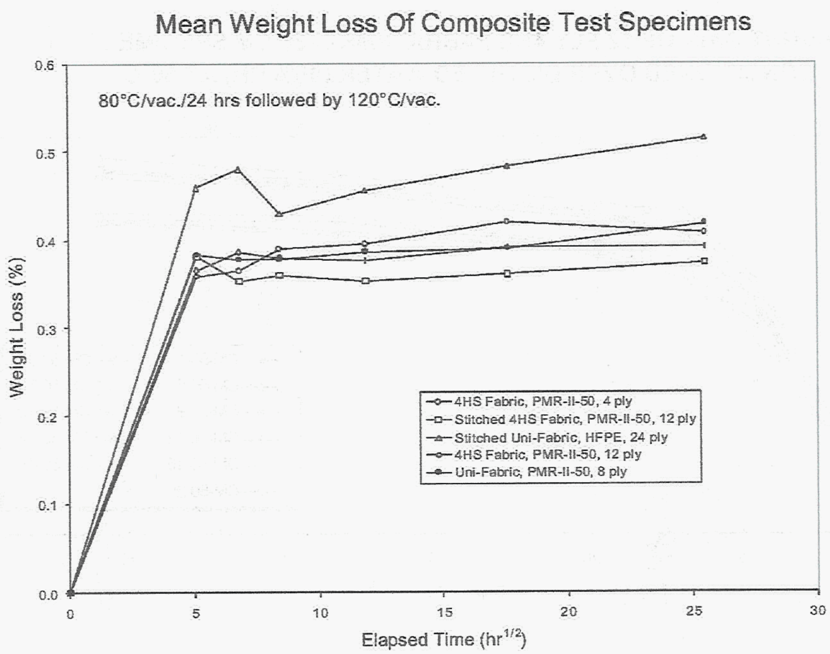
Load Train w/ Clip-On Extensometers



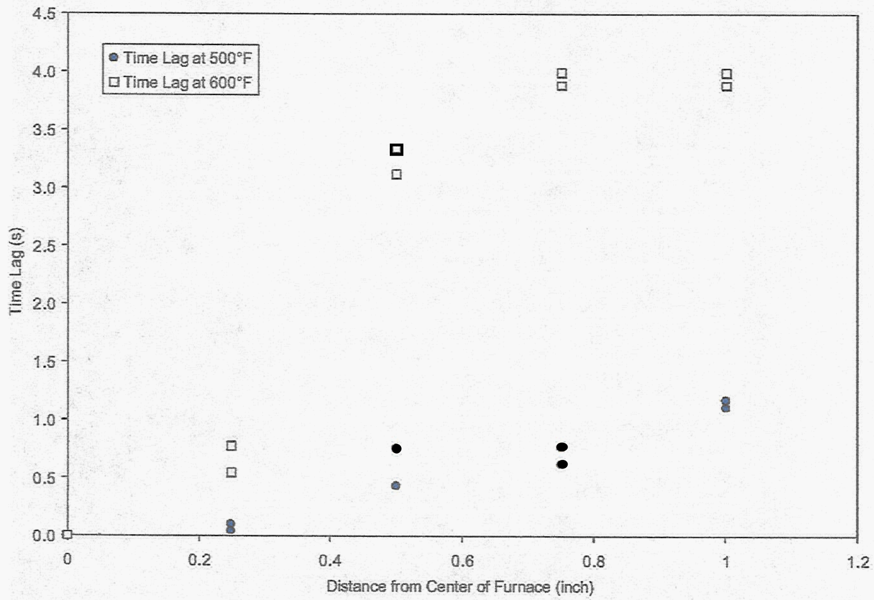
Specimen and Anvils



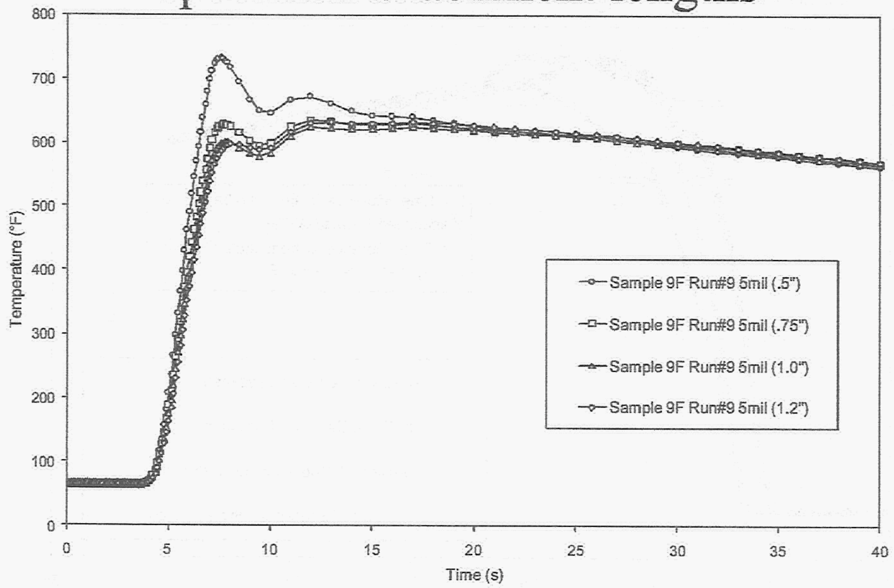
Dry Specimens



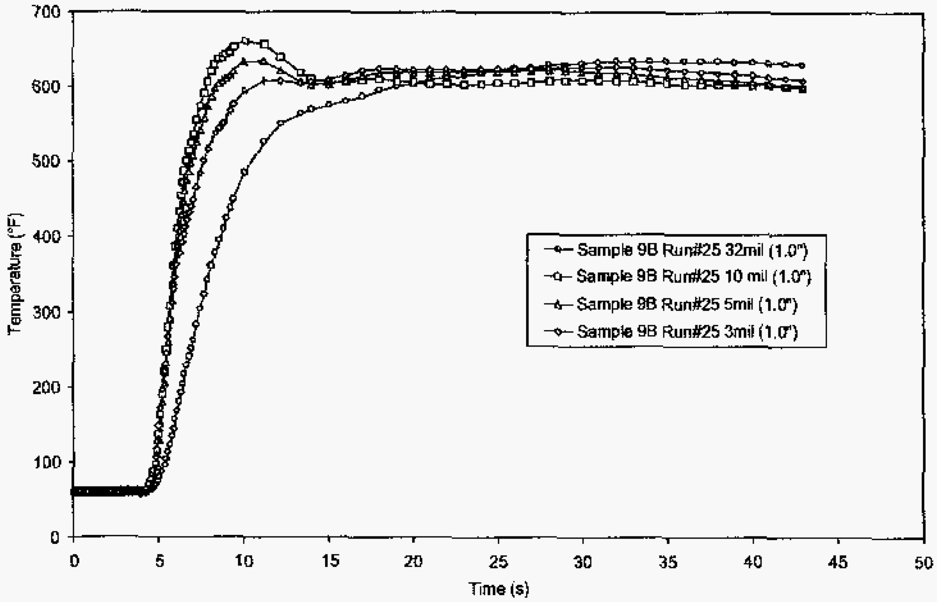
Time lag of front verses back face due to specimen position within the furnace



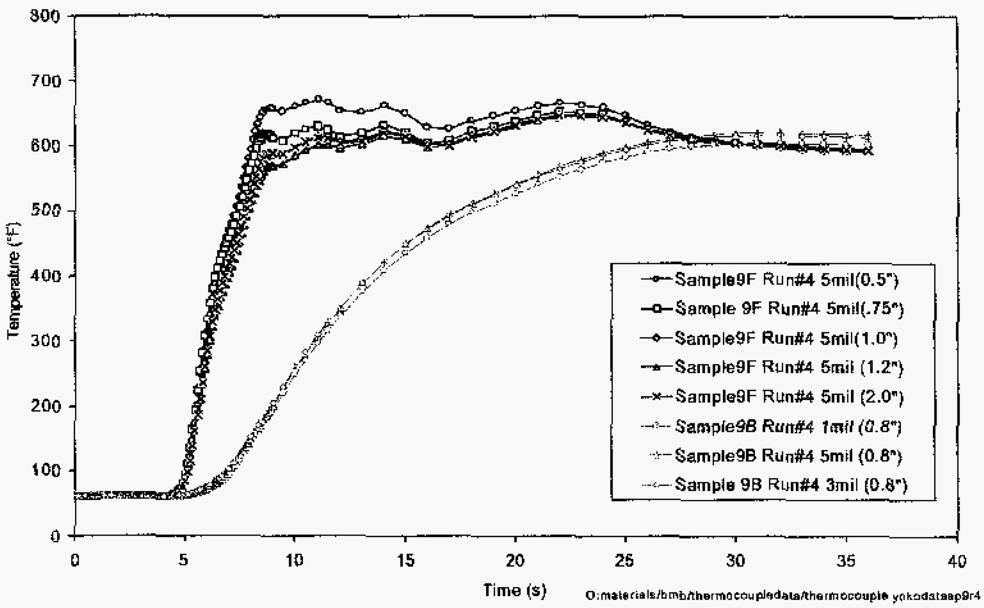
Data for thermocouples with varying specimen attachment lengths



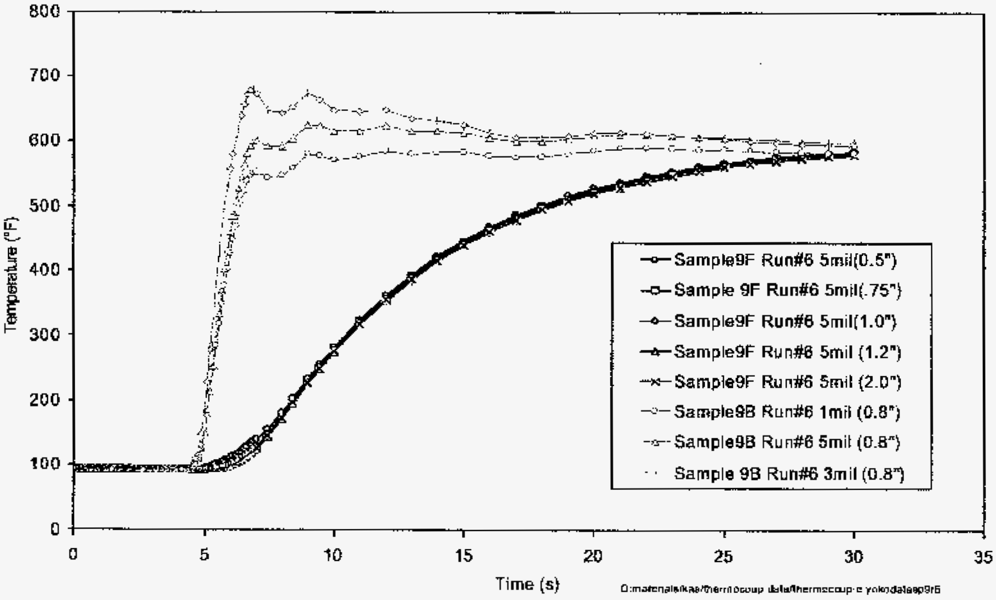
Effect of thermocouple wire diameter on recorded temperatures



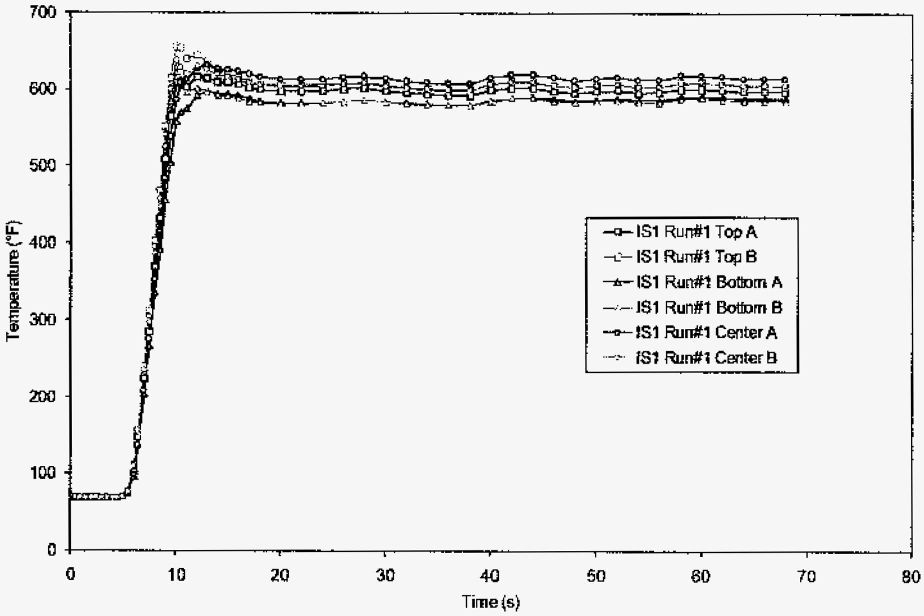
Time-temperature data for Specimen 9 with the front face covered



Time-temperature data for Specimen 9 with the back face covered



Time-temperature data as a function of location



Conclusions

- Adhesive attachment of the thermocouple to the test specimen was found to be the best way to insure accurate temperature measurements of the composite.
- A high temperature epoxy loaded with 15% carbon black gave the best transfer of radiant heat to the thermocouple while maintaining adequate adhesive properties.
- The thickness of the adhesive covering the thermocouple was found to be important when measuring the temperature of the material during heat up. However, upon reaching the test temperature variations due to adhesive thickness were second order.
- In the use environment (200°F/sec to 600°F) and under the test conditions studied (1.2 inch attachment length) thermocouple wire size did not affect the reported temperature until it exceeded 10 milli-inches in diameter.

Conclusions

- In the use environment (200°F/sec to 600°F) and under the test conditions studied (5 milli-inch wire) thermocouple wire attachment length needed to be at least 1 inch to insure accurate temperature data.
- A hold time in the neighborhood of fifteen seconds from the time the surface of the material reaches 600°F is sufficient for the center of the material to reach 600°F and to insure isothermal test conditions for the 12-ply panel material.
- The temperature of the gage can be measured ¼ inch from the failure plane and still be an accurate measure of the temperature of the material in the gage.