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EVALUATION OF HIGH TEMPERATURE POLYMERS

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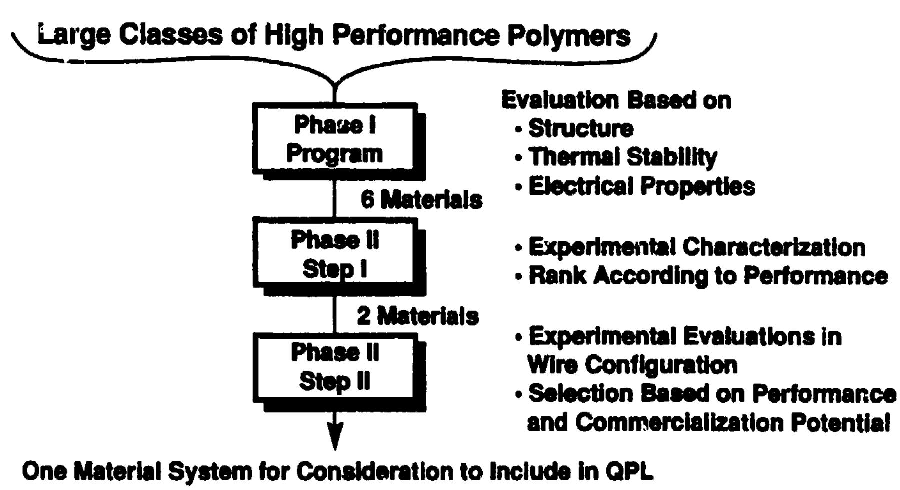
High Temperature Aerospace Insulation

- **Goal**
  - **Identify and develop arc-track resistant insulation materials that can operate reliably at 300°C**
- **Phase I SBIR program, July 1991 to January 1992**
- **Monitored by Mr. George Slenski, and Mr. Eddie White of USAF Wright Laboratory/Materials Directorate**
- **Phase II program: October 1992 to September 1995**
- **Contract monitor: Mr. John Nairus**

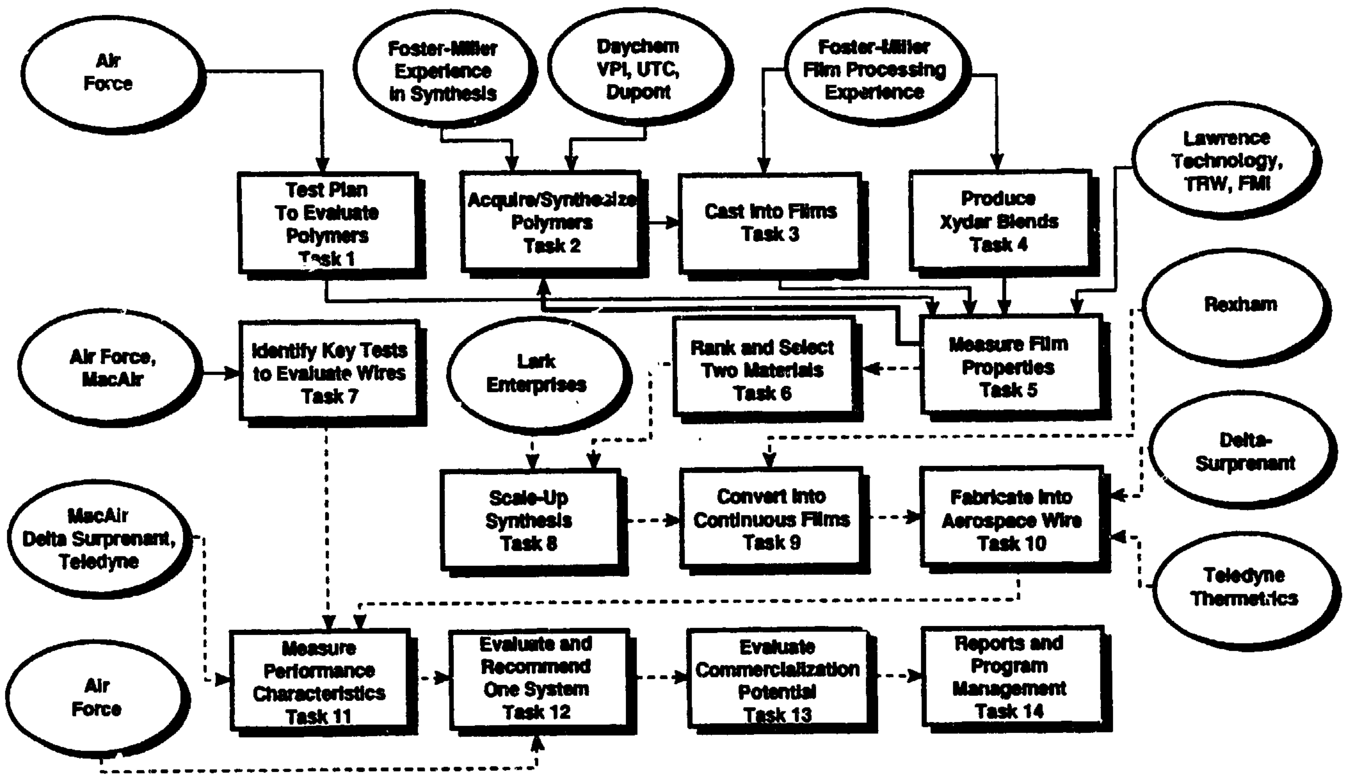
Foster-Miller Approach to Develop a 300°C Rated, Arc-Track Resistant Aerospace Insulation



Advantages and Disadvantages of Key Structural Features

Structural Features	Advantages	Disadvantages
Fluorine content	For low dielectric constant, low loss factor, high volume resistivity, uniform electrical properties over a wide range of temperatures, resistance to arc-tracking	Aliphatic fluoropolymers, such as Tefzel, have poor mechanical properties at high temperatures. To overcome this limitation, must incorporate other features
Liquid crystalline	Solvent resistance, high thermal stability, and possible improved resistance to arc-tracking	Liquid crystalline polymers are difficult to process, need to incorporate additional features, e.g., polyimide
Polyimide	High thermal stability, abrasion resistance, and good processability	Poor resistance to arc-tracking. Improved through introduction of additional features, e.g., flourinated groups, crystallinity
Aromatic	High thermal stability	Highly aromatic polymers yield conducting char upon pyrolysis
Rigidity/ stiffness	Rigidity increases thermal and mechanical capability, and reduces susceptibility to solvents	High rigid polymers can be intractable, difficult to process, and low elongation to break. Some degrees of flexibility desired
Cross-linking	X-linking significantly increases thermal stability. This process is widely used in the development of 371°C-rated composites	X-linking greatly reduces flexibility, reduces elongation to break, and embrittles
Carbon /hydrogen ratio	High carbon to hydrogen ratio increases thermal capability of polymers	High carbon to hydrogen ratio may cause the formation of conductive char and susceptibility to arc-tracking

Detailed Program Plan



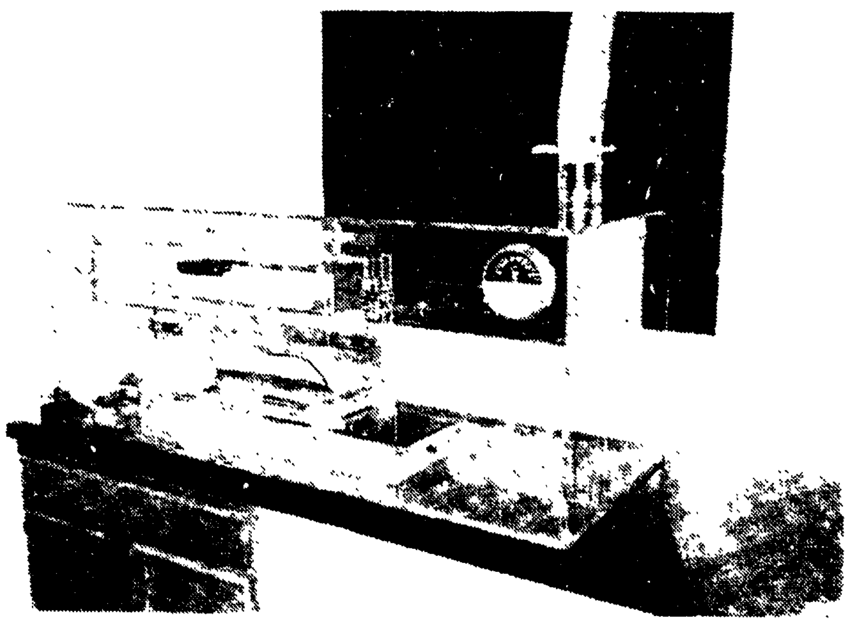
Performance Goals for Selected Materials

- Arc-track resistance
  - >180 sec using ASTM D495
  - Concern: 0.125 in. thick samples
  - Develop alternate test for thin films
- Lifetime > 15,000 hr at 300°C
- Cost comparable to Kapton
- Amenable to manufacture into aerospace wire configurations on current equipment with little or no modification

Initial Set of Materials

6F-PBO-PI	Hoechst Celanese/Foster-Miller
6F-PBO thermoplastic benzoxazole polymer	Daychem Laboratories, Dayton, OH
3F-PBO-PE	Virginia Polytechnic Institute
Low-char Polyimide	Dupont
6FDA (20%) PMDA (80%)-4BDAF	Virginia Polytechnic Institute
36FDA-PDA	United Technologies, Hartford, CT
Xydar blends	AMOCO/Foster-Miller

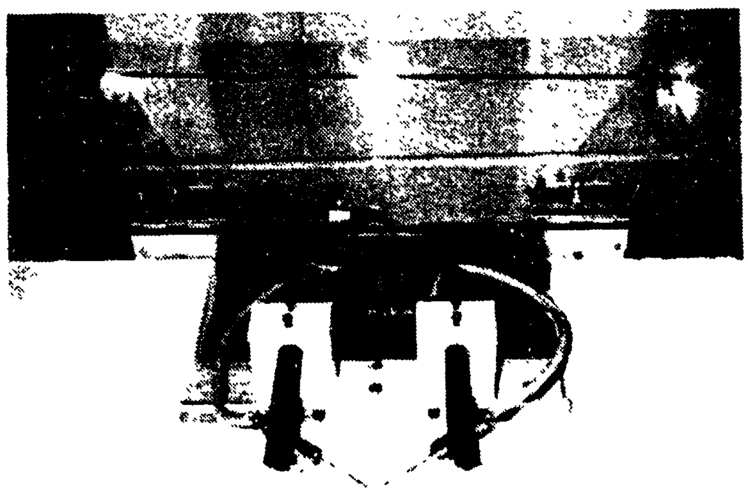
Mechanized Film Casting Setup



Verification of Procedures

- Polyamic acid of Kapton was cast into films, cured and tested to verify procedures
- Arc tracking results agree with published data
- Film cashing, thermal treatment and test procedures validated

Arc Track Resistance Tester



- Test method ASTM D495
- Apparatus

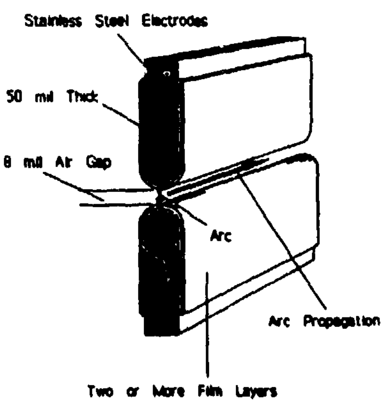
Arc Track Resistance - Critical Parameters

- Film thickness
  - Test method requires 125 mil
  - Our tests indicate 5 mil thickness is sufficient
- Pressure of electrodes on films
- Angle of electrodes
- Volatile content of films
- Heat generation during testing
- Surface cleanliness

Arc Tracking Resistance - Ribbon Test

A High-Voltage, Low-Current Test was Developed by Sigma Labs which Highly Differentiates Test Films

Arc Tracking Test Setup



- Steel electrodes, 2 in. long covered with layers of film
- Distance arc tracks in 3 min at 7.5 KV is noted
- Smaller distance that arc tracks equals better arc tracking resistance

Initial Evaluation

- Arc-track testing indicated that these materials may not meet program goals
- A new set of polymers were included in the evaluation

New Materials

6FDA-TFMB	- High Tg, high fluorine content (31%)
LaRC-CPI	- Highly crystalline materi_l
Aorimide	- Arylene ether phosphene oxide - Expected to form a non-conductive char
Siloxane Copolyimides	- "in situ" silicon dioxide formation
DuPont fluorinated polyimide	- High fluorine content (43%)
Polyimide-clay hybrids, proprietary polymer	

Candidate Arc-Track Resistant Polymer
Electrical Properties

Polymer	D-495 Arc Track Resistance (seconds)	Ribbon Arc Track Resistance (inches)	Dielectric Constant	Dissip'n Factor	Dielectric Strength (KV/mil)
Kapton HN200	120-180	0.75	3.40	0.002	6100
Upilex	180-240	-	3.50	0.001	5100
PBO	180-240	1.6	3.48	0.011	6000
6FDA/PMDA/4BDAF (30 mins, 400°C)	60-120	0.69	3.25	0.003	6740
6F-PBO-PI (30 mins, 420°C)	60-120	-	3.22	0.006	5400
6F-PBO-PE (20 mins, 400°C)	< 60	1	2.66	0.007	5570
Proprietary HT Polymer	180-300	-	2.92	0.006	7000
36FDA/m-PDA (40 mins, 400°C)	< 60	-	3.12	0.003	7200
LARC CPI	< 60	-	-	-	
TRW Partially Fluorinated Polyimides	-	-	3.1	0.001	6000
PerFluoro(Ethylene ethoxyethylene)	180-240	-	2.1	.0001	4050
3F-PBO-PE (20 mins, 400°C)	< 60	-	4.04	0.014	
DuPont Low Char Polyimide (15 mins, 400°C)	< 60	-	4.83	0.013	
DuPont Fluorinated Polyimide	< 60	2	-	-	
6FDA/TFMB	< 60	-	3.12	0.005	
AORIMIDE	< 60	-	-	-	
52% Siloxane Copolyimide	< 60	-	2.72	0.003	
Polyimide 8% Clay Hybrid	< 60	-	-	-	6200

Candidate Arc-Track Resistant Polymer
Thermomechanical Properties

Polymer	Tg (°C)	Thermogravimetric Analysis (°C @ 5% wt. loss in air)	Coefficient Of Thermal Expansion (ppm/°C)
Kapton HN200	360-410	527	20
Upilex	> 500	590	0.8 (20-100°C)
PBO	None	-	-
6FDA/PMDA/4BDAF (30 mins, 400°C)	279	439	44 (< 0°C) 64 (>100°C)
6F-PBO-PI (30 mins, 420°C)	-	-	40 (-100->200°C)
6F-PBO-PE (20 mins, 400°C)	290	501	35 (< 0°C) 55 (>100°C)
Proprietary HT Polymer	None	-	0
36FDA/m-PDA (40 mins, 400°C)	325	-	34 (-100->200°C)
LARC CPI	258	-	-
TRW Partially Fluorinated Polyimides	-	-	-
PerFluoro(Ethylene ethoxyethylene)	-	466	130 (-100 -> 200°
3F-PBO-PE (20 mins, 400°C)	299	-	-
DuPont Low Char Polyimide (15 mins, 400°C)	230	-	-
DuPont Fluorinated Polyimide	-	-	-
6FDA/TFMB	335	-	-
AORIMIDE	-	-	-
52% Siloxane Copolyimide	-	-	-
Polyimide 8% Clay Hybrid	363	478	16(-100 -> -50°C) 20(100 -> 250°C)

Candidate Arc-Track Resistant Polymer
Physical and Chemical Properties

Polymer	Peak Stress (KSI)	% Strain at Break	Modulus (KSI)
Kapton 200HN	28.9	43	320
Upilex	-	-	-
PBO	-140	2.5	7.5
6FDA/FMDA/4BDAF (30 mins, 400°C)	12.7	6.7	280
6F-PBO-PI (30 mins, 420°C)	13.3	4.6	350
6F-PBO-PE (20 mins, 400°C)	13.3	7.7	276
Proprietary HT Polymer	38.1	6	1680
36FDA/m-PDA (40 mins, 400°C)	16.4	4.4	370
LARC CPI	60	-	-
TRW Partially Fluorinated Polyimides	-	-	-
PerFluoro(Ethylene ethoxyethylene)	-	-	-
3F-PBO-PE (20 mins, 400°C)	-	-	-
DuPont Low Char Polyimide (15 mins, 400°C)	-	-	-
DuPont Fluorinated Polyimide	-	-	-
6FDA/TFMB	-	-	-
AORIMIDE	-	-	-
52% Siloxane Copolyimide	-	-	-
Polyimide 8% Clay Hybrid	-	-	-

Summary

- Most candidate materials failed due to a conductive char
- Fluorine content is not solely responsible for arc-track resistance
- Materials believed to generate non-conductive char failed
- Highly crystalline materials also failed
- Film quality appears to impact arc-track resistance
- Proprietary material, upilex and perfluoro (ethylene ethoxyethylene) are better than Kapton
- 6FDA/PMDA/4BDAF is comparable to Kapton in arc-track resistance