DEVELOPMENT OF FIRE-RESISTANT, LOW SMOKE GENERATING, THERMALLY STABLE END ITEMS FOR AIRCRAFT AND SPACECRAFT

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FIREMEN PROGRAM REVIEW

NASA-AMES RESEARCH CENTER Moffett Field, California

April 13,14, 1978

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FIREMEN PROGRAM REVIEW

DEVELOPMENT OF FIRE RESISTANT, LOW SMOKE GENERATING, THERMALLY STABLE END ITEMS FOR COMMERCIAL AIRCRAFT USING A BASIC POLYIMIDE RESIN

ABSTRACT

The technology for producing cellular materials has been available for many years and a large number of highly flexible and rigid foams have been developed. These foams have also been modified by addition of flame retardants or by reactive additives to produce materials with self-extinguishing characteristics. The many efforts to make conventional foams fire retardant have adversely increased the hazard to personnel, since, once ignited, these foams release large quantities of smoke and toxic products which are often the major cause of death.

Solar offers a new approach to the problem of flammability by the use of new materials obtained by foaming polyimide resins. This recommendation is based upon demonstrated ability of these materials to provide fire protection.

The work conducted under a recently completed program funded by NASA-LBJ Space Center, Mr. D. E. Supkis Technical Monitor, was organized to include the development of processes for producing flexible resilient open cell foam for use in aircraft seating applications. The same polyimide technology was then adapted to fabricate cellular materials for use in thermal acoustical insulation foams, floor panels and wall panels, coated glass fabrics and molded hardware. These products were produced from essentially the same polyimide precursor after modification with fillers or additives to achieve specific properties.

The characterization of the final candidate material for each of the products under study was conducted in accordance with accepted procedures. The flexible resilient foams met physical, mechanical and thermal requirements but were deficient in high cycle fatigue and elongation characteristics. The thermal acoustical polyimide foams were found to give low acoustical attenuation to the 1000 Hz, 2000 Hz and 4000 Hz, but lamination on aluminum foil overcame this deficiency. The only significant deviation in the properties of glass filled polyimide molded resins was the elongation characteristic. The phase dealing with polyimide coated glass fabrics produced materials with outstanding fire-containing properties but did not meet requirements for flexibility and abrasion resistance. Testing of the floor and wall panels is now in progress. Despite some limitations, the properties demonstrated by these materials represent a technological advancement in the art of polyimide resins which warrant additional effort. A continuation program has been undertaken to upgrade the qualities of selected materials from their present level of development, followed by fabrication of these products in larger size and quantity. The materials under study are flexible resilient foams, thermal acoustical insulation materials, wall panels and floor panels.

 Solar has developed new polyimide materials that offer new approaches to the problem of flammability and smoke. These materials will be discussed in this presentation. The presentation will be divided into two parts. The first part covers the work carried out at Solar under a program funded by NASA-LBJ Space Center and will be followed by a review of a continuation program devised to upgrade the quality of candidate materials and to scale up to full size prototype components.

2. Objectives. The objectives of the program are shown.

3. These products were to be produced from essentially the same polyimide resin precursor. The interrelations between the various tasks are shown in this slide.

4. This slide shows the program schedule. The work plan consisted of
5 phases and described the general objective of the work plan for optimizing
selecting and fabricating each of the different types of aircraft structures.

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5. Let's discuss each of the products developed, starting with the flexible resilient foams.

6. Four different foaming methods were studied and a variety of copolyimide resins synthesized for selection of final candidates.

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7. This slide shows a foam produced by microwave processing.

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8. This compares with the same resin foamed by thermal processes. The non-homogeneous structure typical of thermal heating is evident.

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9. Large samples of the candidate material were produced and evaluated for all properties in accordance with ASTM method D-1564 covering testing of flexible polyurethane foams. The results are reported in this viewgraph.

10. Floor and Wall Panels

The polyimide resin used in fabrication of floor and wall panels was essentially that used in the preparation of flexible resilient foams. Major effort of this task involved improvement of the mechanical properties through the use of a variety of methods which included use of reinforcements such as:

- Carbon Fibers
- Glass Fibers
- . Mats
- . Strands
- Honeycomb Configurations

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11. This viewgraph shows the sequence for fabrication of rigid panels from a continuous mat.

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12. This slide shows the preparation of rigid panels using graphite fibers.

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13. This viewgraph shows a configuration using a honeycomb and filling it with a polyimide foam. The technique and data developed in the study of floor panels were applied to the study of wall panels and selections made on the basis of density requirement only. Samples of floor and wall panels were submitted to Boeing for evaluation.

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14. These configurations were selected as candidate for final evaluation.

15. Thermal Acoustical Insulation

Thermal acoustical insulation materials were produced from essentially the same polyimide precursors and same processes used for fabrication of the flexible resilient foams.

16. Direct Foaming

Shows a foam produced by conventional microwave processing.

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17. Foaming on Glass Batting

Shows a polyimide foam coated and then foamed by thermal process on a glass batting.

18. Summary of Results

The results of testing are reported in this viewgraph. Thermal acoustical foams meet all requirements with the exception of acoustical properties. Note that the density of the polyimide foam is at least half that of the conventional glass batting.

19. Acoustical Attenuation, dB

This viewgraph shows the effect of thickness of the polyimide foam slabs on the acoustical attenuation. The lamination of aluminum foil on one side of the foam enhances the acoustical properties to acceptable levels.

20. Molded Shapes

These high strength components were prepared by simply compressing polyimide rigid panels to the desired density.

21. Summary of Results

The major deficiency of the material at the present stage of development is elongation at break.

22. Flexible Coated Fabrics

This phase of the program covered optimization of coating processes to obtain decorative effects and fire containing properties of fabrics. Polyimide resin compositions were found that produced flexible coatings on satin weave glass fabrics in addition to outstanding fire resistance.

23. Summary of Results

The materials produced in this phase of the program show outstanding fire-containing properties, however, were deficient in flexibility and stiffness.

24. The technology developed under this study has provided the basis for small scale pilot plant processes. These processes require additional effort to optimize the products to large scale production.

A new program has been initiated to investigate optimization of processes for fabrication of:

- . Flexible Resilient Foams
- . Wall Panels and Floor Panels
- . Thermal Acoustical Insulation

25. A program schedule detailing the various tasks is shown. This program which covers a period of 24 months is organized to proceed with investigation of all materials concurrently since there is technology transfer between the various tasks.

26. The interrelation of the various tasks is presented. As it is shown, all products will be produced from essentially the same polyimide resin precursor.

CONCLUSION

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Work on this program has been started in January 1978. The major contributions to date are:

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- . Improved Thermal Acoustical Foam Material
- Resilient Foams Possessing High Flexibility
 - . Continuous Processes for Producing Polyimide Foam Resins.

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S.O. 6-4501-7 NAS9-15050

Development of Fire-Resistant, Low Smoke Generating, Thermally Stable End Items for Aircraft and Spacecraft

for

National Aeronautical & Space Administration Lyndon B. Johnson Space Center Houston, Texas 77058

Mr. D. E. Supkis



ADR 1826-9 June 1977

OBJECTIVES

- Optimization of the properties of polyimide foams for application in five different types of aircraft cabin structures.
 - Resilient Foams
 - Thermal Acoustical Insulation
 - Floor and Wall Panels
 - Molded Structures
 - Coated Fabrics
- . Use of a single resin formulation

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. Fabrication of large size prototype samples



	♥ START OF PROGRAM 1976 1977												
REPORTING TASK	JUNE	JULY	AUG	SEPT	OCT	NOV	DEC	JAN	FEB	MARCH	APRIL	MAY	JUNE
PHASE I FLEXIBLE RESILIENT FOAMS													
Materials procurement Advanced polyimide synthesis Foaming studies Scale-up process Characterization & selection Sample preparation			.	Y	1							¥	
PHASE II LAMINATE FLOOR AND WALL PANELING						}	l						
Tooling Resin and reinforcement study Process parameters/substrates evaluation Specimens preparation and screening Fabrication of test specimens for Boeing Advanced testing Sample preparation		E			.								7
PHASE III OPTIMIZATION AND CHARACTERIZATION OF THERMAL ACOUSTICAL INSULATION MATERIALS													
Advanced polyimide synthesis Foaming studies & preliminary testing Specimen preparation & candidate selection Final characterization Sample preparation	*			*									
PHASE IV INVESTIGATION OF PROCESSES FOR HIGH STRENGTH POLYIMIDE MOLDED SHAPES													
Resin & reinforcement selections Evaluation of process hardware Compression molding, prototype Decorative effects Advance testing Sample preparation		*				[F	
PHASE V OPTIMIZATION OF FLEXIBLE POLYIMIDE COATED FABRICS					; ,)				; ;				
Evaluation of fabrics Decorative effects Specimen preparation & testing Sample preparation				*				 				T	
PHASE VI REPORTING AND COORDINATION													
Monthly progress reports Mid-term report Mid-term review Final report End of contract presentation													

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Program Schedule

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FLEXIBLE RESILIENT FOAMS

MAJOR OBJECTIVES:

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- . Improved hydrolytic resistance
- . New heating methods to achieve homogeneous cellular structure.
- . Improved fatigue resistance
- . Large scale processing

FOAMING STUDIES

Four different foaming methods were studied:

- Thermal
- Vacuum
- Dielectric
- . Microwave

Advanced Synthesis

A total of 90 copolyimide compositions evaluated.





Foaming by Thermal Processes

Property	ASTM Method	Units	Goal	Actual
Density	D-1564	Kg/m ³ lbs/ft ³	40.0 2.5	19.2 1.2
Tensile Strength	D-1564	N/m ² psi	82.7 x 10 ³ 12.0	92.4 x 10 ³ 13.4
Elongation	D-1564	8	30-50	39
Tear Resistance	D-1564	N/m lbs/inch	175.1 1.0	210.0 1.2
Identation Load Deflection 25%	D-1564	N/3.2 dm ² lb-force/50 in ²	111.2-155.6 25-35	164.0 37.0
65%		N/3.2 dm ² lb-force/50 in ²	667-1112.0 150-250	1260.0 283.0
Compression Set 50%	D-1564	% Loss	7-10	6.2
Corrosion	FTMS No. 151		None	No Evidence
Resilience Rebound Value	D-1564	8	50 min.	75
Dry Heat	D-1564	<pre>% Loss Tensile Strength</pre>	20 max.	10.3 (increase)
Humidity 73.9°C (165°F) 100% R.H.	D-1565	% Loss IDL	20 max.	7.5
Fatigue 10,000 cycles 20,000 cycles	D-1564	% Loss IDL	20 max. 20 max.	14.0 24.0
Odor	a shara saligagina	Mar and a second	None	Not detectable
Oxygen Index	D-2863	% Oxygen	40 min.	45
Smoke Density DS uncorrected	NBS	Optical density	30-50 max.	1.0
Thermostability	Thermogravimetric Analysis	Loss 204°C (400°F)	None	No loss
Toxic Product of Combustion HC1 HF SO2 H2S		10 ppm max. 10 ppm max. 10 ppm max. 10 ppm max.		None present None present None present None present

Summary of Results. Flexible Resilient Foams

FLOOR AND WALL PANELS

OBJECTIVES:

- . Fire-containing properties
- Low weight, high strength

APPROACH:

Polyimide resins modified with reinforcing fillers:

- Carbon Fibers
- Glass Fibers
- Mats
- Strands
- Honeycomb Configurations







Polyimide Foam Filled Honeycomb

CANDIDATES

Floor Panels

- . Chopped carbon mat reinforced polyimide foams
- . Glass strands reinforced polyimide foams
- . Polyimide foam filled honeycombs

Wall Panels

- . Chopped carbon mat reinforced polyimide foams
- Polyimide foam filled honeycombs

THERMAL ACOUSTICAL INSULATION

OBJECTIVES:

- . Fire resistant materials
- . Acoustical attenuation

APPROACHES:

- Direct Foaming
- Foaming on Glass Battings
- . Coating Glass Battings

CANDIDATE:

Unfilled Polyimide Foam



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Summary of Results. Thermal Acoustical Insulation

Property	ASTM Method	Units	Goal	Actual
Density	D-1564	Kg/m ³ lbs/ft ³	9.6 0.6 max.	5.6 0.35
Breaking Strength	ссс-т-191	N/m lbs/in.	175.1 1.0 min.	744.2 4.25
Wicking as received	Water immersion	cm in. precipitate	1.0 max 0.25 max None	None detectable None detectable None detectable
Wicking after oven drying 71.1°C (160°F)	Water immersion	cm in. precipitate	1.0 max. 0.25 max. None	None detectable None detectable None detectable
Flexibility		deterioration after bending on one-foot radius	None	None detectable
Corrosion (Aluminum)		Pitting	None	no pitting)
Elevated Temperature Resistance		Weight loss	15 mg max.	12 mg (water)
Oxygen Index	D-2865	<pre>% oxygen</pre>	40 min.	45
Smoke Density DS Uncorrected	NBS	Optical Density	30-50 max.	2.0
Verticle Bunsen Burner Test, 60 seconds		Flame Time seconds	10 max.	0
		Burn length cm in.	15 max. 6 max.	3.0 1.2
		Dripping		None detectable
1000°C (2014°F) Flame Test (Meker Burner) 10 minutes		Cold Face Temp. °C °F	260 500	142 288
Vibration		l Hr 30 Hz 5 cm amplitude	No damage	None detectable
Acoustical Properties		Absorption Coefficient 1000 Hz 2000 Hz 4000 Hz	0.736* 0.965* 0.916*	0.533 0.949 0.737
*Owens Corning PL 105 500	₩		L	l

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ACOUSTICAL ATTENUATION, dB

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	1000 Hz	2000 Hz	4000 Hz
Owens Corning - PF-105-500W- 3 inches	11	20	29
Polyimide Foam - 3 inches	6	9	13
Polyimide Foam - 6 inches	9	13	19
Polyimide Foam - 3 inches/0.01" Al Foil	11	17	25
Polyimide Foam - 6 inches/0.01" Al Foil	10.5	22	31.5

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MOLDED SHAPES

OBJECTIVES:

. Development of high strength polyimide foams to replace conventional plastics.

APPROACHES:

- . Compression mold polyimide compositions into high density components.
- . Contribution of reinforcements to impact strength.

CANDIDATE:

. Glass filled polyimide resins

Property	ASTM Method	Units	Goal	Actual
Specific gravity	D792	g/cc	1.0-1.5	1.23
Tensile Strength	D-638	psi n/m ²	8000-12,000 55.1 x 10 ⁶ - 82.7 x 10 ⁶	6866 47.3 x 10 ⁶
Elongation	D-638	8	4-8	1.1
Impact Strength	758-48	ft-1b/in. J/m	7-12 374-640	7.3 390
Heat Distortion Temperature (264 psi)	D-648	°C °F	148.9-176.7 300-350	Higher than: 204.4 400
Rockwell Hardness (Alpha)	D-785		R110-R130	R102
Oxygen Index, LOI	D-2863		40 minimum	60
Smoke Density D uncorrected s	NBS		30-50	1.0
TGA	-	°C °F	Stable to: 204.4 400	400 - 752

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Summary of Results -	Molded	Shapes
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Viewgraph No. 21

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FLEXIBLE COATED FABRICS

- OBJECTIVE Obtain fire hardening properties and decorative effects of weaved fabrics.
- APPROACH Evaluate and select fabrics compatible with the polyimide resins and with processing parameters.
- CANDIDATE Style 180 and 120 satin weave glass fabrics.

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				Actu	al		
Property	ASTM Method	Units	Goal	#2 3.0 mil	#3 5.0 mil		
Specific Gravity	D-792	g/cc	1.0-1.5	0.95	0.96		
Bursting Strength	D-751-68	kPa psi	275 minimum 40 minimum	3000 436	2040 296		
Abrasion Resistance	FTMS 1916		250 cycles no loose fibers	200**	250		
Blocking	FTMS 191		not higher than 3	1	1		
Flex-Crack Resistance	D-2176-69		5000 cycles	890	477		
Stiffness	FTMS 1916	cm in.	2.5 minimum 1.0 minimum	22.3 8.8	24.6 9.7		
Coating Adhesion*	D-3002-71	% coating removed	0	0	0		
Oxygen Index, LOI	D-2863		40 minimum	60	60		
Smoke Density D_ (uncorrected)	NBS		30-50	1.0	2.0		
TGA	-	°C °F	Stable to: 204.4 400	400 725			
*T-Peel test for adhesion was not possible for this type of material. 1.							

Summary of Results - Coated Fabrics

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Proposal No. 9-BC72-3-7-86P QR6-6474-1

Development of Fire-Resistant, Low Smoke Generating, Thermally Stable End Items for Commercial Aircraft and Spacecraft Using a Basic Polyimide Resin

Submitted to:

National Aeronautics and Space Administration Lyndon B. Johnson Space Center Houston, Texas 77058

Mr. D. E. Supkis

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PROGRAM SCHEDULE

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PROGRAM FLOW DIAGRAM

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