D5

N79-31171

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

by

J. Gagliani, Solar Turbines International

This presentation is divided into four parts. The first part covers experimental data pertinent to flexible resilient foams followed in order by low density wall panels, high strength floor panels and thermal accoustical insulation.

The schedule which covers each task under study is shown in Figure 1 and the interrelations between the various products and tasks are shown in Figure 2. The tasks and the objectives of the phase of the program dealing with f ble resilient foams are shown in Figure 3.

These objectives were achieved by modification of the resin compositions through advanced synthesis and by optimization of all the process parameters. Modification of the basic prepolymers was carried out by alteration of the resins with aromatic and aliphatic diamines. The corresponding terpolyimide foams obtained were then evaluated for the most critical parameters as shown in Figure 4 and Figure 5. As reported, aromatic terpolyimide foams did not produce the desired compression set properties (15% loss maximum after 24 hours at 90% compression) and were eliminated from further study.

The properties of foam derived from terpolyimides modified with aliphatic diamines approached the requirements for compression set (see Group IV) and met the fatigue requirements.

Next, an evaluation of the effect of the heterocyclic diamine component on the compression set of the foams was carried out. The data of Figure 6 show that higher ratio of the heterocyclic diamine produces foams with improved compression set properties, however when ratios higher than 0.4 were used the foams obtained were highly reticulated and not suitable for sealing applications.

The two candidates selected, specifically the 1701-1 and 1702-1 were further evaluated to study the contribution of surfactants on compression set properties. These data are shown in Figure 7 where the improved properties of the 1701-1 foams are clearly shown. At this point of the program, four polyimides precursors were selected for further evaluation in further studies.

The efforts were continued with evaluation of the foaming process parameters. The foaming process consists of simply placing the powder precursor on a suitable substrate followed by foaming in a microwave oven. The expanded mass is then heat cured at 500°-550°F to obtain resiliency and flexibility.

The foaming parameters studied were:

Power output
Powder loading
Preheat temperature
Preheat time
Foaming time
Curing temperature

Figure 8 shows that power output in the range of 2.5 to 10 kW produces foaming but higher power outputs are desirable since they cause incipient curing. The effect of powder loading on the foaming behavior of polyimide precursors is shown in Figure 9. Powder loadings higher than 2.4 Kg/m^2 are essential. The powder precursor does not have to be preheated as shown in Figure 10, however when the preheating time is extended and the temperature is maintained at 250°F improved compression set properties are obtained (Figure 11).

The foaming time in the high frequency field has also been found to be critical as shown in Figure 12 where improved compression properties are achieved by using higher power outputs and longer foaming time. The last step in the preparation of the polyimide foam involves curing the expanded mass to achieve flexibility and resiliency.

The data of Figure 13 show that higher temperature and longer curing time cause foam degradation and poor compression set properties. The data points represent an average of six determinations carried out on large size foams (1000 g of powder precursors). This concludes the work carried out in the task dealing with flexible resilient foams.

The next study involved evaluation of processes and compositions to fabricate wall panels. The tasks and objectives are shown in Figure 14. Optimization of the polyimide compositions previously developed was achieved with the development of rigid foams meeting the density requirements. This study was continued with development of new techniques to produce low density panels in a one-step microwave process as shown in Figure 15. The precursor and additives are mixed, spread over a substrate and foamed in a microwave cavity by restricting the rise. The finished rigid panel is characterized by possessing low density core and high density skins.

The same technology is now being used to produce high strength floor panels. The tasks and objectives of this task are shown in Figure 16.

A major task of this program was the development of thermal acoustical polyimide materials to replace conventional glass batting insulation. The tasks and objectives of this last study are shown in Figure 17. The studies dealing with advanced synthesis and with foaming studies carried out in the task dealing with flexible resilient foams are completely applicable to fabrication of polyimide foams for use in thermal acoustical insulation. The optimization of glass batting and foams was then initiated. Figure 18 shows the effect of polyimide foam coatings on the burnthrough resistance of PF-105-700 fiberglass batting. The coatings were applied by spray techniques using liquid polyimide precursors and foamed at 550°F. As shown, polyimide coatings improve the burnthrough resistance of the fiberglass batting at any resin loading. The burnthrough requirements were met at a loading of 0.048 Kg/m^2. The tests were made with a Meker burner and carried out until burnthrough occurred.

A second approach to the problem involved modification of the polyimide foams with additives to produce improved fire resistance. Figure 19 shows the effect

of a combination of glass microballoons and glass strands on the burnthrough resistance of polyimide foams. The filled foam did not fail after 10 minutes exposure to the Meker burner, while the unfilled foam failed in 2.5 minutes. The two candidate materials, the polyimide coated fiberglass batting and the filled polyimide foams were then tested in the NASA-JSC Fire Rig, but did not meet the minimum burnthrough requirements (5 minutes). Failure appeared to be more mechanical due to thermal cracking, than to material failure.

To reduce the thermal stresses and improve the burnthrough resistance, new crosslinked polyimide foams have been developed which are at under evaluation.

The program is continuing with the major tasks listed in Figure 20.

NAS9-15484
February 1, 1979

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

Attn: Norman R. Lamb
Mail Code: BC721 (3)

SOLAR TURBINES INTERNATIONAL
An Operating Group of International Harvester
2200 Pacific Highway, P.O. Box 80966, San Diego, California 92138

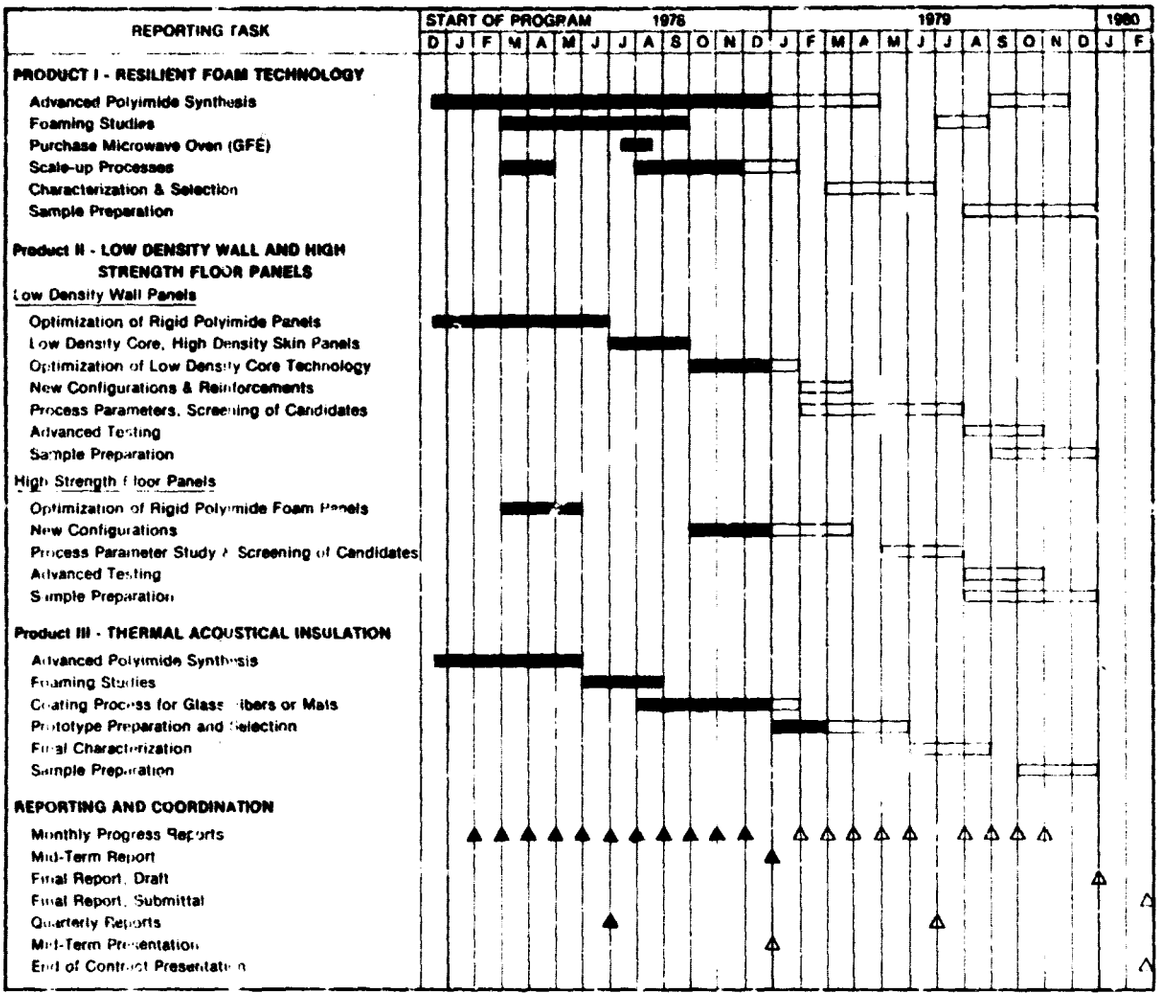


Figure 1. Program Schedule

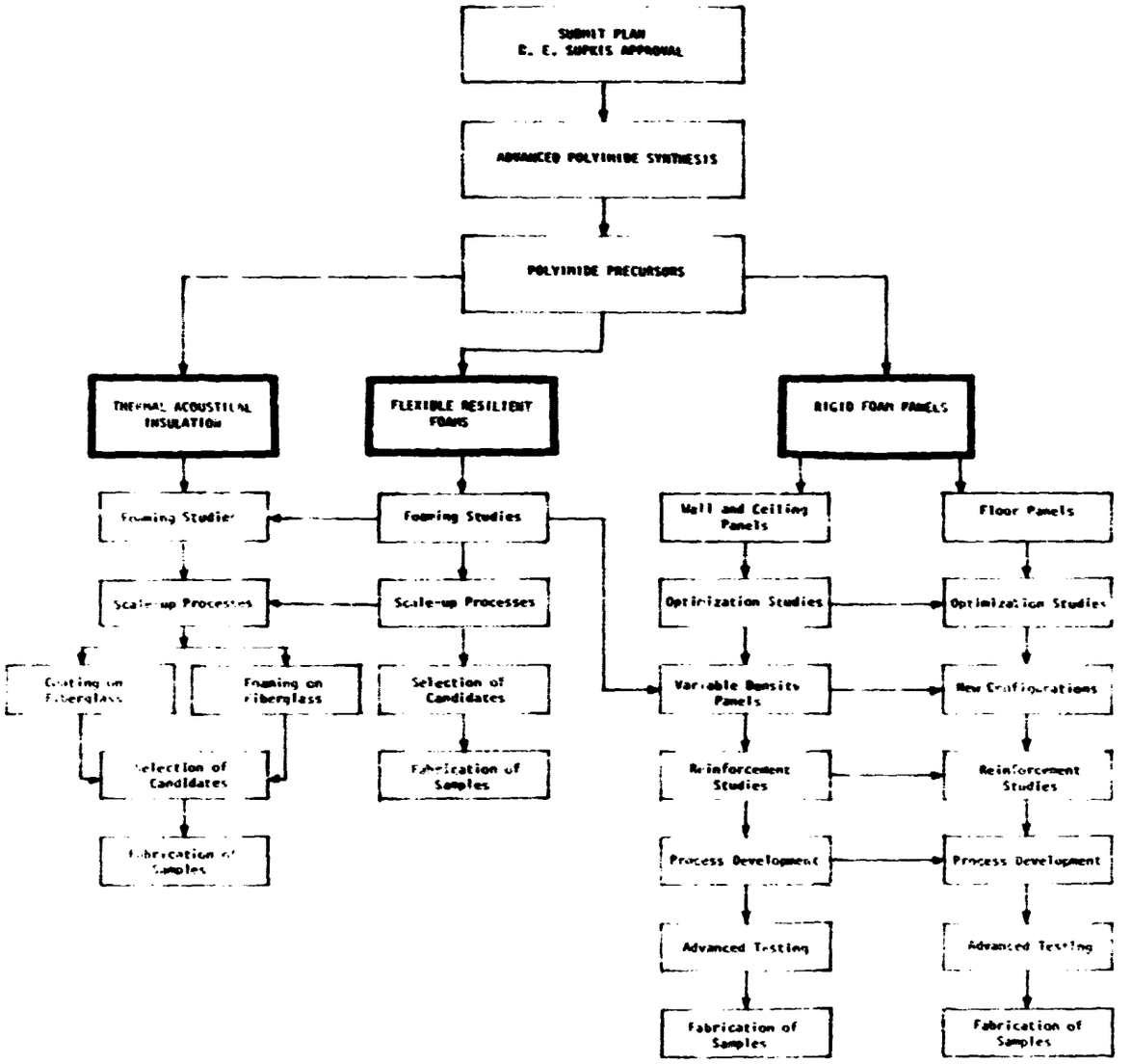


Figure 2. Program Flow Diagram

FLEXIBLE RESILIENT FOAMS

TASKS

- ADVANCED POLYIMIDE SYNTHESIS
- FOAMING STUDIES
- SCALE-UP PROCESSES

OBJECTIVES

- IMPROVEMENT OF COMPRESSION SET AND FATIGUE PROPERTIES

  OPTIMIZATION OF ALL PROCESSES FROM RESIN SYNTHESIS TO FINAL FOAMING

  SCALE-UP TO LARGE SIZE FOAMS

Figure 3. Flexible Resilient Foams

Foam Resin Number	Composition	Mole Ratio	Density		Resiliency Ball Rebound	% Loss After 30 Minutes Recovery	Type of Foam
			kg/m³	lbm/ft³			
1710-1-4	BTDA:2,6DAP:MDA:DAMPS	1:0.3:0.65:0.05	—	—	—	—	Flexible, resilient, DAMPS not compatible
1710-1-5	BTDA:2,6DAP:MDA:DAMPS	1:0.3:0.55:0.15	—	—	—	—	DAMPS not compatible
1710-1-6	BTDA:2,6DAP:MDA:DAMPS	1:0.3:0.40:0.30	—	—	—	—	DAMPS not compatible
1710-1-7	BTDA:2,6DAP:MDA:DAMPS	1:0.3:0.65:0.05	10.9	0.68	50-60	58.0	Flexible, resilient, good homogeneous cellular structure
1710-1-8	BTDA:2,6DAP:MDA:DAMPS	1:0.3:0.55:0.15	19.9	1.24	60-70	50.0	Flexible, resilient, good homogeneous cellular structure, some striations
1710-1-9	BTDA:2,6DAP:MDA:DAMPS	1:0.3:0.4:0.30	16.8	1.05	70	48.4	Flexible, resilient, good structure; some striations
1710-1-10	BTDA:2,6DAP:MDA:TDA	1:0.3:0.65:0.05	8.2	0.51	60-70	58.5	Flexible, resilient; homogeneous cell structure
1710-1-11	BTDA:2,6DAP:MDA:TDA	1:0.3:0.55:0.15	8.7	0.54	90	49.1	Flexible, resilient; homogeneous cell structure; some striation present
1710-1-12	BTDA:2,6DAP:MDA:TDA	1:0.3:0.4:0.30	12.2	0.76	60	51.7	Flexible, resilient; flaws and striations
1710-1-13	BTDA:2,6DAP:MDA:DAT	1:0.3:0.65:0.05	8.17	0.51	80-85	4.44	Flexible, resilient, homogeneous cellular structure
1710-1-14	BTDA:2,6DAP:MDA:DAT	1:0.3:0.55:0.15	9.77	0.61	>90	30.4	Flexible, resilient, homogeneous cellular structure
1710-1-15	BTDA:2,6DAP:MDA:DAT	1:0.3:0.4:0.3	9.93	0.62	80	40.8	Flexible, resilient, medium size cellular structure with some flaws
1710-1-16	BTDA:2,6DAP:MDA:XDA	1:0.3:0.65:0.05	9.93	0.62	75-80	59.0	Flexible, resilient, fine cellular structure
1710-1-17	BTDA:2,6DAP:MDA:XDA	1:0.3:0.55:0.15	18.42	1.15	60	52.4	Flexible, resilient fine cellular structure, flaws and striations
1710-1-18	BTDA:2,6DAP:MDA:XDA	1:0.3:0.4:0.3	18.74	1.17	55-60	44.3	Semi-rigid and hard foam with flaws
1710-1-19	BTDA:2,6DAP: Jeffamine AP-22	1:0.3:0.7	6.41	0.4	60	45.3	Flexible, resilient, homogeneous cellular structure

Figure 4. Properties of Advanced Aromatic Terpolyimide Systems

[illegible] Number	Aliphatic Diamine	Density		90% Compression Set - Loss After 30 Minute Recovery	[illegible] Ball Rebound	Foam Characteristics
		lbs/ft^3	kg/m^3			
[illegible]	None	1.538	8.[illegible]	52	55	Flexible, resilient, good structure
Group 1						
1720-1-1	Propyl	1.44	23.0	46	50	Flexible, resilient, good structure
1720-1-8	Butyl	1.32	21.1	63	45	Flexible, resilient, good structure
[illegible]	Hexa	[illegible]	[illegible]	48	[illegible]	Flexible, resilient, good structure
1720-1-3	Octa	0.943	15.1	39	50	Flexible, resilient, good structure
1720-1-4	Dodeca	1.62	25.9	42	60	Flexible, resilient, striated
1720-1-5	Jeffamine D-230	1.11	17.8	21	70	Flexible, resilient, large cell size, brittle
1720-1-6	Jeffamine D-400	--	--	--	--	Poor foam, collapsed on heating
1720-1-7	Jeffamine D-2000	--	--	--	--	Poor foam, collapsed and degraded on heating
Group 2						
1720-1-12	Propyl	0.840	13.4	40	40	Flexible, resilient, good structure
1720-1-9	Butyl	1.25	20.0	53	53	Flexible, resilient, good structure
1720-1-13	Hexa	0.817	13.1	47	55	Flexible, resilient, good structure
1720-1-10	Octa	1.40	22.4	43	35	Flexible, resilient, good structure
1720-1-14	Dodeca	3.32	53.0	46	70	Flexible, resilient, poor structure
1720-1-11	Jeffamine D-230	--	--	--	--	Brittle, very large cell size, poor foam
Group 3						
1720-1-15	Propyl	--	--	--	--	Rigid foam, collapsed and degraded on heating
1720-1-16	Butyl	1.48	23.7	63	50	Flexible, resilient, fair structure
1720-1-17	Hexa	1.37	21.9	71	50	Flexible, resilient, fair structure
1720-1-18	Octa	1.33	21.2	68	45	Flexible, resilient, good structure
1720-1-19	Dodeca	0.778	13.5	45	70	[illegible]ible, resilient, good structure
Group 4						
1720-1-25	Propyl	1.33	21.2	40	50	Flexible, resilient, good structure
1720-1-20	Butyl	0.835	13.[illegible]	25	45	Flexible, resilient, good structure
1720-1-24	Hexa	1.44	23.0	30	55	Flexible, resilient, medium cell size
1720-1-21	Octa	0.845	13.5	22	70	Flexible, resilient, medium cell size
1720-1-23	Dodeca	0.565	[illegible].04	23	65	Flexible, resilient, good structure
1720-1-27	Jeffamine D-230	--	--	--	--	Brittle, very large cell size, collapsed on heating
Group 5						
1720-1-26	Butyl	1.15	18.3	31	50	Flexible, resilient, good structure
1720-1-27	Hexa	0.399	6.36	7	55	Flexible, resilient, highly reticulated

Figure 5. Aliphatic Terpolyimide Foam Precursors and Foam Characteristics (1702-1 Resin System)

Foam Resin Number	Composition	Molar Ratio	Concentration of Surfactant AS-2 (%)	% Loss After 30 Minutes Recovery	Type of Foam
1702-1-62	BTDA:2,6DAP:MDA	1:0.3:0.7	0.0125	40.0	Fine, homogeneous cellular structure
1701-1-5	BTDA:2,6DAP:MDA	1:0.4:0.6	0.0125	19.6	Medium-large homogeneous cellular structure
1701-1-7	BTDA:2,6DAP:MDA	1:0.42:0.58	0.0125	12.1	Reticulated foam with medium size cellular structure
1701-1-8	BTDA:2,6DAP:MDA	1:0.44:0.56	0.0125	—	Highly reticulated foam with large and weak cellular structure
1701-1-10	BTDA:2,6DAP:MDA	1:0.5:0.5	0.0125	—	Highly reticulated foam with chopped strands like cell structure. Poor - hollow foam

Figure 6. Flexible, Resilient, Polyimide Foams: Effect of Molar Concentration Of 2,6DAP On Compression Set Loss Values

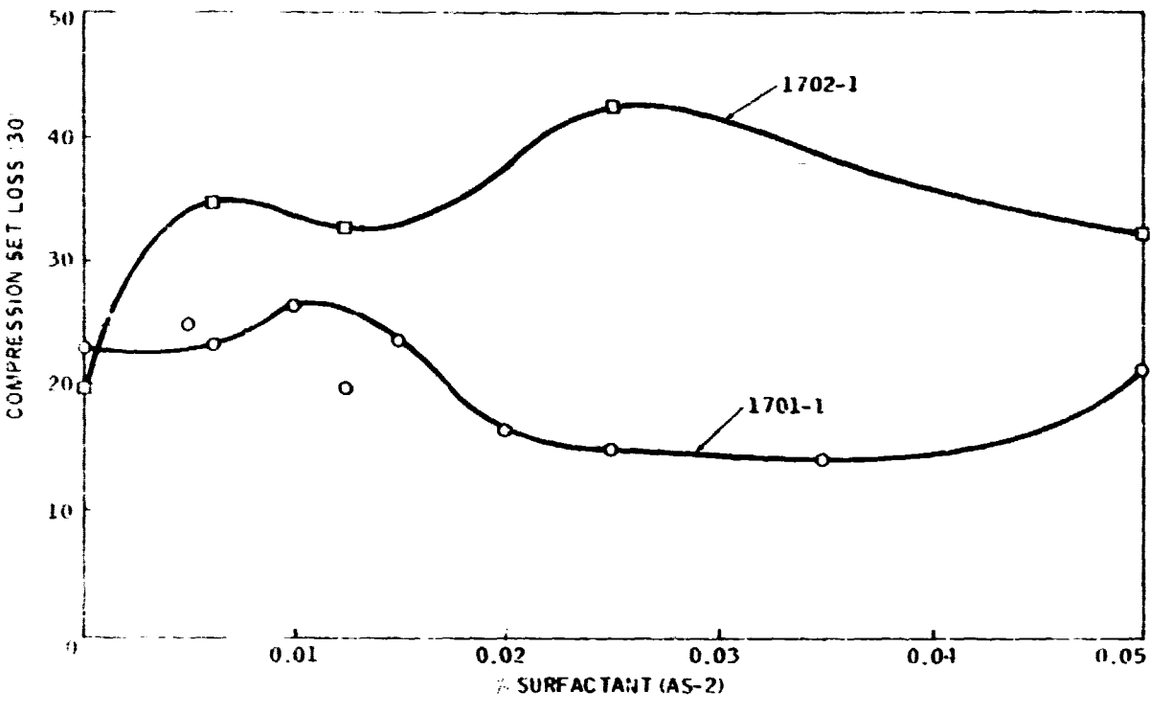


Figure 7. Flexible, Resilient Polyimide Foams; Effect of Surfactant Concentration (AS-2) On Compression Set Loss

Power Output (kW)	Time to Foam (Seconds)	Total Foaming Cycle (Seconds)	Foaming Quality
2.5	120	240	fine cellular structure
5.0	75	210	fine cellular structure
10	60	180	fine cellular structure large portion of foam cured in microwave

Figure 8. Foaming Behavior of 1702-1 Precursors At Various Power Outputs

Figure 9. Effects of Powder Loading in Properties of Polyimide Foams

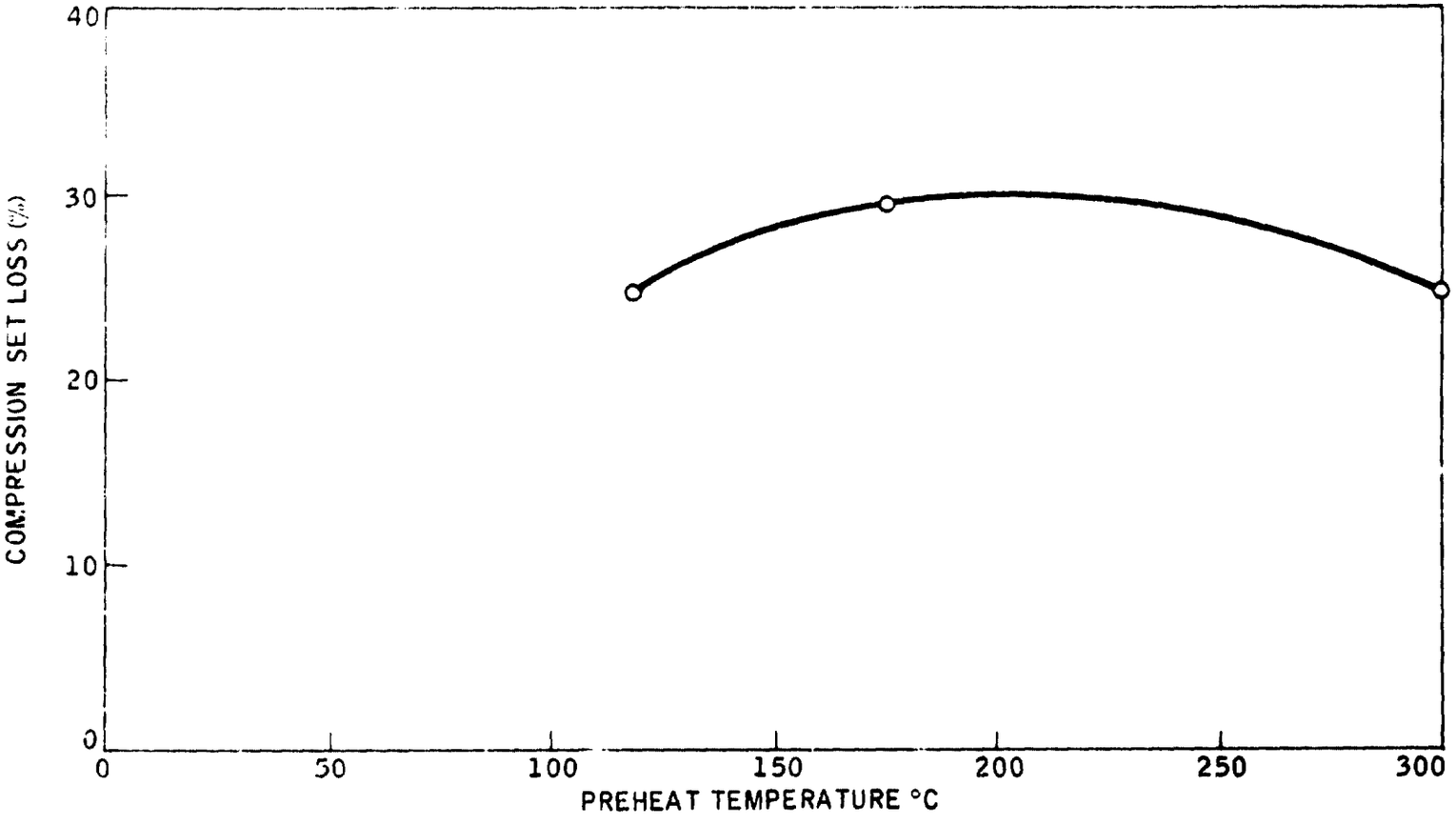


Figure 10. Effect of Preheat Temperature (2 Min.) on Compression Set Loss of 1701-1 Polyimide Foams Modified With 0.015 Percent AS-2

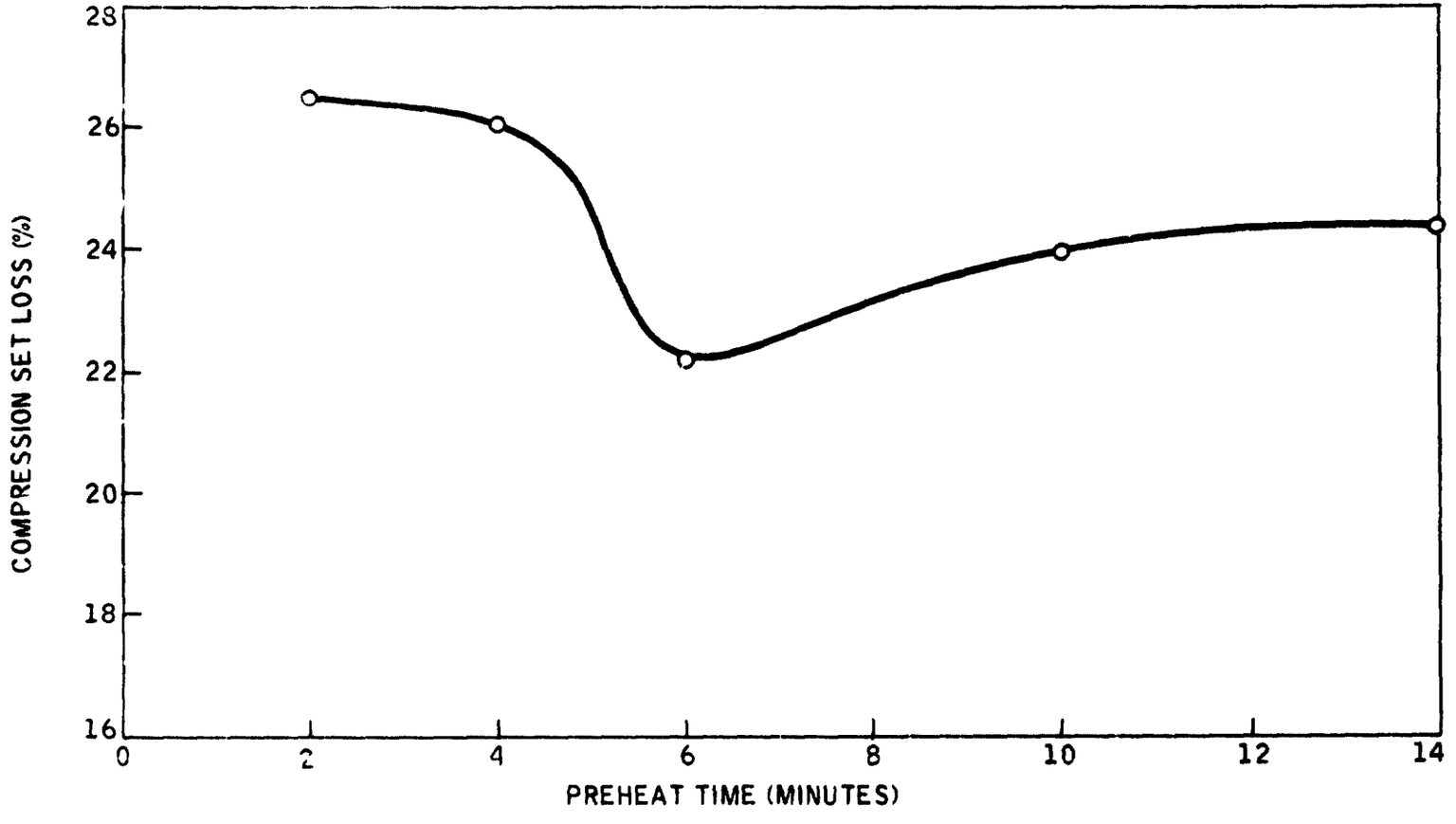


Figure 11. Effect of Preheat Time on Compression Set Loss (250°F) of 1701-1 Polyimide Foams

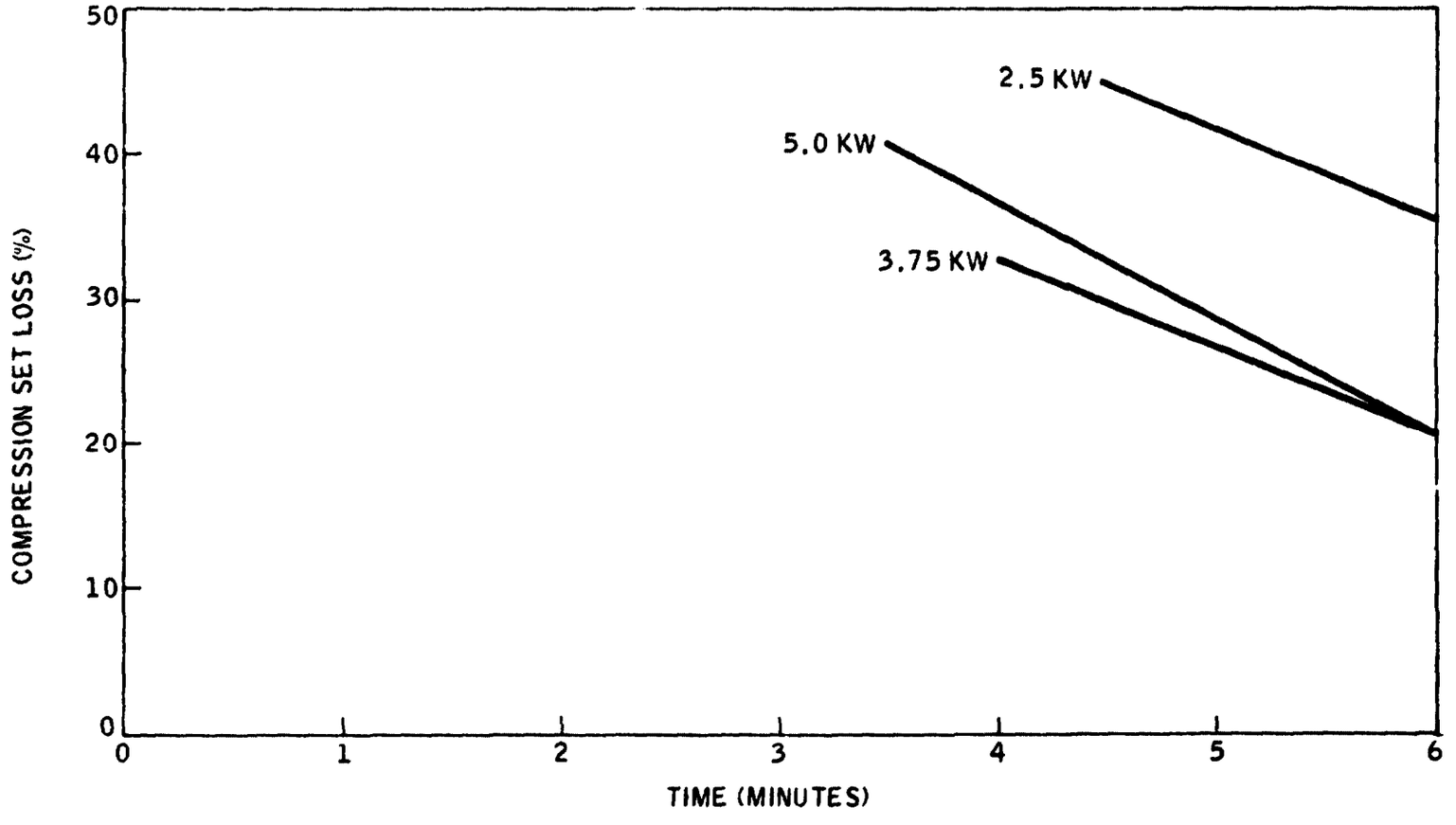


Figure 12. Effect of Foaming Time on Compression Set Loss of Aliphatic Terpolyimide System (1720-1)

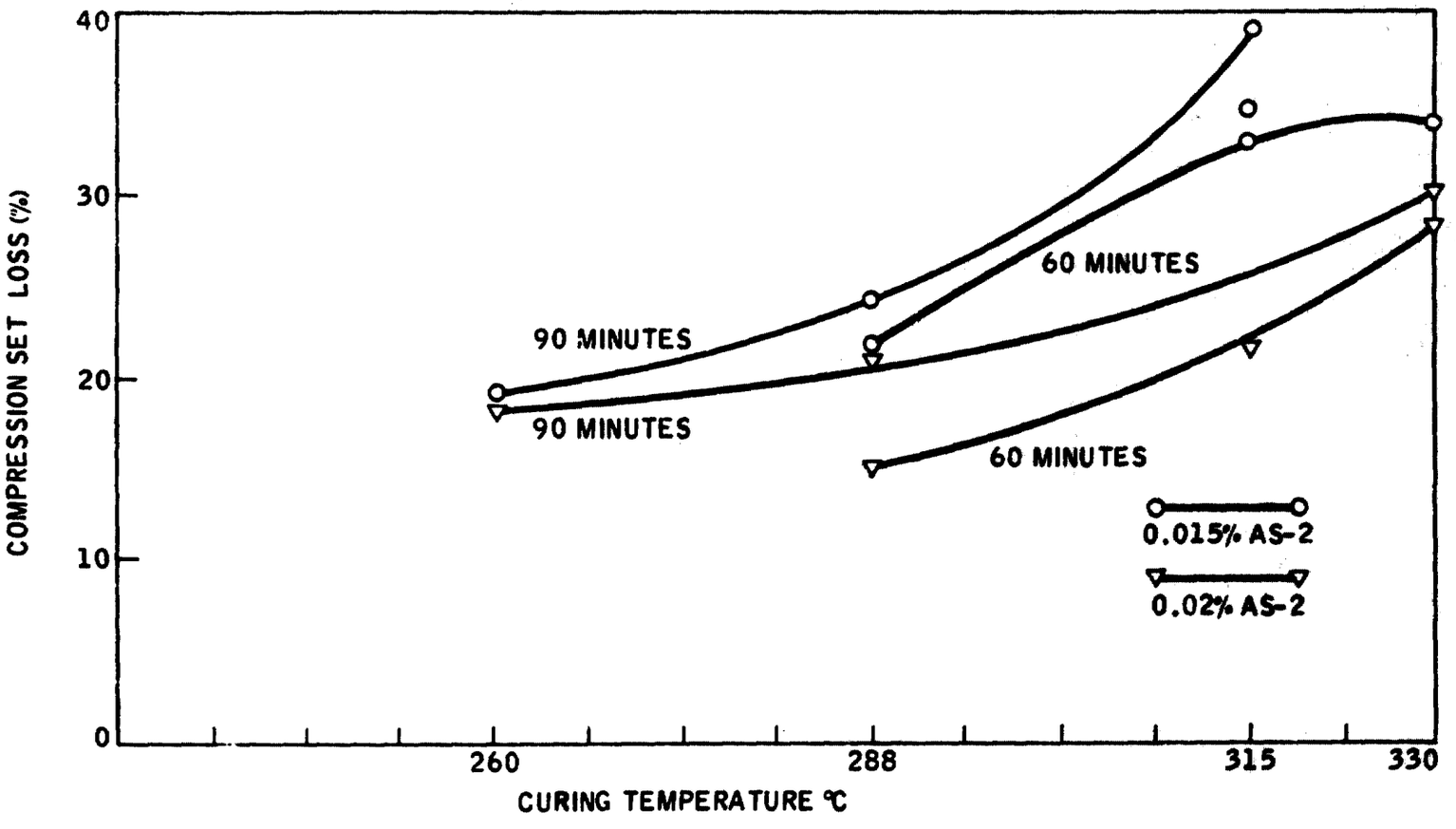


Figure 13. Effect of Curing Temperature on 90% Compression Set Values of Foams Derived from 1701-1 Precursors Modified With 0.015% and 0.02% AS-2 Respectively

LOW DENSITY WALL PANELS

TASKS

- OPTIMIZATION OF RIGID POLYIMIDE FOAM PANELS
- LOW DENSITY CORE, HIGH DENSITY SKIN PANELS
- OPTIMIZATION OF LOW DENSITY CORE TECHNOLOGY

OBJECTIVES

- DEVELOPMENT OF TECHNIQUES TO PRODUCE FINAL WALL PANEL CONFIGURATIONS IN A ONE-STEP PROCESS WITHOUT THE USE OF ADHESIVES.
- FABRICATION OF WALL PANELS HAVING LOW DENSITY CENTERS AND HIGH DENSITY EDGES TO MEET DIRECT SCREW WITHDRAWAL REQUIREMENTS.

Figure 14. Low Density Wall Panels

Figure 15. Fabrication of Rigid Low Density Panels From Powder Polyimide Precursors in a One-Step Microwave Process

HIGH STRENGTH FLOOR PANELS

TASKS

- OPTIMIZATION OF RIGID POLYIMIDE FOAM PANELS
- NEW CONFIGURATIONS

OBJECTIVES

- DEVELOPMENT OF PANEL CORE MEETING HIGH TRAFFIC AREA REQUIREMENTS

  DEVELOPMENT OF RIGID PANELS WITH VARIABLE DENSITY CHARACTERISTICS

Figure 16. High Strength Floor Panels

THERMAL ACOUSTICAL INSULATION

TASKS

- ADVANCED POLYIMIDE SYNTHESIS

  FOAMING STUDIES

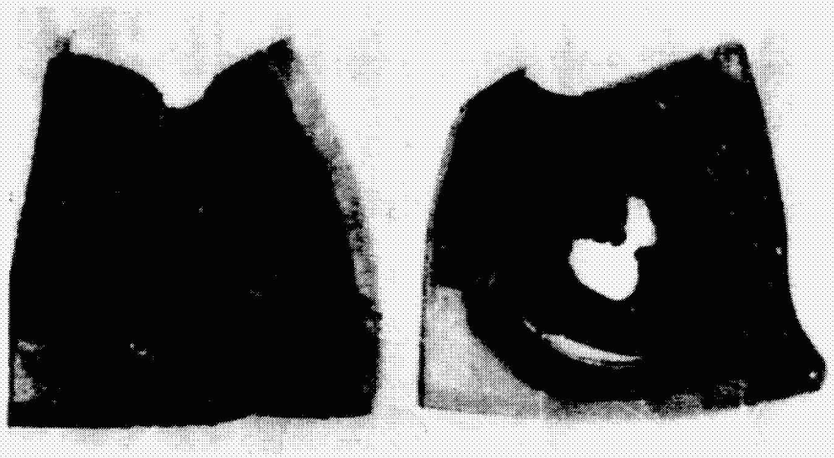
  COATING PROCESS FOR GLASS FIBERS AND MATS

OBJECTIVES

OPTIMIZATION OF THE BURNTHROUGH PROPERTIES OF THE FOAMS

Figure 17. Thermal Acoustical Insulation

Figure 18. 1702 Polyimide Resin Spray Coated & Foamed on Owens-Corning PF 105-700 Fiberglass 20 x 20 CM Effect of Loading on Burn Test



GLASS & MICROBALLOONS **UNFILLED**

NO FAILURE AFTER TEN MINUTES EXPOSURE **BURNTHROUGH 2.5 MINUTES**

Figure 19. Effect of Filers on Burnthrough Characteristics of Polyimide Foam: Left 1702-1, 1.0% AS-2; 20% Glass Strands 3% Microballoons

FUTURE PLANS

- Development of large scale foam processing.
- Fabrication of shaped flexible foams by the use of closed or open molds.
- Optimization of the microwave process to produce rigid panels with densified skins.
- Development of thermal acoustical materials meeting the burnthrough requirements
- Selection of one or more candidates for each of the products under study.

Figure 20. Future Plans